

H. M. Woodroffe, Industrial Mineral Division

CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 59-72

REGENERATION OF LIME FROM CALCIUM CARBONATE WASTE SLUDGES
FROM THE BEAVERLODGE (SASK.) PLANT OF ELDORADO
MINING AND REFINING LIMITED

by

E. G. JOE

RESEARCH AND DEVELOPMENT LABORATORY
ELDORADO MINING AND REFINING LIMITED

H. M. WOODROFFE, H. S. WILSON and J. S. ROSS

INDUSTRIAL MINERALS DIVISION

This document was produced
by scanning the original publication.

Ce document est le produit d'une
numérisation par balayage
de la publication originale.

Mines Branch Investigation Report IR 59-72

REGENERATION OF LIME FROM CALCIUM CARBONATE WASTE
SLUDGES FROM THE BEAVERLODGE (SASK.) PLANT OF
ELDORADO MINING AND REFINING LIMITED

by

E. G. Joe*, H. M. Woodrooffe**, H. S. Wilson***
and J. S. Ross***

==

SUMMARY

The problem of regenerating lime from a calcium carbonate sludge at the Beaverlodge mill was investigated jointly by the Industrial Minerals Division of the Mines Branch and by Eldorado Mining and Refining Limited.

During the milling of uranium ore at this plant, a calcium carbonate sludge is precipitated as a waste product. Because of the location, remote from sources of supply, transportation costs of lime are five to six times the price at the lime plant.

Several calcination processes and several methods of preparing a calciner feed were studied during the investigation.

Although it was technically possible to regenerate lime by three processes, it was concluded that the most desirable economically for the conditions prevailing at Beaverlodge was the one utilizing the Herreshoff roasting furnace.

*Research Engineer, Research and Development Laboratory,
Eldorado Mining and Refining Limited, Ottawa, Canada.

Head, Non-Metallic Minerals Section, and *Scientific Officers,
Industrial Minerals Division, Mines Branch, Department of
Mines and Technical Surveys, Ottawa, Canada.

TABLES

	<u>Page</u>
Table 1. - Chemical and Size Analyses of Beaverlodge Sludges	3
Table 2. - Results of Pellet Binder Tests	12
Table 3. - Comparison of Lignin Sulphonate Binders	13
Table 4. - Pelletizing with Calcium Carbonate Slurry ..	14
Table 5. - Assay of Calcining Tests on Pellets	20
Table 6. - Results of Large Scale Calcination of Pellets.	21
Table 7. - Summary of FluoSolids Tests Conditions	24
Table 8. - Chemical Analyses for FluoSolids Reactor Tests	29
Table 9. - Operating Conditions for FluoSolids Reactor Tests	29
Table 10. - Product Distribution for Test Number 3	29
Table 11. - Results of Calcining Tests on Beaverlodge Pellets at Elevated Temperatures	43
Table 12. - Calcination of Calcium Carbonate Pellets in Herreshoff Furnace	45
Table 13. - Calcination of Coarse and Fine Spiractor Calcium Carbonate Composite	47
Table 14. - Calcination of Fine Spiractor Calcium Carbonate	48

FIGURES

	<u>Page</u>
Figure 1. - 6" Diameter Spouted Bed Drier	17
Figure 2. - Dorr Oliver 4" FluoSolids Reactor	26
Figure 3. - Shaft Kiln for Sludge Pellet Calcinations	33
Figure 4. - Travelling Hearth Kiln	37
Figure 5. - Calcination of Calcium Carbonate Pellets.....	40
Figure 6. - Calcination of Calcium Carbonate Pellets.....	41
Figure 7. - Calcination Rate of Beaverlodge Coarse and Fine Sludge Composite	49
Figure 8. - Calcination Rate of Beaverlodge Fine Sludge	50

=====

INTRODUCTION

Eldorado Mining and Refining Limited operates a basic carbonate leach plant for the treatment of uranium ore at Beaverlodge Lake, Saskatchewan, approximately 500 air miles northeast of Edmonton. The nearest lime producer to that area is about 700 miles distant.

The addition of lime to the Eldorado Beaverlodge carbonate leach circuit has effected economies in the reagent costs. Although daily requirements of lime are only $7 \frac{1}{2}$ tons, high transportation costs, handling charges and the limitation of shipping lime in the hydrated form increased the cost of this reagent at the mill by six to seven times the producer price. It was realized that further substantial savings could be achieved by calcination of the calcium carbonate sludge from the circuit to yield a continuous supply of lime.

The selection of a process and equipment for lime regeneration was made more difficult by the small daily tonnage required and the floor space limitation of 20 ft by 25 ft and a limited height of 50 ft. The design of most lime calcination equipment is predicated on treating larger tonnages of coarse limestone. However, the relatively high value of lime at Beaverlodge allowed the consideration of techniques and processes not generally deemed economic by lime producers in more accessible locations.

Eldorado Mining and the Industrial Minerals Division, Mines

Branch, undertook an investigation of the lime calcination possibilities, with a view to selecting a workable process accompanied by reasonable capital and operating costs. The work involved visits to actual lime-producing plants and a considerable amount of laboratory and pilot plant testing in various types of calcination units.

During the investigation, several plants were visited by representatives of Eldorado Mining and Refining Limited.

DESCRIPTION OF CARBONATE SLUDGES

Calcium carbonate sludges tested were produced either by direct precipitation with lime slurry or formed in a conical spiractor. Precipitated sludge was mainly slime, with most of the particles in the minus 10 micron size range. Spiractor sludges consisted of two sizes. One was a coarse discharge with particles mainly spherical in shape and ranging in size from 35 to 100 mesh. The fines from the spiractor were distinctly granular, although the size particles were all minus 40 microns.

All sludges were light brown in colour and contained minor impurities of U_3O_8 , sodium sulphate and sodium carbonate. Directly precipitated sludges were extremely difficult to filter, and cloth blinding was quite common. The spiractor discharge product was readily de-watered and washed in a classifier. Spiractor fines were more readily filtered than directly precipitated fines. Since it was necessary to wash out U_3O_8 and sodium salt impurities prior to calcination, the spiractor pro-

ducts were considered to be the more desirable type of sludge to treat. The spiractor at Beaverlodge produces approximately 60% of the calcium carbonate as coarse material and 40% as fines.

Chemical and size analyses on the sludges are as follows:

TABLE 1
Chemical and Size Analyses of Beaverlodge Sludges

Analyses	Direct Pptd Sludge	Spiractor Product	
		Coarse	Fine
% U ₃ O ₈ (dry wt.)	0.27	0.16	0.13
% Na (dry wt.)	4.08	2.21	2.49
% CaCO ₃ (dry wt.)	90	90	90
% H ₂ O	43.5	21.8	41.5
<u>Weight %</u>			
+35 mesh		0.4	
-35 +65 mesh		53.4	
-65 +100 mesh		39.2	
-100 +200 mesh		6.7	
-200 mesh (74 micron)		0.3	
+40 micron	4.93		
-40 +2- micron	4.00		19.0
-20 +10 micron	12.67		27.2
-10 micron	78.40		53.8

Sludges were re-washed with water to recover entrained uranium values and to lower the sodium content to approximately 1% (dry weight basis) prior to calcination.

LIME REGENERATION PROCESSES

Lime is recovered from waste sludges from chemical and industrial plants in an attempt to reduce stream pollution, to ease the burden of purchasing and maintaining increasing waste disposal areas, and to obtain a valuable by-product. Various lime regeneration processes have been developed, each having its own merits depending on the chemical content of the sludge, capacity required, space limitations, thermal efficiency, capital cost, operating cost, location, and the required quality of product.

The Rotary Kiln

The rotary kiln has become one of the standard calcining mechanisms in the lime industry and will not be described here. The limitations of a process involving a rotary kiln are high initial capital costs, high capacity, need of large plant area, and, depending on the size of the feed, the necessity of a relatively low sodium and silica content. The main limitation is the required capacity which, if less than 50 tons of lime a day, may not provide as economical an operation as other processes, depending on local conditions. Sludges are calcined in rotary kilns in a number of ways.

Wet sludge or filter cake as feed: This method is the most common means of reclaiming lime from waste sludge and is particularly popular in the pulp and paper industry. Here, depending on local conditions, the sludge is washed to reduce the Na_2O content to less than

approximately 2.5 %. The filter cake or re-slurried cake, normally containing from 50 to 75 % solids, is then fed to a rotary kiln equipped with chains fastened by one end to the kiln wall. Firing temperatures approximate 2200°F, and exit gases are approximately 500°F. Dust collectors are usually essential. Depending mainly on the density of the sludge, the resultant product varies from a high percentage of nodulation to dust. The lime produced commonly has a CaO content equivalent to primary lime. A common operating problem with this method is the continual ring accretions on the kiln walls, and excessive balling due to such impurities as sodium and silica.

Field trips were made to observe rotary kiln operations at the plants of Aluminum Company of Canada Limited at Wakefield, Quebec, and Canadian International Paper Company at La Tuque, Quebec.

Dry sludge as feed: This process, with a few innovations, involves washing the sludge and then drying it in a cage mill utilizing the exhaust kiln gases as a source of heat. The dried sludge is air-conveyed to a collector and fed into a mixer. Here it is mixed with incoming wet sludge. The resultant moist sludge is automatically divided into two parts, one part being the sole feed to the cage mill and the other dropping onto a screw conveyer. The moist sludge on the conveyer is dried further by hot exit kiln gases passing through the conveyer compartment into the cage mill.

This process is relatively new and has the advantage of

lower fuel consumption and greater kiln capacity than the usual process involving wet sludge as feed to the cage mill. Although most of the dust resulting from the exceptionally high dust conditions is not lost, this process has the disadvantage of high dust recirculation and possible overburning, as well as higher than normal capital and operating costs.

Pellets and briquettes as feed: Pellets of calcium carbonate sludge, commonly made in inclined pan or horizontal drum pelletizers with the use of organic binders such as lignin sulphonate, can be used as feed for rotary kilns. At least a large portion of the sludge must be dry before pelletizing. To avoid decrepitation and the resultant high dust losses the pellets, normally from 1/4 to 1 in. in diameter, should be essentially dry before being fed to a standard rotary kiln. This may be done by utilizing waste exhaust gases. Advantages of this process are the lack of balling, the lack of ring-building causing costly shut-downs, a uniformly sized product from which is produced a high quality lime, a greater capacity due to a dry feed, lower fuel costs, and a minimum dust loss. Offsetting the advantages are the increased capital cost and the additional operating cost incurred during the drying of the sludge, pelletizing, and drying of the pellets.

Briquettes of calcium carbonate are also used for kiln feed. In this case, the dried sludge with a minor amount of water and binder is compressed into briquettes by closed mould processes. The briquettes can then be fed directly to the kiln. The advantage of this

type of sludge preparation over that involving the use of pellets is that briquettes are harder than pellets and are produced dry. The disadvantage is that the operating and capital costs of briquetting are greater than those of the actual pelletizing process.

The Ellernan Vertical Kiln

The Ellernan vertical kiln was designed to calcine crushed stone plus 1/4 and minus 1 1/2 in. in size. The preferred size is 1 1/4 to 1 1/2 in. This kiln is composed of a feed hopper separated by baffles from an underlying calcining chamber. The vertical calcining chamber contains numerous staggered baffles and has multiple continuous discharge mechanisms at the bottom. These discharge mechanisms allow the rate of flow of stone through each section of the chamber to be varied independently so that the resulting product is uniform. Gases at approximately 2800°F are produced in a separate combustion chamber and pass into the calcining chamber under tunnel beams below the two top layers of baffles.

This process is designed for feed similar in size to pellets and briquettes. However, because of the difficult operating conditions experienced by some of the few known operators, and the preferred size of feed, a process using the Ellernan kiln was not investigated further.

Vertical Kilns

Standard vertical kilns have not been adapted to the calcination of sludge. However, vertical shaft furnaces are used to calcine

pellets and briquettes of iron ore concentrates. As a result, a field trip was undertaken to the Hilton Mine at Shawville, Quebec, where 40-ft-high rectangular furnaces calcine pellets of magnetite 9/16 in. in diameter at a temperature of 2300°F. Heat is forced through the bed by burning bunker C oil at a rate equivalent to 0.5 ton per ton products.

Herreshoff Roaster

The Herreshoff furnace consists of one or more horizontal circular hearths stacked one above the other in a refractory-lined cylindrical steel shell. Rabble arms are moved across each hearth by means of a vertical rotating shaft passing through the centre of the furnace. In this way, material fed to the top hearth is rabbled across each hearth, passing through an opening in the hearth to the underlying one, and is eventually discharged. Heat for calcination may be supplied by the combustion of fuel in burners on certain hearths. A fan circulates air from the bottom of the furnace towards the top. Wedge roasters operate on somewhat similar principles.

Because of the large possible differences in temperatures between the top and lower hearths, wet as well as dry material may be calcined efficiently. Wet or dry sludge and wet or dry pellets of sludge may be calcined in a Herreshoff furnace. For the production of lime, dust losses and scale build-up are relatively low. The main limitation is that the largest economical unit has a rated output of lime in the order

of 50 tons a day.

Field trips were undertaken to study the calcination of wet sludge in a Wedge roaster at Allied Chemicals, North Philadelphia, Pennsylvania, and the calcination of wet sludge in a Herreshoff furnace at Netcong, New Jersey.

FluoSolids Reactor

Operation of the FluoSolids lime reclaiming plant at the City of Lansing, Michigan, was observed during a field trip. Here wet sludge containing 65 % solids is dropped in front of a cage mill into the waste gas stream from the roaster, where it is broken up, dried, and removed to a cyclone. The cyclone product is divided into two parts with one part being returned to be mixed with the incoming wet sludge and the other portion going to the reactor. The FluoSolids reactor consists of a vertical steel cylinder divided into a larger upper calcining and a smaller lower cooling compartment by a perforated arch. Heat is supplied to the large compartment by oil burners, and air is forced upwards from the bottom of the reactor. Lime pellets are formed on top of the perforated arch, due to the combined action of the upward flow of air and molten sodium carbonate impurities. Calcined pellets drop to the lower cooling compartment and eventually to a bucket elevator.

This process is relatively new to the lime industry.

Travelling-Hearth Tunnel Kiln

The Calcimatic travelling-hearth tunnel kiln has been developed recently by Surface Industrial Furnaces Limited, primarily for the calcination of limestone. A patent on the process is pending. As a result,

until the kiln was brought to the attention of Mr. E. G. Joe, it had not been investigated as a possible calcining apparatus for calcium carbonate sludge.

The kiln is doughnut-shaped, with the tunnel width approximately one-third of the overall diameter. Both hearth and arch are lined with refractories and the horizontal hearth rotates on rollers. The system is contained by water seals. The tunnel is divided into the required number of combustion chambers by means of vertical baffles extending from the arch downward almost to the top of the calcining bed. The temperature of each chamber is automatically controlled by standard burners in the arch. Feed, preferably 1/4 to 4 in. in size, is fed through the arch onto the hearth to the required depth, where it is rotated in a direction opposite to the flow of hot gasses. Lime is discharged after one rotation immediately before the feeder is reached.

With regard to the problem in question, the main desirable features of the Calcimatic hearth are that the exact time of travel of the feed and the exact required temperature conditions can be controlled and the equipment may be designed for small tonnages of small-sized feed. The equipment is relatively small and compact and has a relatively low capital cost. However, the Calcimatic kiln is limited in that it does not process fines suitably because of excess dust losses and has a relatively high heat consumption.

A field trip was made to Toronto to observe the calcination of pellets under conditions existing in a Calcimatic kiln.

PREPARATION OF CALCINER FEED

Because the calcium carbonate sludge was produced by precipitation and from a conical spiractor, it was necessary to investigate the preparation of feed of both materials. Various methods of preparation were investigated to determine which would be suitable for the calcination investigation.

Precipitated Wet Sludge

Filter Cake

A one-hundred-pound batch of sludge was filtered and washed with 40 lb of a 5% solution of lignin sulphate on a filter pan and the resultant cake was cut into 1 in. x 1 in. x 3/4 in. pieces. Calcium carbonate sludge containing water alone does not possess much strength. The sludge contained about 40 % water and it was hoped that by washing it with the lignin sulphate solution, which is a binder, the strength of the cubes would be increased.

Extrusion

An attempt was made to produce a feed by means of a small extrusion machine, the die containing 3/8 in. diameter holes. It was found that the consistency of the workable sludge required a moisture content so high that the streams extruded would not support their own weight. Consequently they slumped and so could not be fed to a calciner.

Pelletizing

Pellet Binder Tests

Pelletizing tests were made on dried pulverized lime sludge, using various binders to determine which would be most suitable. Only those binders which could be completely burned out in calcining were tested. These included water, corn starch, arrowroot starch, potato starch, and lignin sulphate. The binders were dissolved in water and then sprayed onto the dried calcium carbonate as it revolved in a small concrete mixer. The strengths of the resultant pellets were measured by repeatedly dropping them from a height of 12 in. onto a steel plate until they fractured. The number of drops obtained from ten pellets were averaged. Strengths were measured on pellets in both green and dry states. Conditions and results are shown in Table 2.

TABLE 2

Results of Pellet Binder Tests

Binder	% Moisture	Binder, lb/ton CaCO ₃	Binder, lb/ton CaO*	Drop Strength	
				Green	Dry
Water	25.5	-	-	4	1
Corn starch	26.0	24.5	48.5	10	2
Arrowroot starch	26.0	24.5	48.5	15.7	4
Potato starch	26.0	24.5	48.5	14.3	2.6
Lignin sulphate	22	19.6	38.8	9	13.5
Lignin sulphate	27	48.4	95.8	30	30+

* Assuming 90% conversion of CaCO₃ to CaO.

Based on previous Mines Branch experience, a strength of 10 was considered adequate to be handled in a rotary kiln. The three starches gave green strengths that were satisfactory, but the dry strengths were much too low. The lignin sulphonate alone gave satisfactory dry strengths.

Samples of two types of lignin sulphonate were obtained from Lignosol Chemicals Ltd., Quebec, Que. The two types were Lignosol B.D. (calcium lignosulphonate) and Lignosol TSD (ammonium lignosulphonate). Two hundred pounds of calcium carbonate sludge were dried and pulverized for pelletizing tests using the two binders which were made into solutions containing 3 1/2 and 7% solids. Pelletizing was done in a Lurgi disc pelletizer. The green and dry strengths of the pellets were measured by means of drop tests. Pelletizing conditions, results, and cost of binder are shown in Table 3.

TABLE 3

Comparison of Lignin Sulphonate Binders

% Binder	% Moisture	Binder, lb/ton CaCO ₃	Binder, lb/ton CaO*	Binder, Cost/lb ¹	Binder, \$/ton CaO	Drop Strength	
						Green	Dry
3 1/2% BD	24	22	43.7	3	1.31	+30	15
7% BD	24	44	87.4	3	2.62	+30	+30
3 1/2% TSD	24	22	43.7	5	2.18	+30	+30
7% TSD	24	44	87.4	5	4.37	+30	+30

*Assuming 90% conversion CaCO₃ to CaO.

1. F. o. b. Quebec, Que. - carload lots.

Pelletizing of Filter Cake

In the pelletizing tests to this point, the sludge was dried prior to pelletizing. Tests showed that filter cake could be slurried at 50% solids and be fluid enough to be sprayed. Consequently, wet filter cake was repulped with lignin sulphonate solution at 50% solids. The resultant slurry was then sprayed onto dried calcium carbonate in the Lurgi pelletizer. The slurry was sprayed in equivalents of 27 and 54 lb of lignin sulphonate per ton of calcium carbonate. The green and dry strengths of the pellets were measured. The pelletizing conditions and results are shown in Table 4.

TABLE 4

Pelletizing with Calcium Carbonate Slurry

Run No.	Lignosol BD, lb/ton CaCO ₃	% Moisture	Wet Strength	Dry Strength
1	27	25.0	26.5	3.3
2	54	25.0	28.1.	4.4

The wet strengths were satisfactory for handling, but the dry strengths were too low.

Drying of Pellets

Tests showed that the pellets would shatter if heated to a high temperature rapidly unless they were first dried. It was necessary to determine the maximum temperature at which they would dry without

shattering. The 5 in. by 5 ft rotary kiln was equipped with a thermo-couple so that the exhaust gas temperature could be measured. A range of temperature from 350° to 1000°F was used with a 5 minute retention time. The results were as follows:

<u>Temperature</u>	<u>Effect on Pellets</u>
350° F	Nil
450°	Nil
525°	Nil
800°	Shattered
1000°	Shattered

This indicates that green pellets could be introduced into a rotary kiln at an exhaust gas temperature of up to 800°F.

Spiractor Product

Pelletizing

An attempt was made to pelletize a coarse spiractor product described on page 2 under Description of Carbonate Sludges. This sludge contained 20.59% moisture. A partial chemical analysis (dry weight basis) is as follows:

% Ca	-	37.0
% CO ₂	-	42.7
% Na	-	0.90
% U ₃ O ₈	-	0.12

This coarse material was dried and then pelletized in the Lurgi disc pelletizer, using lignin sulphonate as a binder. The resultant pellets were very weak; the green strength was between 3 and 4. The spiractor product was reduced to 91.6% minus 325 mesh with one pass through a Raymond Pulverizer. From this material, pellets were made which could be readily handled in the calciner.

Drying

One of the problems encountered in this investigation was the necessity to dry the sludge prior to pelletizing. Drying tests were carried out in a spouted-bed drier at the National Research Council.

The drier used for these tests is shown diagrammatically in Fig. 1. Hot combustion gas is introduced at the bottom of the vertical column through a 1/2 in. orifice. Wet feed is introduced continuously at the top of the column, and the dry product discharges through overflow pipes at the top of the bed, part way up the column. Discharge gases are vented to a cyclone from the top of the column. Combustion gas is produced in a separate combustion chamber operating at a positive pressure. For these tests, propane gas was the fuel used.

Dried, minus 48 mesh spiractor sludge containing less than 1.5% Na was the initial charge. The operation was started by adding the dry sludge to the column until discharge started through the lower overflow pipe. Simultaneously, air was introduced through the

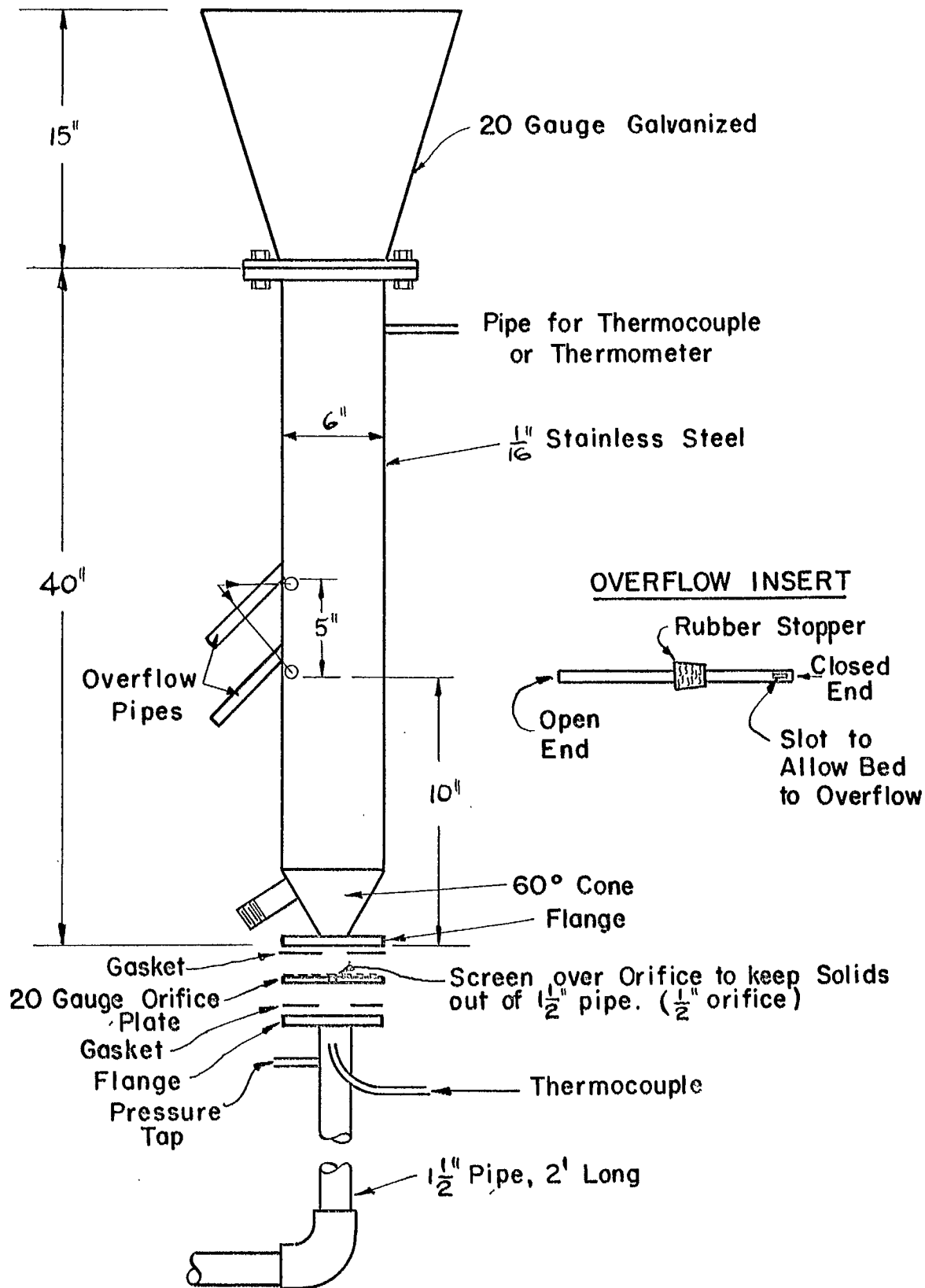


Figure 1 Spouted Bed Drier

orifice at equivalent pressure to combustion gas flow (0.173 c f m.) . When discharge commenced, the burner was ignited and the bed temperature was raised to 300°F, at which stage a continuous feed of wet sludge was introduced. The wet sludge was prepared by mixing the dry -48 mesh material with water to a moisture content of 31.4%. The rate of feed was 44 lb/hr. The moisture content of the product was 0.4%. Under steady conditions the gas temperature was about 1425°F and the bed temperature about 190°F. Actual heat consumption was calculated as 1960 Btu per lb of water evaporated, based on a heat of combustion of 2385 Btu per cu ft (standard) for propane. Further heat balance calculations based on a sensible heat of combustion gases indicate a heat consumption of about 1270 Btu per lb of water evaporated. The difference is apparently caused by low thermal efficiency of the burner.

The material balance showed a large dust loss. The dust recovery from the cyclone was very small, although there may have been some dust adhering to the wall of the column as it was wetted by condensing water.

CALCINATION

Rotary Kiln

Two rotary kilns were used in the calcination investigation: a 5 ft. by 5 in. propane-fired, and a 12 ft by 12 in. natural gas-fired kiln. A gas-fired muffle kiln was also used in the investigation.

Calcination of Filter Cake

The 1 in. by 1 in. by $3/4$ in. pieces of filter cake were introduced into the 12 ft by 12 in. rotary kiln by means of a screw feeder. The temperature of the kiln was maintained at about 1800°F. The feeder abraded the cubes, forming oval-shaped pellets ranging in size from $1/4$ in. to 5 in. in length. The retention time in the kiln was about 15 minutes. The larger pellets were not completely dry when they emerged from the kiln, which indicated insufficient retention time. A kiln with a longer retention time would be required to dry and calcine wet sludge.

Calcination of Pellets from Precipitated Sludge (Small Scale)

Sludge containing approximately 40% water was dried to about 5 % and pulverized. Pelletizing was done in the Lurgi disc using an 8% solution of lignin sulphonate. The moisture content of the pellets being 27%, the lignin content was 2.16% or 43.2 lb per ton of calcium carbonate. Green and dry strengths of the pellets were over 30.

The green pellets were fed to the 5 ft by 5 in. rotary kiln at a temperature of 1800°F, and shattered when they encountered the hot zone of the kiln. Pellets were then dried and fed to the same kiln. As they could only be retained in the kiln for 5 minutes, they were passed through 6 times for a total retention time of 30 minutes. Little evidence of flaking or crumbling was noticed. Dried pellets were also fired in the gas-fired muffle kiln for 45 minutes at 1800°F. The products from both kilns were slaked in near boiling water.

Assay results of the sludge, raw and calcined pellets are shown in Table 5.

TABLE 5
Assay of Calcining Tests on Pellets

Product	Assay %			
	Na	SO ₄	Ca	CaO
Sludge	2.08	1.11	35.6	-
CaCO ₃ pellets	2.50	1.64	35.2	-
Rotary kiln calcined pellets	3.44	2.66	-	84.5
Muffle kiln calcined pellets	3.80	2.61	-	83.4

The calcined pellets produced in both the rotary and stationary kilns were slaked and used in the neutralization of bicarbonate in Beaver lodge pregnant solution. Neutralization efficiencies were approximately 85%, which compared favourably with commercially produced lime and lime hydrate.

Calcination of Pellets (Large Scale)

A one-hundred-pound batch of calcium carbonate sludge was dried and pelletized in the Lurgi disc pelletizer. The dried pellets contained approximately 1% lignin sulphonate as a binder. They were fed into the 12 in. by 12 ft rotary kiln. The firing conditions were as follows:

Feed rate	- 25 lb/hr
Kiln speed	- 1.2 r p m
Kiln lining temperature	- 1780°F
Bed temperature	- 1820°F
Retention time	- 30 min

The pellets retained their strength and shapes during the calcination, and no build-up of material around the wall of the kiln was noted during the four-hour test period. The results of the calcination and subsequent slaking tests are shown in Table 6.

TABLE 6

Results of Large Scale Calcination of Pellets

Product	Ca %	CO ₂ %	Na %	SO ₄ %	U ₃ O ₈ %	CaO %
Pellets, CaCO ₃	36.4	37.6	1.68	0.18	0.62	
Calcine, CaO	60.8	2.0	2.46	-	-	89.2
Slaked lime, Ca(OH) ₂	48.6	1.60	2.04	1.05	0.87	

The freshly slaked lime was used in the neutralization of Beaverlodge pregnant solution and yielded an overall neutralizing efficiency of 85%.

Calcination of Spiractor Product

The washed spiractor product was calcined in the 12 ft kiln at a temperature initially of 1830°F and a retention time of 15 min. Reactivity of the resulting lime was poor, due to the low retention time,

but was increased considerably when the temperature was raised to 2150°F. At this higher temperature the material began to form rings around the inside periphery of the kiln. This build-up became so severe that the kiln was shut down. Apparently this type of feed to a rotary kiln would create a ringing problem which would be difficult to overcome.

Discussion of Results

The space available for the calcining equipment at the Beaverlodge mill is limited. This imposes restrictions on the selection of a rotary kiln for this application. The test made in the 12 ft kiln showed that the larger pellets which formed in the kiln were not completely dry and any calcination was very slight.

The spiractor precipitate which was fired in the 12 ft kiln showed a marked tendency to build up on the wall of the kiln. Even with a rotary kiln equipped with a chain, this would create a serious problem in a commercial operation using a relatively small diameter kiln.

Of the binders which were tested, lignin sulphonate appears to be the most satisfactory. It imparts strength to pellets in both the green and dry states, whereas the others gave increased strengths in the green state only. Of the two lignin sulphonates tested, the lignosol TSD gave slightly higher strengths at lower concentration, but either the TSD or the BD type was quite satisfactory.

The granular spiractor precipitate was too coarse to be made into pellets of sufficient strength to withstand the handling to which they would be subjected. This precipitate would have to be pulverized before it could be satisfactorily pelletized.

As with the granular spiractor product, pellets made by spraying a CaCO_3 slurry onto dry material did not result in pellets of sufficient strength in the dry state.

Extrusion of the sludge did not show any promise, as the amount of water required was so high that the extruded streams would not support their own weight.

Satisfactory pellets could be produced from pulverized material, using a lignin sulphonate binder. A concentration of 1% binder was adequate to impart sufficient strength to the pellets. The pellets would shatter if fed into a kiln where the exhaust gases were 800°F or higher. A retention time of 30 minutes in a kiln having a maximum temperature of 1800°F resulted in a product having a neutralizing efficiency of 85%.

A spouted bed drier appeared to dry the spiractor precipitate satisfactorily, but the efficiency of the burner and dust collecting systems was not sufficiently high for a practical application of this method.

To sum up the preceding discussions, it appears that the calcium carbonate will have to be dried, pulverized, and pelletized

with a lignin sulphonate binder. It can then be calcined satisfactorily in a rotary kiln having a maximum temperature of 1800°F and giving a retention time of about 30 minutes. It is necessary that the kiln be of sufficient length to maintain exhaust gases at a temperature of less than 800°F.

FluoSolids Reactor

Introduction

Three calcination tests were run on Beaverlodge calcium carbonate sludge by the Dorr-Oliver Company, using their FluoSolids roaster at Westport, Connecticut, U. S. A. The sludge was formed by direct precipitation with lime slurry, which resulted in the particles being mainly minus 10 microns. Sodium carbonate in the sludge was 4 to 5%. By water washing and filtering, samples were obtained containing less than 1% sodium carbonate on a dry weight basis. These samples were calcined in the FluoSolids roaster under conditions shown in Table 7.

TABLE 7

Summary of FluoSolids Test Conditions

Test No.	% Na ₂ CO ₃ in Dry Sludge Feed	% Total Na in Dry Sludge Feed	Seed Rate, % Wt. of Total Feed	Temp., °F	Air Rate, Scfm
1	0.75	0.58	10	1540	5.6
2	0.15	0.17	15	1635	5.0
3	0.66	0.37	20	1635	5.0

All tests were unsuccessful because defluidization of the bed occurred.

Apparatus

A cross-sectional view of the laboratory FluoSolids reactor used in these tests is shown in Fig. 2. It consists of a 4 in. inside diameter stainless steel tube 9 ft long mounted vertically inside an insulating firebrick chimney. The tube is heated externally by hot combustion gas from four Maxon burners supplied by a ring manifold as shown, the object being to bring the unit up to operating temperature, to hold the fluidized bed at reaction temperatures, and to offset radiation losses.

Half the fluidizing air is supplied through a hose connection passing into the bottom of the reactor through a perforated conical bottom plate. The remainder of the fluidizing air and gas, for conveying feed, passes through a pipe extending upward 12 in. into the fluid bed. This pipe is equipped with a ball check to prevent runback of solids.

Feed is metered by means of a pressurized screw feeder, and is picked up by the fluidizing air and gas mixture and conveyed through the feed pipe into the reactor.

The air and gas are metered by a Rotameter mounted on a panel board, which also carries manometers connected to pressure taps and a pyrometer connected through a multi-point switch to thermocouples as shown in Fig. 2.

The reacted material is discharged partly as underflow

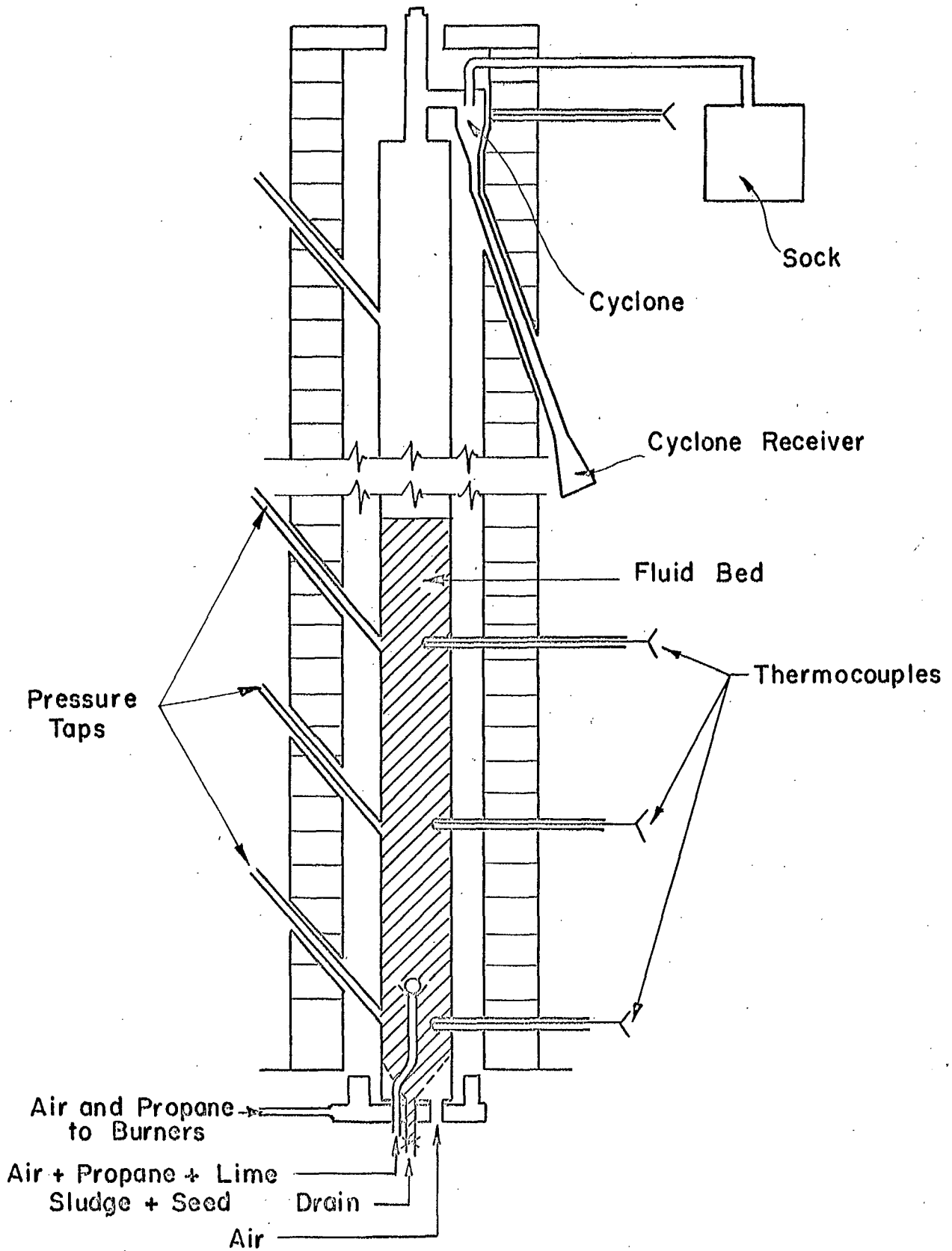


Figure 2 Dorr Oliver 4" Fluo Solids Reactor

through a discharge pipe located at the bottom of the conical bottom and partly as "carryover" which is collected in a dust collection system consisting of a hot cyclone mounted on top of the reactor and within the brick chimney and a woven glass wool sock (see Fig. 2). The underflow and cyclone products are collected periodically during the test. The bottom plate which carries the air and gas intake and the underflow connection is attached by a flanged connection to the vertical reactor tube, so that the whole assembly can be removed for cleaning and inspection.

Procedure

The sludge as received contained approximately 4 to 5% Na_2CO_3 . Since Na_2CO_3 in excess of 1% in the sludge usually causes excessive pelletization and defluidization, the sludge was water-washed and filtered to lower the content.

Dried sludge containing 0.75% Na_2CO_3 (0.58% Na) was mixed with 10% by weight of calcium carbonate seed pellets for feed for Test No. 1.

Dry sludge analysing 0.15% Na_2CO_3 (0.17% total Na) was mixed with 15% seed pellets for feed for Test No. 2.

For Test No. 3 nearly all soluble sodium compounds were removed by washing (0.03% total sodium in dry washed sludge). After filtering and drying, sodium carbonate sufficient to give 0.85% Na_2CO_3

in the final sludge was added. This sludge was mixed with 20% seed pellets for feed to the reactor.

Chemical analyses of the three feed charges are shown in Table 8.

Three FluoSolids tests were made using sludge with Na_2CO_3 contents ranging from 0.15% to 0.75% (0.17% to 0.58% total Na) and seed additions from 10 to 20% (Table 7). In each test, a starting bed of seed pellets was put in the reactor, fluidized with air, and heated to a temperature of about 1600°F. Propane and feed were started. Temperatures, feed rate, and air and gas rates were adjusted to the required amounts. Proper bed level was maintained by withdrawing the underflow product periodically. After displacing the starting bed material once, a formal test was started. Operating conditions of the three tests are given in Table 9.

Observations

The dry sludge feed for Test No. 1 analysed 0.75% Na_2CO_3 and 0.58% total sodium. The 0.75 Na_2CO_3 accounts for 0.326% Na, leaving 0.254% Na which is probably present as sodium sulphate and minor amounts of sodium bicarbonate or caustic. In this test, defluidization occurred before a formal test could be started. The defluidized bed consisted of large lumps of pellets and fine dust. Scale had built up on the reactor walls to a thickness of 1/2 in. Pellets that were produced were soft and easily broken.

In Test No. 2 the sludge was washed so as to give a dried

TABLE 8

Chemical Analyses for FluoSolids Reactor Tests

	Tot. CaO	Avail. CaO	*Na ₂ CO ₃	MgO	SiO ₂	LOI	**Tot. Na	NaHCO ₂
Feed to Test #1	47.9	0.70	0.75	1.1	0.43	44.8	0.58	-
Feed to Test #2	51.3	0.47	0.15	-	0.21	44.5	0.17	0.46
Feed to Test #3	-	-	0.66	-	-	-	0.37	0.12
Test #1 Underflow	-	75.8	-	-	-	9.6	-	-
Test #1 Bed	-	84.3	-	-	-	3.7	-	-
Test #2 Underflow	-	89.7	-	-	-	1.5	-	-
Test #3 Underflow	89.1	81.2	-	-	-	-	-	-
Test #3 Bed	89.1	81.2	-	-	-	-	-	-

*Determined by titration of water leach filtrate.

**Determined by flame photometer.

TABLE 9

Operating Conditions for FluoSolids Reactor Tests

Test Number	1	2	3
*% Na ₂ CO ₃ in dry sludge	0.75	0.15	0.66
**% Total Na in dry sludge	0.58	0.17	0.37
% Seed in feed	10.0	15.0	20.0
Air rate, scfm	5.6	5.0	5.0
C ₃ H ₈ rate, scfm	0.22	0.19	0.19
Temperature, °F	1540	1635	1635
Feed rate, gm/min	97.5	83.5	83.5
Space rate, ft-s	4.75	4.75	4.75
Bed depth, ft	3.0	3.0	3.0

*Determined by titration of water leach filtrate.

**Determined by flame photometer.

TABLE 10

Product Distribution for Test Number 3

Product	Wt g	% Wt
Prerun underflow	5410	83.2
Prerun cyclone	970	14.9
Prerun sock	130	1.9
Formal underflow	2170	83.3
Formal cyclone	375	14.4
Formal sock	60	2.3

feed assaying 0.17% total sodium. In Test 2 the bed defluidized after 3 hr operation and before a formal test could be started. The defluidized bed consisted of soft large lumps of pellets and dust. A thin scale of 1/16 in. to 1/8 in. formed on the reactor walls. Although this test was run at 1635°F, the pellets were also quite soft.

It was thought that other soluble sodium compounds (Na_2SO_4 , NaHCO_3 or NaOH) might have been responsible for soft pellets and defluidization. Consequently, for Test No. 3 the sludge was thoroughly washed to remove nearly all the soluble sodium salts. The total sodium assay on the dried sludge was 0.03%. Fresh soda ash was added to give 0.85% Na_2CO_3 in the sample. Seed rate was increased to 20% in an attempt to produce smaller pellets and thereby decrease the possibility of defluidization. This test was run for 3 1/2 hr before defluidization occurred. The pellets were slightly harder than those in Tests 1 and 2 but not as hard and strong as those produced from water softening of paper-mill lime mud. Reactor wall scale of 1/16 in. to 1/8 in. was formed.

Discussion of Results

In Test 1 and 2 the bed defluidized before a formal test could be started. Only samples of underflow products taken shortly before defluidization occurred were analyzed for available CaO. A formal run of approximately 1 hr duration was made in Test No. 3 before defluidization occurred. Carryover on the formal part of the test

was approximately 17% of the gross production. Product analyses are shown along with the feed assays in Table 2. Product distribution of Test No. 3 is shown in Table 10.

Vertical Kiln

Introduction

A standard method of producing lime from limestone is calcination in a vertical shaft kiln, where heat is applied near the bottom of the shaft and the calcined product is periodically withdrawn from the bottom. In the investigation of this process as a possibility for calcination of sludge pellets, it was recognized that generally shaft kilns were used for treating limestone of around 8 in. diameter and that difficulty might be encountered in getting sufficient hot gases through the kiln. However, it was thought that the relative simplicity and low capital cost of this type of furnace warranted some preliminary test-work. A pilot shaft kiln was built and equipped with an induced draft fan at the top. Beaverlodge sludge was pelletized and the pellets charged to the shaft kiln. Even under induced draft, it was difficult to get sufficient gas through the bed for proper calcination. The pellets abraded during their movement and filled the interstices. The calcines that were produced were dead-burned because of technical difficulties in the flow of the material through the kiln.

Apparatus

Static load tests on dried pellets indicated that they could withstand a pressure equivalent to 25 vertical feet of pellets. A

vertical shaft was built with K-28 insulating firebrick. It was 21 ft in height with an internal cross section of 4 1/2 in. by 4 1/2 in. A sketch of the shaft kiln is shown in Fig. 3. Wall thickness was 4 1/2 in. and the walls were sheathed in asbestos board. Firing ports were located on each side of the kiln, 3 ft from the bottom. Propane burners were located at each port. Temperature thermocouples were located at 1 ft intervals above the firing ports up to 6 ft and at 6 ft intervals thereafter. A fan and an air aspirator were used as alternative methods for providing draft at the top of the furnace. Calcines were discharged from the bottom of the kiln through a movable steel plate.

Procedure and Observations

Feed to the shaft kiln consisted of CaCO_3 pellets ranging in size from 1/2 in. to 3/4 in. The pellets containing 0.94% Na were made in a Lurgi pelletizer from fine calcium carbonate precipitate from Beaverlodge bound with lignin sulphonate solution. The resultant green and dry drop strengths were 25 and 17, respectively. Sufficient pellets were dried at 110°C to load the kiln 12 ft above the firing ports. The loading capacity was found to be 6 2/3 lb per vertical ft. Six feet of wet pellets was added on top of this charge.

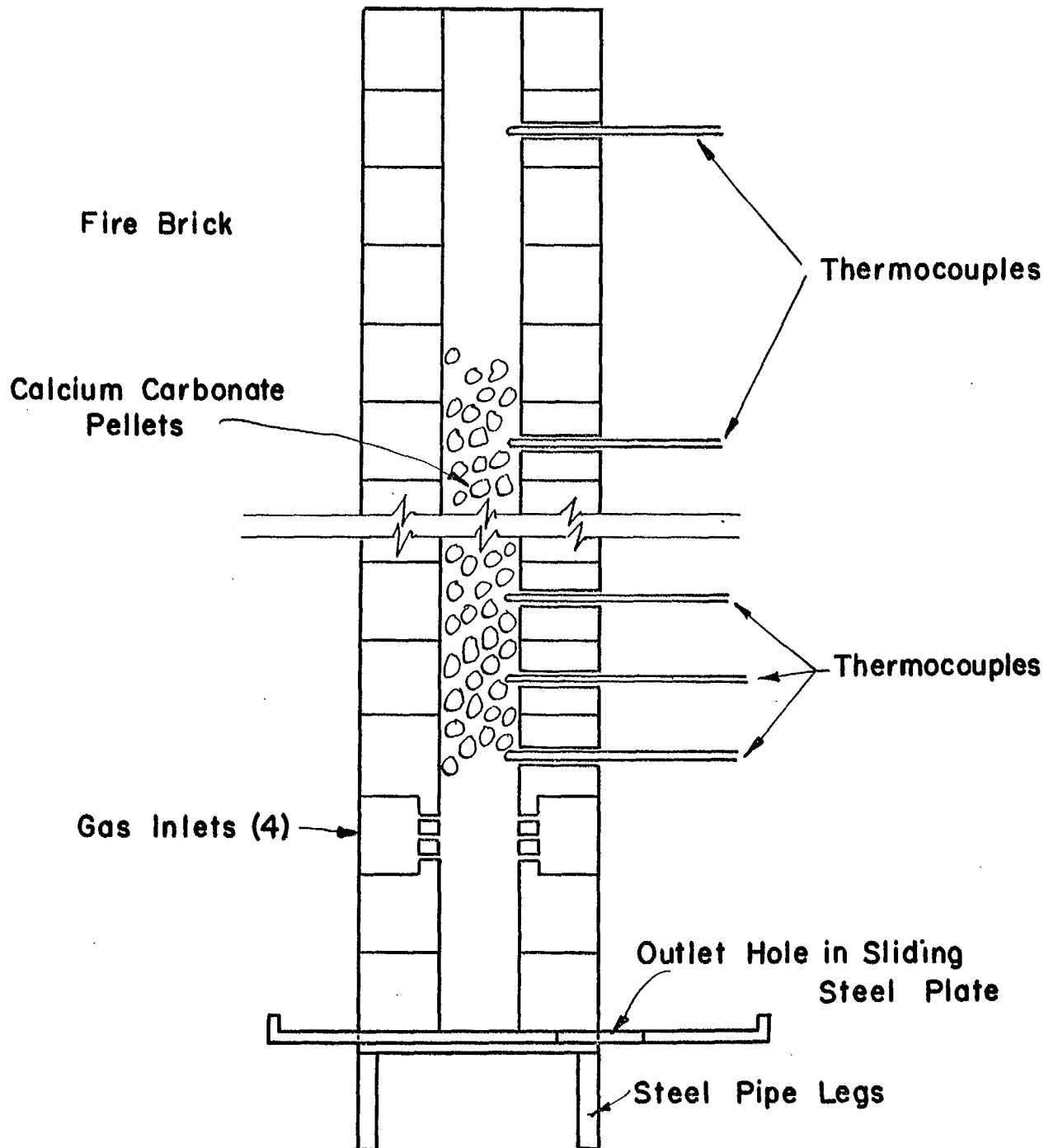


Figure 3 Shaft Kiln for Sludge Pellet Calcinations.

The pellets were first fired at over 2000°F, but heat penetration was poor. It was possible to attain a steady temperature one ft above the firing ports of only 750°F. However, the pellets at over 2000°F were quite stable and retained their shape after firing. The bed was lowered by 6 ft and refired. Heat penetration was improved somewhat, but the steady temperature attained 1 ft above the port was still only 1250°F.

Some bridging was noticed when attempts were made to drop the bed another 6 ft. This was probably due to the wet pellets agglomerating under pressure and slow drying conditions. Firing 6 ft of pellets resulted in a steady temperature of 1500°F 1 ft above the ports.

Exit gas flows were estimated by a combination of the Hagen-Poiseuille Law and the Kozeny Equation. With a 12 ft bed, the gas flow was calculated to be 18.6 scfm per sq ft surface area. At 6 ft bed depth, the gas flow was 40.1 scfm per sq ft surface area. These gas flows under induced draft were too low to allow proper heat distribution through the bed. The reactivity of the lime produced was poor, due to dead burning. Examination of the bed after testing showed that the pellets had been abraded and the resultant fines had filled the interstices between pellets.

Discussion of Results

Calcination of calcium carbonate pellets in a shaft furnace proved unsuccessful under the conditions used, because of poor heat penetration through the bed. Some consideration was given to pressure firing of the pellets. However, information from Surface Combustion Co. Ltd. of Toledo, Ohio, U.S.A., who are builders of pressure-fired shaft furnaces, indicated that a major problem of dead burning would be encountered in producing good quality lime by this method. The main problem is that the reaction is endothermic and an extremely high temperature is required at the firing ports for sufficient heat to penetrate the bed. Also, because of the relatively small size of the furnace feed, the pressure drop per foot of depth is considerable and increases with increased depth.

A shallow furnace would be more readily penetrated by hot gases, but heating efficiency would decrease. Further test work on the shaft kiln was postponed and test work on other calcination methods was started.

Travelling-Hearth Tunnel Kiln

Introduction

Several conventional types of calcining furnaces have been tested for regenerating lime for the Beaverlodge carbonate circuit. An unusual type of calciner considered was a travelling-hearth tunnel kiln. This unit is a high temperature doughnut-shaped furnace into which a thin bed of calcium carbonate pellets can be charged. The

pellets are heated by radiation and reflection from gas or oil burners. The hearth is rotated during the calcination period and the calcines are removed from the bed by a continuous scraper. The results of test work on Beaverlodge sludge showed that this type of furnace can produce lime without dead burning.

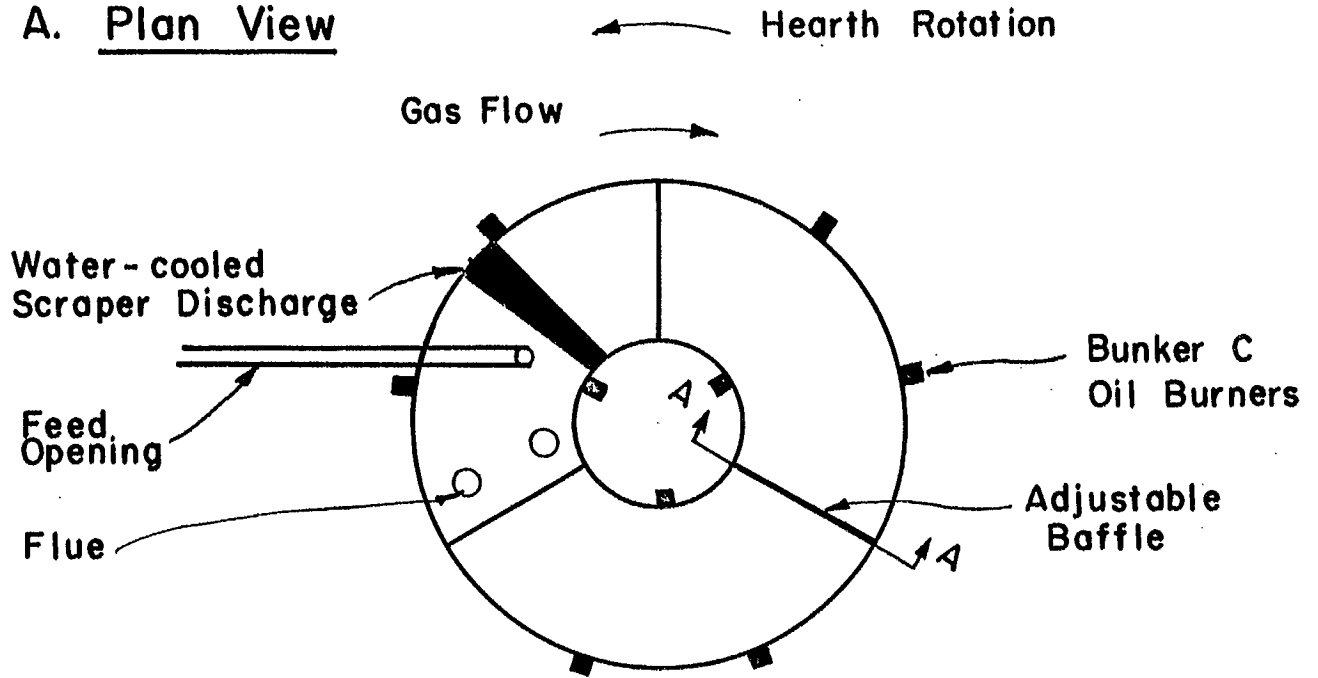
Two series of tests were carried out in muffle furnaces to determine the effectiveness of this process and the size of furnace required to yield 7 1/2 tons of lime per day. Over 90% conversion of CaCO_3 to CaO was obtained at temperatures of 2200-2400°F with retention times of less than 15 min. No evidence of dead burning was noticed. A kiln size of 18 ft 4 in. outer diameter with a 3 ft 6 in. hearth was recommended for producing 7 1/2 tons of lime per day. Also recommended was a feed hopper above the furnace into which the furnace discharge gases could be exhausted to dry and preheat the pellets.

The main disadvantages of using the travelling hearth tunnel kiln were the relatively high Btu consumption (10 million Btu per ton CaO estimated) and the necessity of making pellets to feed the kiln.

Apparatus

A sketch of the furnace is shown in Fig. 4. The unit is doughnut-shaped, the hole diameter usually being 1/3 the overall diameter. The hearth bottom is of insulating firebrick and the top

A. Plan View



B. Section AA

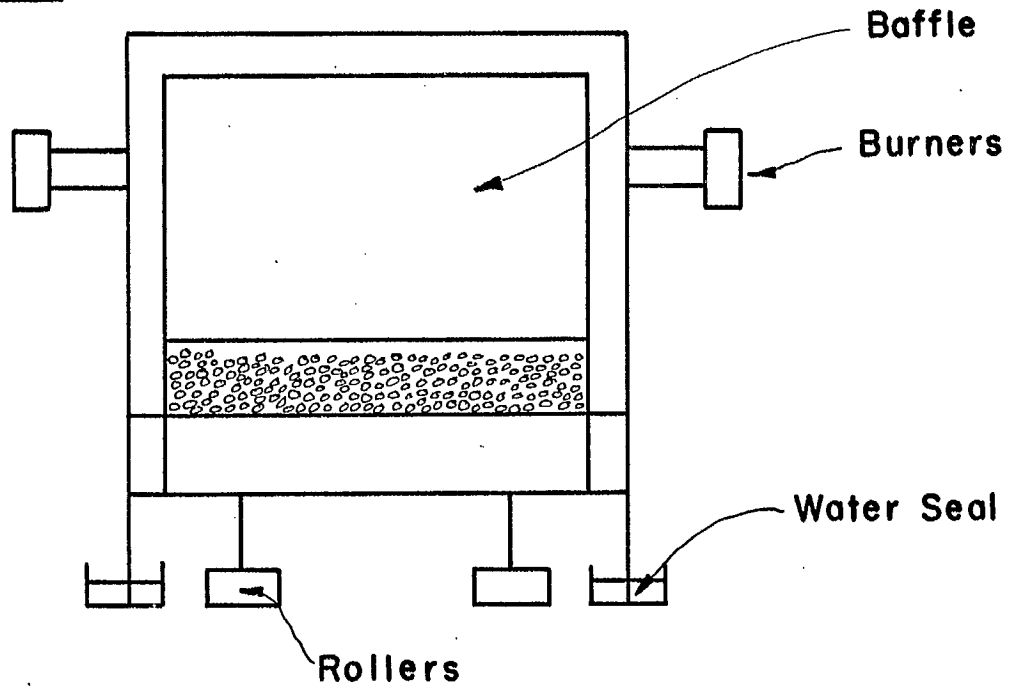


Figure 4 Travelling Hearth Kiln

of the hearth is castable alumina (70% Al_2O_3). The walls and roof are all insulating firebrick. The hearth rotates on rollers and the whole system is kept gas-tight by water seals. The furnace can be heated either by gas or oil. Hot gases travel counter current to the rotation of the hearth by variation of baffle height between each chamber. Thus the deepest baffle would be near the feed section of the kiln and the shallowest at the discharge end. Since the unit is air-tight, all gases must pass under the baffles. For test work gas-fired muffle furnaces were used to simulate one section of the kiln hearth.

Procedure

Feed less than 10 mesh in size was not recommended for this type of furnace, because the velocity of gases as they pass beneath the baffles could lift the charge from the hearth and cause heavy dusting. Since calcium carbonate sludges from Beaverlodge were all minus 10 mesh, it was necessary to make pellets to feed the furnace. The CaCO_3 pellets, containing approximately 1% Na, were made in a Lurgi pelletizer from pulverized spiractor product bound with 5% lignin sulphonate solution. The pellets ranged from 1/2 to 3/4 in. in diameter and had a dry drop strength of 16.

In the first series of tests, dried pellets were loaded onto a 70% alumina sagger in a thin bed of 1 1/2 pellets depth. The sagger measured 7 in. by 12 in. and the pellets charge was equivalent to 4.5 psf. The loaded sagger was heated in a gas-fired

muffle kiln. Calcination temperatures were varied from 2000°F to 2200°F. Separate charges of pellets were fired at each temperature for 5, 10, 15 and 25 minutes. After each firing, the pellets were removed, cooled, and sampled for CO₂ analysis. Samples were also slaked to determine whether any dead burning of the lime occurred.

In a second series of calcination tests, a gas-fired muffle kiln with 8 square feet of hearth area was used to calcine pellets at temperatures of 2300°F and 2400°F. Pellets were loaded into the kiln at 3.1 psf of hearth area.

Calcination tests were made at 2300°F for retention times of 10, 12 1/2 and 15 minutes. Another test was made at 2400°F for 7 1/2, 10 and 12 1/2 minutes. In a special test, crushed pellets were loaded on a high alumina castable sagger and calcined at 2400°F for 60 minutes to determine whether any undesirable reaction of the lime with alumina occurred.

Results

The results of calcination tests are shown graphically in Fig. 5 and 6. As noted in Fig. 5, over 90% conversion of CaCO₃ to lime was accomplished in 15 minutes at 2200°F. No evidence of dead burning was encountered on slaking the calcines. The slaking rate was increased considerably by crushing the calcined pellets to fines. No adhesion of the pellets to the alumina hearth was observed.

Increasing the temperature of the kiln from 2200°F to 2300°F

% CO₂ vs TIME
 - 3/4" + 1/2" SIZE

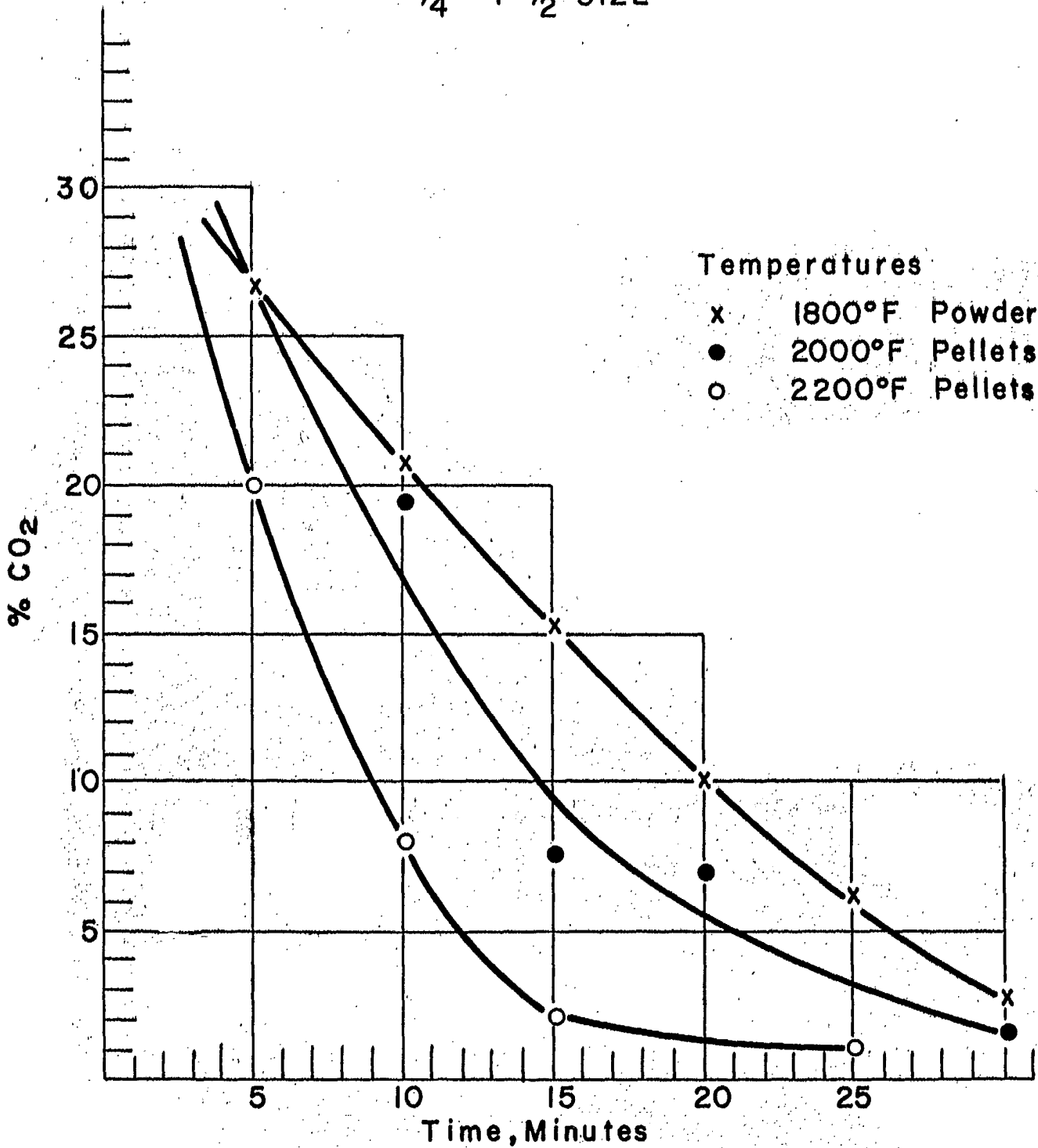


Figure 5 Calcination of Calcium Carbonate Pellets

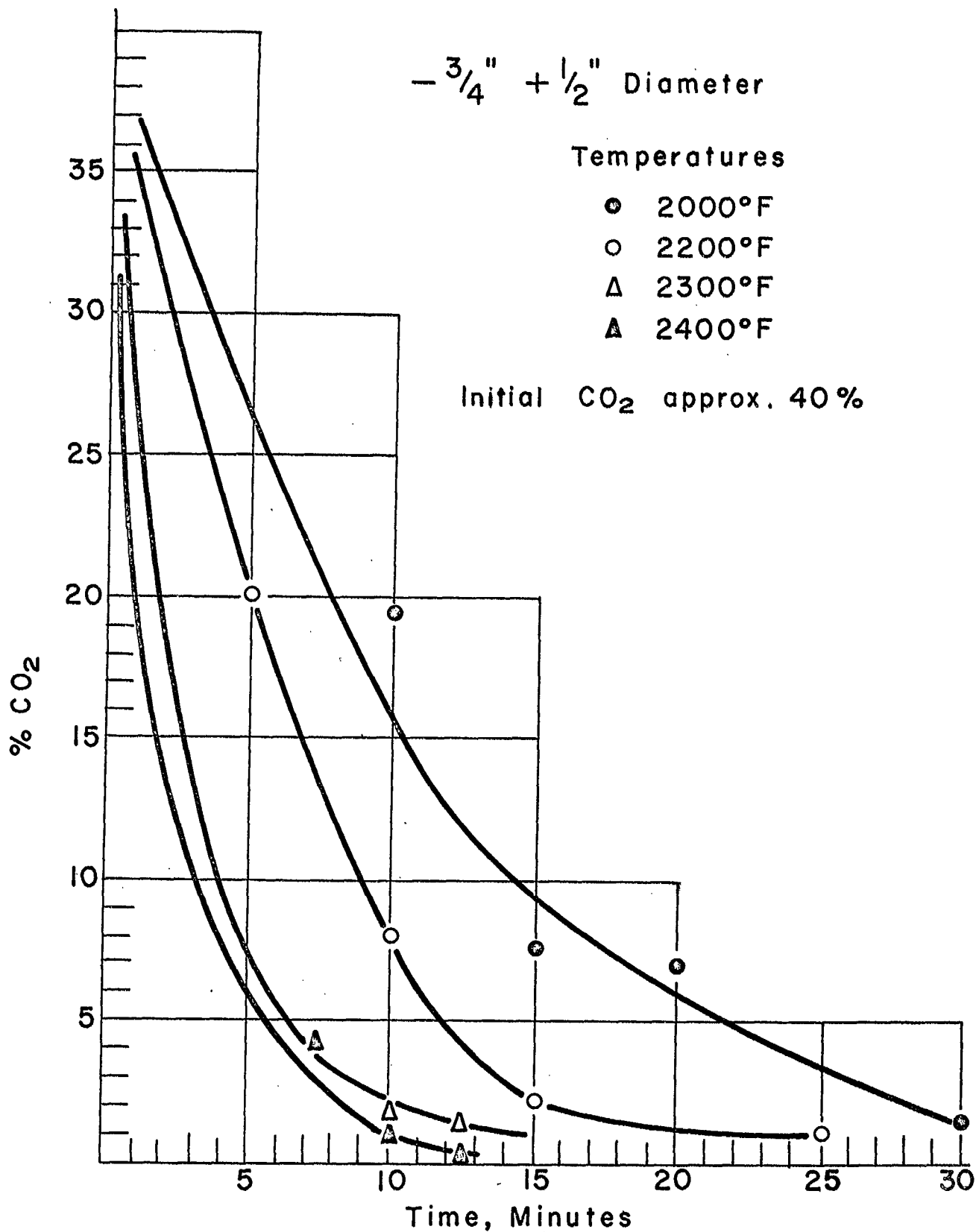


Figure 6 Calcination of Calcium Carbonate Pellets

gave a significant increase in the rate of calcination, as shown in Fig. 6. However, a further increase to 2400°F did not yield a comparable increase in the rate. The detailed results of the calcination tests at temperatures of 2300 and 2400°F are shown in Table 11. No evidence of reaction of the lime with castable alumina was noted after calcining at 2400°F for over an hour. All calcines slaked readily after 2 to 3 minutes at 180°F.

Discussion of Results

The principle of the travelling-hearth tunnel kiln for lime calcination is to heat rapidly by radiation and reflection a thin bed of pellets. To ensure that complete calcination takes place, sufficient hearth bed must remain exposed so that the bottom of the charge is also heated. The designers of the furnace have from the results of extensive tests, decided that an average bed depth of 1 1/2 pellets or stones would be the most desirable rate of charging this unit. The main advantages of this type of furnace appeared to be lower capital cost, compactness, and simple operating control. This last advantage was evident, since only the speed of the hearth is controlled and temperatures are automatically regulated.

The main disadvantages of this type of calciner in regenerating sludge were the higher Btu requirements for lime production and the necessity of making pellets to feed the furnace. The pelletizing step requires considerable capital cost for equipment along with the increased

operating costs of sludge drying and binding agents.

TABLE 11
Results of Calcining Tests on Beaverlodge Pellets
at Elevated Temperatures

Pellet Charge, psf Hearth	Temp., °F	Time, Min.	Calcine Yield, psf Hearth	Calcines		
				% CaO	% CO ₂	% Na
3.1	2300	10	1.9	88.7	1.70	1.44
3.1	2300	12 1/2	1.8	89.0	1.40	1.52
3.1	2300	15	1.8	89.5	1.34	1.49
3.1 Wet pellets*	2300	20	-	90.0	1.30	1.26
3.1	2400	7 1/2	1.9	85.6	4.03	1.52
3.1	2400	10	1.8	89.8	1.05	1.34
3.1	2400	12 1/2	1.8	90.5	0.43	1.49
Crushed pellets in sagger	2400	70		90.6	N. D.	0.85

*Decrepitated to fine flakes

N. D. - Non-detectable.

Herreshoff Roaster

Introduction

A Herreshoff roaster owned by the Mines Branch was used to determine the feasibility of calcining wet pellets prepared from un-

washed sludge. The wet sludge was tested in a Herreshoff roaster at the Nichols Engineering Laboratory at Netcong, New Jersey.

Mines Branch Roaster

Apparatus and Procedure

An 18-in. glo-bar heated single-hearth Herreshoff furnace with two rabble arms 180 deg apart was used at the Mines Branch for the initial test work.

Initially, 5 lb of wet pellets having an average drop strength of 15 and containing 18.9 % water after being bonded with a 3 1/2 % solution of B D lignin sulphonate were fed into the furnace. The initial furnace temperature was 450°F and this was raised gradually to 710°F in 8 1/2 min. Because of excessive decrepitation the furnace was then turned off and cleaned out.

The furnace was then maintained at a temperature of 450°F and fed with 50 lb of similar pellets banded with a 3 1/2 % solution of B D lignin sulphonate. After 10 minutes the furnace was turned off and allowed to cool. The resulting material was then discharged and fed back to the furnace mentioned at a constant temperature of 1800°F. Samples were removed after 17 and 22 min. Similar drying and calcining tests were carried out on 50 lb samples of wet pellets having the following properties: (a) an average moisture content of 18.4 % after using a 7 % B D solution as a binder, and a drop strength of plus 30; and (b) a 3 1/2 %

TSD lignin sulphonate solution as a binder, an average moisture content of 19.5%, and a drop strength of plus 30.

Observations and Results

With the initial 5 lb sample of pellets, furnace temperatures of over 500°F resulted in the decrepitation and shattering of the pellets.

The approximate percentage of fines from the dried 50 lb samples originally containing 3 1/2% BD, 7% and 3 1/2% TSD samples were 35, 85, and 85% respectively. These fines were caused by the action of the rabble teeth rather than by decrepitation.

Because of these excessive fines, samples containing 7% BD and 3 1/2% TSD were not calcined. Clinkers, comprising 10 to 20% of the product and up to 2 in. in size, were formed during the calcination of the sample containing 3 1/2% BD. The rabble arms were coated with the sludge. Assay results of the two samples taken are shown in Table 12.

TABLE 12

Calcination of Calcium Carbonate Pellets in Herreshoff Furnace

Product	% CaO	% CO ₂	% SO ₄	% Na	% U ₃ O ₈	L. O. I.
17-minute sample	69.5	12.2	6.1	7.95	0.56	11.9
22-minute sample	72.8	11.8	6.0	7.75	0.56	11.4

Nichols Engineering Roaster

Apparatus and Procedure

The sludges, as described in Table 1, were washed to yield the following feed products:

Sample 1	-	coarse spiractor product	-	% Na	-	1.10
				% U ₃ O ₈	-	0.084
Sample 2	-	fine spiractor product	-	% Na	-	1.48
				% U ₃ O ₈	-	0.093
Sample 3	-	fine spiractor product	-	% Na	-	1.77
				% U ₃ O ₈	-	0.17

Samples 1 and 2 were combined at 57 and 43 % dry weight basis, respectively, for calcination tests.

The apparatus consisted of a gas-fired, single-hearth 18-in. Herreshoff furnace having a rabble blade spacing of approximately 2 in. Three rabble blades were on one arm and two blades on the other. The rabble speed was 4 rpm.

The furnace was heated to 1800°F and charged with approximately 18 lb of wet sludge. The charge lowered the furnace temperature and the start of calcination was taken as the time when the temperature of the charged furnace reached 1665°F.

Two runs were made. In Run No. 1, a combination of coarse and fine spiractor material (sample 1 and sample 2) similar to what is expected from the Beaverlodge operation was fed to the furnace. For Run No. 2, fine spiractor sludge with a sodium content of 1.77 % (sample 3) was calcined in the Herreshoff furnace to

determine the effect of the higher sodium content.

Observations and Results

The results of the tests are shown in Tables 13 and 14 and the calcination rates are indicated graphically in Figs. 7 and 8. The calcines from both tests had the consistency of granulated sugar. Although dust losses were not measured, the flue gas discharged during calcination appeared to be relatively clean. Both calcines slaked slowly at near boiling temperature. The higher sodium fines slaked slower than the lower sodium coarse and fine material. The black residue was observed after slaking to have the appearance of fine ore particles.

TABLE 13

Calcination of Coarse and Fine Spiractor Calcium Carbonate Composite

Calcination Time (minutes)	Temp., °F	Calcine Assay %				% CaO (1)	Calcination Efficiency % (2)
		CO ₂	Ca	Na	U ₃ O ₈		
0	-	41.3	35.3	1.27	0.088	-	-
16	1800	4.26	60.2	1.73	0.17	80	89.2
33	1860	2.09	61.5	1.85	0.17	85.3	94.8
63	1840	1.84	62.0	1.79	0.17	85.9	95.5
78	1850	1.29	62.0	1.79	0.17	87.0	96.9

Charge: 9.0 lb spiractor coarse containing 21.8% H₂O
 9.0 lb spiractor fines containing 41.5% H₂O
 Sludge approximately 90% CaCO₃

(1) Calculated by CaCO₃ purity x calcination efficiency.

(2) Calculated by % CO₂ removed.

TABLE 14

Calcination of Fine Spiractor Calcium Carbonate

Calcination Time (min)	Temp., °F	Calcine Assay %				% CaO	Calcination Efficiency %
		CO ₂	Ca	Na	U ₃ O ₈		
0	-	38.7	36.0	1.77	0.17	-	-
22	1780	2.73	60.4	2.0	0.23	84.1	93.5
37	1820	1.43	60.4	2.67	0.24	86.9	96.6
52	1810	1.41	61.1	2.83	0.24	87.0	96.7
67	1800	1.15	61.1	2.59	0.24	87.5	97.3

Charge: 18 lb sludge containing 41.5% H₂O.

Sludge approximately 90% CaCO₃.

Discussion of Results

Because of the lack of a temperature gradient the single-hearth Herreshoff furnace is not the proper equipment for conducting calcination tests on wet pellets. A multiple-hearth Herreshoff furnace, which would allow the pellets to dry before entering the hot zones, would be unquestionably preferable.

However, on a laboratory scale this single-hearth furnace is suitable for the calcination of wet sludge, as indicated by the foregoing tests. A high recovery of a desirable product was obtained without the added cost of drying, pulverizing and pelletizing and without the need for more than one hearth. A multiple hearth would

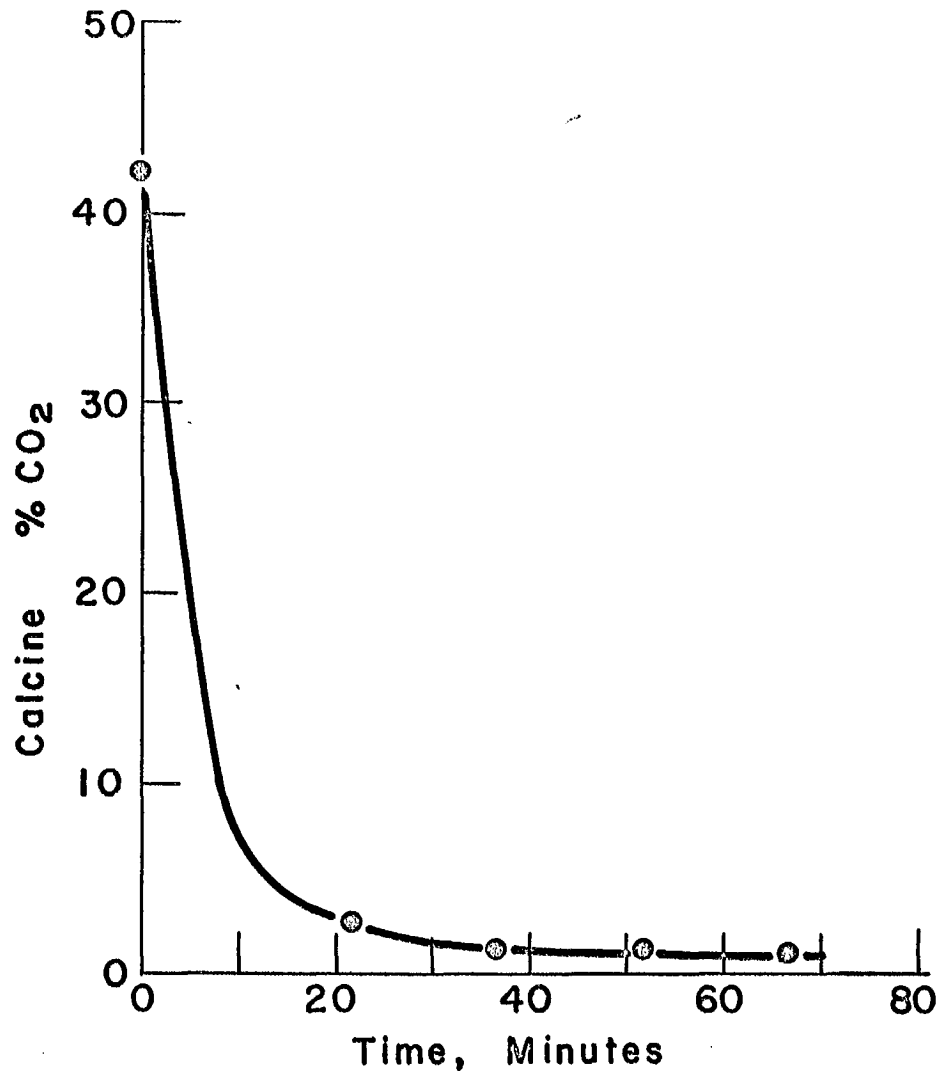


Figure 7 Calcination Rate of
Beaverlodge Coarse and Fine
Sludge Composite

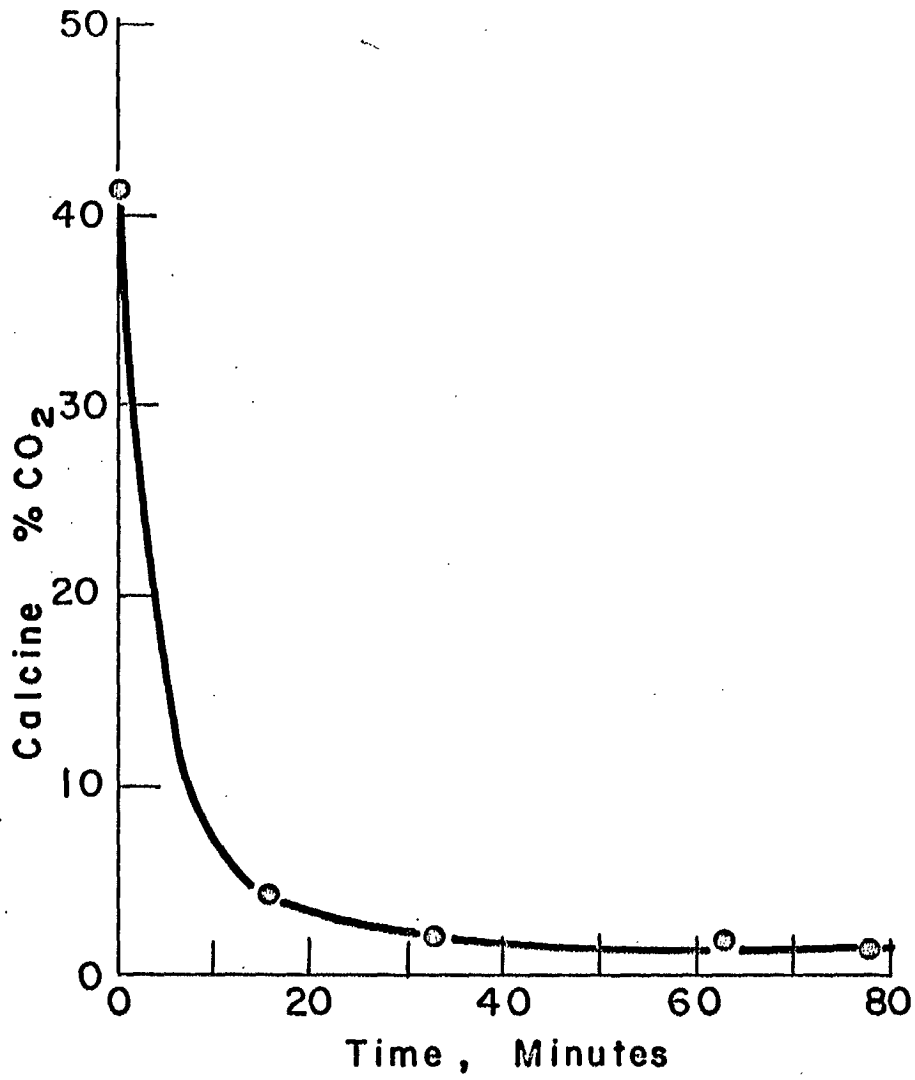


Figure 8 Calcination Rate of
Beaverlodge Fine Sludge

reduce overburning and the loss of fines to a minimum, as well as increase the rated capacity of the unit. The size of a five-hearth Herreshoff furnace required to produce a rated output of 7 1/2 tpd of lime would be 14 ft 3 in. outside diameter and 22 ft high. This space requirement comes well within that available.

DISCUSSION

In the Beaverlodge mill, chemical lime is used as a reagent in processing uranium ore. Direct calcination of limestone for such an application involves the dissociation of calcium carbonate to yield economically a product of suitable chemical properties. Normally this is accomplished in either a vertical or rotary lime kiln.

The regeneration of lime from a precipitated calcium carbonate sludge is a separate problem. There are factors to be considered which are not present in burning a limestone. The most important of these are the finely divided particle size, the excess moisture, and the presence of chemical impurities. These have a bearing on the selection of a preferred regeneration process suitable for a particular sludge.

The object of the current investigation is to regenerate lime for use in the uranium recovery process, and consequently a product must be prepared with the required chemical properties. The sludge contains alkali salts as impurities which might affect certain calcination processes, particularly when using a rotary kiln. Past experience

elsewhere has demonstrated that sodium salts aid in the formation of rings and concretions in the kiln. These are costly to remove.

The discussion of results of this investigation may be separated into two parts:

- (a) Technical Considerations.
- (b) Economic Considerations.

Technical Considerations

(1) Rotary Kiln

For an operation of this size, in selecting a rotary kiln for the calcination of a carbonate sludge, the length must be sufficient to decrease temperatures of the exhaust gases to a point where the prepared sludge will dry without shattering. It would be preferable in the rotary kiln process to include equipment for pelletizing the sludge into an acceptable feed. Although it is technically possible to calcine Beaverlodge sludge by means of a rotary kiln, the above factors should be considered in order to avoid unduly high dust losses and consequently recover a high output of lime.

(2) Fluo Solids Reactor

During the investigation it was not found possible with Beaverlodge sludge to obtain a satisfactory fluidized bed for the calcination period. As a consequence this process is considered unsuitable for this application.

(3) Vertical Kiln

Prepared pellet feed formed too impermeable a bed for suitable calcination in a vertical kiln. The resistance of the bed reduced the draft to the point where proper calcination could not be achieved.

(4) Travelling-hearth Tunnel Kiln

Although it was technically possible to calcine the sludge in a travelling-hearth kiln, it was found necessary to prepare the feed by pelletizing.

(5) Herreshoff Roaster

The investigation demonstrated that this unit was suitable for the direct calcination of the carbonate sludge. Although a slightly higher loss of fines occurred, it was not excessive and was only slightly above that experienced in calcining a pelletized feed. The product from this furnace had the necessary chemical activity to be suitable in the uranium recovery process.

Economic Considerations

Installation of the rotary kiln process for the regeneration of lime at Beaverlodge mill would require a high ratio of capital expenditure to daily output, for the following reasons:

- (1) High installed cost of a small-capacity rotary kiln.
- (2) Desirability of installing pelletizing equipment.
- (3) The modifications necessary to the building to house a rotary kiln installation.

From the experience of Azbe and others in the lime industry, it is known that the thermal efficiency of small capacity rotary kilns burning lime is low.

Although the travelling-hearth unit with its pelletizing equipment fits the space requirement imposed by the Beaverlodge operation and the installed capital cost is not unreasonable, the operational cost would be increased by the lower thermal efficiency of this unit and the need to prepare the feed by pelletizing.

A Herreshoff unit of suitable capacity can be installed in the space available. The fuel consumption and capital cost of the equipment are lower than those of the travelling-hearth process. In addition, no pelletizing is required.

CONCLUSION

As a result of this investigation, it is the conclusion of the authors that chemically active lime may be regenerated from calcium carbonate sludges by three calcining processes:

- (1) The rotary kiln.
- (2) The calcimatic furnace to the travelling-hearth tunnel kiln.
- (3) The Herreshoff roasting furnace.

Of these, the authors further conclude that the Herreshoff furnace is the most economical -- both in capital and operating cost -- for the particular application and to suit the conditions prevailing at the Beaverlodge mill of Eldorado Mining and Refining Company.