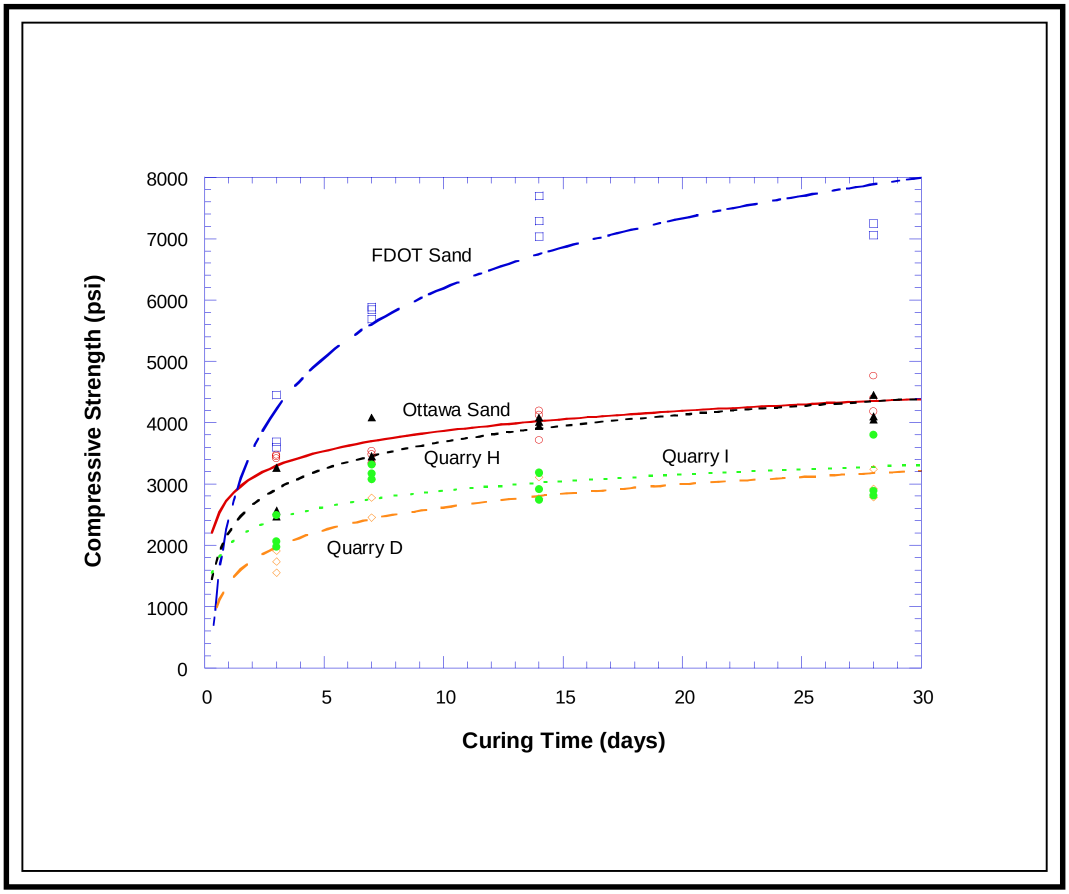
RESEARCH AND TECHNO-ECONOMIC EVALUATION: USES OF LIMESTONE BYPRODUCTS

FINAL REPORT



Department of Geological Sciences
University of Florida

UNITS AND CONVERSION FACTORS

LENGTH

meter (m)	1 m = 3.28083 feet = 39.370 inches = 100 cm = 10^{10} A
centimeter (cm)	1 cm = 10^{-2} m
millimeter (mm)	1 mm = 10^{-3} m = 0.0394 inches
micrometer (μm)	1 μm = 10^{-6} m = 10^{-3} mm = 10^{4} A
nanometer (nm)	1 nm = 10^{-9} m = 10^{-7} cm = 10 A
angstrom (A)	1 A = 10^{-8} cm = 10^{-4} μm = 10^{-1} nm, or 0.1 nm
(inch)	1 inch = 2.54 cm
(foot)	1 foot = 30.48 cm

MASS

gram (g)	1 g = 10-3 kg = 2.205 × 10 -3 lb
kilogram (kg)	1 kg = 1000 g = 2.2046 lb
pound (lb)	1 lb = 0.4536 kg = 453.6 g

VOLUME

liter (l)	1 liter = 1000 cm^3 = 1.0567 quarts (U.S.)
cubic centimeters (cm^3)	
cubic foot (ft^3)	1 ft^3 = 0.02832 m^3 = 28.32 liters = 7.477 gallons
(gallon)	1 gallon = 3.788 liters

TEMPERATURE

degrees Celsius (°C)	5/9 (°F - 32); F = Fahrenheit
kelvins (K)	K = °C + 273.15; C = Celsius; absolute zero = -273.15°C

PRESSURE

(bar)	1 bar = 0.9869 atm = 10^{5} Pa
pascal (Pa)	1 pascal = 10^{-5} bars
atmosphere (atm)	1 atm = 760 mm Hg
1 lb/in^2 (psi)	1 lb/in^2 = 6891 Pa
1 lb/ft^2	1 lb/ft^2 = 47.85 Pa

ENERGY

foot pound (ft·lb)	1 ft·lb = 1.356 J
joule (J)	1 J = 10^{7} ergs = 0.239 cal

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FINAL REPORT

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DEPARTMENT OF GEOLOGICAL SCIENCES
COLLEGE OF LIBERAL ARTS AND SCIENCES
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The opinions, findings, and conclusions expressed in this publication are those of the authors and not necessarily those of the Florida Department of Transportation or the U.S. Department of Transportation. This report does not constitute a standard, specification, or regulation.

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EXECUTIVE SUMMARY

The stockpiling and disposal of byproduct fines produced by the coarse aggregate industry in Florida is one of the most important problems facing the industry today. Both coarse (minus-3/8" by plus-200 mesh) and fine (minus-200 mesh) fractions of byproduct fines represent highly under-utilized resources suitable to applications in the construction market. This is of particular interest to the Florida Department of Transportation (FDOT), as use of these materials in applications such as engineered backfills, direct addition to concrete mixes as filler (minus-200 mesh), and fine aggregate (minus-3/8" by plus-200 mesh) and agglomeration (minus-200 mesh) for use as a manufactured fine aggregate for flowable fills and concrete offer a means by which the life of a major resource in the state may be extended. Furthermore, use of these materials in high volume, technically and economically feasible applications will lead to both economic and environmental benefits for the coarse aggregate industry through reduced storage and disposal costs, and increased revenues from the sale of fines.

Part I: Evaluation and Characterization of Materials

In order to evaluate the nature of byproduct fines production in the state of Florida, with an emphasis on identifying high volume economic uses for these materials that are attractive to coarse aggregate producers in the state, three goals were identified. The first of these was to estimate the current and future quantity and quality of byproduct resources at selected sites in the state. This was accomplished using a questionnaire and visits to selected producers identified by the Florida Department of Transportation (FDOT) to have significant inventories and/or be future producers of fines and screenings, in order to quantify both the present and future magnitude of the byproduct fines problem. The questionnaire used was modified after one developed by the International Center for Aggregate Research (ICAR) as part of a national study of byproduct fines production.

For the purpose of this report, fines were defined as either coarse (minus-3/8" by plus-200 mesh) or fine (minus-200 mesh), with the fine category representing the greater waste and storage concern as identifying by aggregate producers in the state. Although the quantity of coarse fines produced annually exceeds that of the fine category, producers tend to sell approximately 78 percent of the coarse category as compared to 34 percent of the fine. As a result, producers identified the need for more research and marketing directed at developing products for the minus-200 mesh fines, particularly given that total byproduct fines production is estimated at 300 million tons over the next ten years.

The second goal of the study was to characterize the physical and mineralogical characteristics of these byproduct fines presently stockpiled and sold commercially in both state and national markets. This has been accomplished through an investigation of the particle-size distribution (gradation), moisture content, and mineralogy of byproduct fines collected from coarse aggregate producers identified by the FDOT, representing a variety of limestone and dolomitic limestone/dolomite lithologies presently exploited by the aggregate industry. Wet sieve analysis of bulk fines, hydrometer test analysis of minus-200 mesh fraction samples, and x-ray diffraction (XRD) of both bulk fines and the acid insoluble fraction (2N HCl) were undertaken in order to satisfy this goal. Evaluation of the resulting compositional and physical data can be used as input in the development of specifications and test procedures used to evaluate and approve fines for the production of manufactured aggregate materials and in other high volume applications. Furthermore, this data should aid in identifying the most appropriate economic use for fines based on spatial constraints associated with lithologic variation.

Part II: Evaluation and Characterization of Products

The third goal of the study was to identify potential economic uses for these fine materials, increase productivity, and extend the life of an important natural resource based on temporal and spatial variations in composition. This has been fulfilled through a review of the literature available on the use of byproduct fines, evaluation of economic data, and testing of processing methods on fines from three sites identified by the FDOT and representing different lithologies. The literature review focused on the published and unpublished literature on agglomeration and/or compaction of fines, as well as relevant computer programs that relate to the production of manufactured aggregate materials. However, other high volume uses which might be of interest to the FDOT (backfill, flowable fill, and direct additives to concrete) were investigated as well.

The four processing methods investigated for the agglomeration of minus-200 (< 75 µm) limestone fines were drum granulation, pan granulation, roll-press flaking, and roll-press briquetting. These processes form the basis for most fine powder agglomeration found in industry today, and are believed to be useful in providing granules for use as aggregate in concrete. All four of the processes have/or are currently being used to produce agglomerated limestone for use as agricultural liming agents.

Based on investment cost data developed for limestone granulation and compaction units (Table 2-1), the cost for a “wet” granulation process (drum granulation and pan granulation) is slightly higher than that for a “dry” compaction unit (roll-press flaking and roll-press briquetting). Most of the cost difference is due to greater costs for instrumentation, piping and ductwork, auxiliary facilities and buildings. The process equipment cost is essentially the same for both units (Tables 2-2 and 2-3) because the cost of the compactor and associated equipment for the compaction plant is about equal to the cost of the granulator and drying system in the granulation plant. Most of the peripheral equipment is about the same for each type of unit.

Tables 2-4 and 2-5 show the calculated conversion costs for both compaction and granulation. This analysis shows that the conversion costs including utilities, labor, maintenance, taxes, insurance, and capital recovery are about 30 % higher for wet granulation than for compaction. Given a yearly production of 78,800 tons, a savings of $488,000 per year would be realized in operating costs with the compaction plant. This would be an ongoing savings in addition to the estimated $296,000 savings in the investment cost for the compaction plant compared to wet granulation.

The key to successfully adapting any one of these processes to produce a granule suitable for aggregate use is through identifying a binder capable of producing a limestone granule with adequate crush strength for use as concrete aggregate. Samples were ultimately granulated using a wet processing method similar to drum granulation, but using a pug mill, as it was determined to be more cost effective, mechanically more simple, and more efficient than any of the other wet or dry processing methods investigated. Sodium silicate, Portland cement, and calcium sulfate hemihydrate ($CaSO_4 \cdot ½H_2O$) were investigated as potential binders.

Final granular aggregate products were evaluated in 2”×2” Portland cement concrete (PCC) test cubes with mixed results. Samples prepared with the sodium silicate binder performed poorly, partially in response to unexpected water solubility of the granules, while the samples prepared with Portland cement as the binder performed much better. Quarry H samples with the Portland cement binder performed almost as well as the Ottawa sand standard, possessing a mean 28-day compressive strength value within 250 psi of the Ottawa sand sample. With the results of the quarry H granules, a reevaluation of the binders used in the granulation process, including binder concentrations, might improve granule strength and PCC test results, providing a valuable, high volume application of granulated byproduct fines as a fine aggregate alternative in PCC or ready mixed flowable fill (RFF).

1 PART I: EVALUATION AND CHARACTERIZATION OF MATERIALS

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INTRODUCTION

The accumulation of fines (minus-3/8") produced by the coarse aggregate industry in the state of Florida is one of the major problems facing the industry today. As construction specifications continue to limit the proportion of fine materials allowed in most applications, continued stockpiling and disposal of this byproduct, and in particular the minus-200 Tyler mesh fraction, has become both economically and environmentally unsound. It is estimated that 100 to 200 million short tons of minus-200 mesh limestone fines accumulate annually in settling ponds and fine screen piles at quarries across the United States. In Georgia, where two to four million tons of byproduct pond fines are produced by the crushed stone industry, it is estimated that the cost of disposal and loss of sales averages 25 to 35 million dollars annually (Hancock and Scott, 1996). According to a survey by the U.S. Bureau of Mines Mineral Industry, plant waste factors for all of the types of fines range from 15% to 25% of total production, a value likely underestimated for the coarse aggregate industry in the state of Florida. The survey also estimates that there are presently 4 billion tons of quarry fines stockpiled in the United States. These quantities are likely to increase another two billion tons by the turn of the century in response to increased production levels, stricter environmental regulations, and an increased demand for clean coarse aggregate products.

Since 1970, Florida's crushed stone industry has grown from about 40 million tons per year to about 90 million tons per year of production. Current technology often requires about two tons of stone be mined and delivered to the processing plant per ton of salable product. Of the byproducts that are generated when crushed limestone aggregate is produced, the screenings fraction (minus-4 mesh by plus-200 mesh) and finer particle sizes (minus-200 mesh) represent the greatest under-utilized resource.

In response to the problems of stockpiling and disposal of byproduct fines, the International Center for Aggregate Research (ICAR) has created a special task force, the Fines Expert Task Group (FETG), in order to find uses for these materials, particularly the minus-200 mesh fraction. If a solution to this problem can be found, it will lead to economic benefits to the aggregate industry through reduced storage and disposal costs, and increased revenues from the sales of fines. However, potential uses of byproduct fines must be identified which are technically and economically feasible and conducive to marketing large volumes of fines in order to be attractive to the aggregate industry. To date, byproduct fines have been primarily limited to use as agricultural additives and soil conditioners, and as fill materials.

Begun in 1995, the ICAR sponsored project "Engineering Uses for Aggregate Fines" has focused on the use of byproduct fines in flowable fills, high fines pavement bases, high fines Portland cement concrete, vertical moisture barriers and slurry walls, and soil stabilization. One outcome of this project was the recognition that the amount, condition, and characteristics of stockpiled fines are poorly known at the national, regional, and state levels, factors which must be addressed prior to the investigation of potential uses for fines.

OBJECTIVES AND SCOPE

The ultimate objective of this research project is to evaluate the nature of byproduct fines production in the state of Florida, with an emphasis on identifying high volume economic uses for these materials which are attractive to coarse aggregate producers in the state. The FDOT is the focus of this project, with the results aimed at enhancing the awareness of FDOT personnel to the geographic distribution, quantities, and properties of coarse aggregate byproducts that may be used as raw material for the production of manufactured aggregates and other secondary applications identified by this study. Phase I of the study is aimed at identifying the volume and characteristics of byproduct fines produced annually in the state of Florida, as well as estimating the quantity and characteristics of byproduct fines already stored at quarries throughout the state. The resulting goals aimed at accomplishing these objectives are:

(1) to estimate the current and future quantity and quality of byproduct resources at selected sites in the state

(2) to characterize the physical and mineralogical characteristics of byproduct fines in Florida presently stockpiled and sold commercially in both state and national markets

The first of these goals was accomplished through the use of a questionnaire (Appendix A) and visits to selected producers identified by the Florida Department of Transportation (FDOT) to have significant inventories and/or be future producers of fines and screenings, in order to quantify both the present and future magnitude of the byproduct fines problem. The questionnaire used in this part of the study was modified after that used by the International Center for Aggregate Research (ICAR) as part of a national study of byproduct fines production.

The second goal was carried out through investigating the particle-size distribution (gradation), moisture content, and mineralogy (including acid insoluble content) of byproduct fines collected from coarse aggregate producers identified by the FDOT as representing a variety of limestone and dolomitic limestone/dolomite lithologies presently mined by the aggregate industry. Wet sieve analysis of bulk fines, hydrometer test analysis of select samples to characterize the particle-size distribution of the minus-200 mesh fraction, and x-ray diffraction (XRD) of both bulk fines and the acid insoluble fraction (2N HCl) have been completed and evaluated in order to satisfy this goal. Collection of compositional and physical data can be used as input in the development of specifications and test procedures which could be used by the FDOT to evaluate and approve fines for the production of manufactured aggregate materials and in other high volume applications. Furthermore, this data should aid in identifying the most appropriate economic use for fines based on spatial constraints associated with lithologic variation.

BYPRODUCT FINES DATABASE

The problem of handling and disposing of fines is one of the largest being faced by the aggregate industry today. Literature searches by Hudson et al. (1997) found no effort to quantify the magnitude of the problem at the national level. In 1992, Kumar et al. used a questionnaire format to seek information from 101 producers all over the country. Their data showed that 130 million tons of fines were being stockpiled annually, but recognized that this number is crude based on the limited scope used and variability of the response data. Hudson undertook the task of compiling a national fines survey using an "information booklet" and received responses from154 companies with 362 plant operations. These plants produced about 287 million tons/year of products with 485 million tons/year of minus-3/8" fines (95 million tons were not marketed). For the minus-200 mesh fines, the figures were about 105 million tons/year with 75 million tons/year not marketed. Stockpile numbers showed about 350 million tons of each material were in storage at the various sites. There were no reported widespread uses for the minus-200 mesh fines. ICAR has an ongoing program to characterize these fines and to find high volume uses for them.

The results presented here were derived from mine visits to several sites and from information booklet (see Appendix A for example) responses. The information booklet used in this study was adapted from the ICAR study to conditions familiar to Florida producers. Florida Limerock and Aggregate Institute (FLAI) members participated in the study as well as some other producers. Responses were received from 11 companies operating 25 mines around the state.

The following components were included in the list of issues incorporated in the information booklet:

1. CHARACTERIZATION
    - i. Mineralogy and chemical composition
    - ii. Size data (3/8" and minus-200 mesh)
    - iii. Wet or dry
    - iv. Contamination due to overburden or other sources
    - v. Quantities
        1. Amounts of fines as percentages of total production
        2. Percentage of fines which are marketable
        3. Percentage of fines which are not marketable
        4. Inventory which is currently not marketable
    - vi. Physical properties
2. MARKET/DISPOSAL
    - i. Current markets and method for disposal
    - ii. Potential markets
    - iii. Competitive materials

iv. Regulations
v. Specifications
vi. Recovery of fines
vii. Disposal

3. PRODUCTION PROCESSING: General flow sheet
4. TRANSPORTATION

Objectives of the Data Collection

Objectives of the information booklet (i.e., questionnaire) inquiries were to build the database information on byproduct fines at mine sites around Florida. Specific objectives include:

- To quantify the magnitude of the fines.
- To determine the geographical distribution of stockpiled fines.
- To determine the quantities and types of fines.
- To investigate current and potential uses of fines.
- To evaluate the cost and technology for transforming fines.

Development of the Information Booklet

The format developed for the information booklet was adapted from the national ICAR study. It was modified to fit crushed stone operations in Florida with regard to rock types, products, and geologic information. The questions were drafted by the University of Florida researchers and reviewed by FDOT staff at the Bureau of Materials and by the Florida Concrete Products Association representatives before distribution. The objective was to gather as much useful information as possible while at the same time meeting the following requirements:

- The information requested should be easy to comprehend.
- It should be easy to fill out.
- It should not take an unreasonably time to complete
- Proprietary concerns of the participants should be given consideration
- Future contacts would be facilitated by requesting a designated contact person.
- Design should include ease and efficiency of data entry into the database.
- Information should be mine specific rather than company-specific to achieve project goals.

The final questionnaire consisted of six pages. The questions covered the following main areas:

- Geographical Location

    - Addresses, telephone, FAX, and mine FDOT mine numbers
    - Market areas
- Properties of the Aggregates Processed
    - Types of aggregate material being processed
    - Geologic formation being mined
    - Production
    - Market area
    - Transportation
- Products
- Known problems
- Product marketing
    - Sizes
    - Products
- Physical and chemical properties
    - Mineralogy
    - Chemistry
    - Physical characteristics
- Process flow sheet
    - Comminution
    - Sizing

The initial response to the questionnaire was poor. However, with the assistance of the Florida Limerock and Aggregate Institute, Inc., a second reminder letter was sent and responses were received from about 40% of all the companies contacted. Though this was less than initially hoped for, the data that was provided covered large geographic areas of the state, a very significant portion of total state production, and a diverse range of producers from large to small in a variety of geologic settings.

DATA ANALYSIS

Response to the Information Booklets

At first appearance, the response to the information booklets seems disappointing. Only eleven companies out of about thirty polled responded. However, a closer analysis of the results showed that the mines reported account for about eighty percent of the crushed stone produced annually in Florida. Data on crushed stone production show that Florida produced about 70 million tons in 1996; the annual production at that time from the companies and mines in this study was about 56 million tons. In addition, there is a good geographic distribution of data with reports from all areas except northwest Florida. Four reports are from east and southeast Florida, eight are from southwest Florida, and eight are from south Florida.

Annual Production of Crushed Stone

In 2000, Florida ranked third in the nation in crushed stone production. Between 1971 and 2000, stone production in Florida increased from about 40 million tons per year to about 90 million tons per year (Fig. 1-1). Maximum production was achieved during 2000, when approximately 93 million tons were mined, with a notable production peak during 1988-89 during which about 75 million tons were mined yearly. The overall trend has been a steady increase in production over this three-decade period with rises and falls in production related to general economic conditions. Crushed stone traditionally ranks second, valuewise, to phosphate rock among the mineral commodities mined instate and normally accounts for about 30 percent of Florida's annual mineral value. In 2000, the value of stone mined was valued at $495 million.

The Florida stone industry produces limestone, dolomite, shell, and marl. Limestone accounts for 95 percent or more of the tonnage. Although limestone and dolomite are mined in 22 counties, five counties (Dade, Broward, Hernando, Lee, and Citrus) account for approximately 70% of this production.

Annual Production of Fines

For the purposes of this report, fines are defined in two categories. The coarser class is the minus-3/8" by plus-200 mesh fraction that includes the commercial grade described as "screenings". The second category is the minus-200 mesh fraction. These size fractions may be separated during storage or disposal though some mines discharge mixtures of these sizes in piles and pits. Most fines are created during the crushing and grinding phases of production and seldom exist in substantial quantities in the ore itself. The fine particle sized fractions are removed

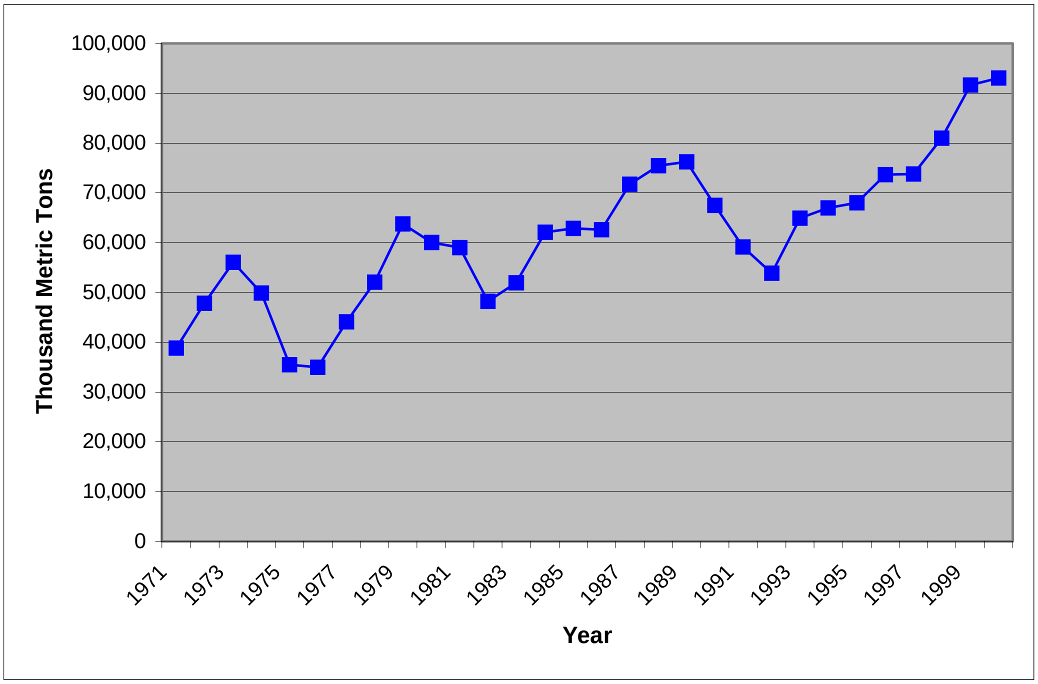

Figure 1-1. Total crushed stone production in Florida (Source: U.S.G.S. Commodity Reports).

from the coarser, more easily saleable products by combinations of washing and screening in one or more steps during processing and sizing.

The minus-3/8" by plus 200-mesh fraction, expressed as a percentage, was obtained for each mine by dividing the minus-3/8" fines produced annually by the total annual production and multiplying by 100. For the data reported, the numerical average production of minus-3/8" is 30 percent of the total annual tonnage (N = 20, Max = 48.3%, Min = 16.7%, Std = 10.6%).

To include the effects of the various annual production rates, a weighted average was calculated as follows:

$$\text{Weighted average} = (AP_m/TP_m)\times(AP_f/TP_f)\times 100$$

where,

AP_m = annual individual mine production (i.e., FDOT mine number 99-999)
TP_m = total annual production reported in this survey
AP_f = annual production of fines fraction (either minus-3/8" or minus-200 mesh)
TP_f = total production of individual fine fractions reported in this survey

The weighted average of minus-3/8" material is 14.9 percent, or half the unweighted value. This value is strongly influenced by large producers that have relatively small percentages of their feed reporting to this fraction of fines. The statistics for the weighted average are N = 20, Max = 7.2%, Min = 0.01, and Std = 2.51%.

The data analysis for the minus-200 mesh fraction shows a numerical average of 13.6 percent where N = 20, Max = 49%, Min = 2.0%, and Std = 14.8%. The numerical average value is rather meaningless with this large standard deviation in the data, but this is an expected result with the great range of values reported for this small data set.

If the data are related to reported annual and total production numbers, the weighted average is 14.2 percent where N = 20, Max = 6.0%, Min = 0.0%, and Std = 2.21%. The weighted average is about the same as the numerical average, but the standard deviation of the data is smaller. This method provides a more meaningful analysis, because the weighting reduces the effect of small producers that generate a significant fraction of minus-200 mesh.

The reporting producers made 21.4 million tons of minus-3/8" fines for the reporting period and sold 16.8 million tons (78 percent). The inventory of this product increased by 4.6 million tons. Respondents reported stockpiles of 75 million tons of minus-3/8" material. During the same period, 8.4 million tons of minus-200 mesh fines were produced, and 2.1 millions tons were sold (34 percent). The inventory of minus-200 mesh fines was increased by 6.3 million tons for the reporting producers. The amount of stockpiled minus-200 mesh material was reported to be 30 million tons. Assuming the factors affecting the respondents are the same for non-reporting crushed stone producers, the statewide total for these inventories can be estimated by increasing these values by twenty-five percent (i.e., 70 Mt produced/56 Mt reported). The resulting inventories would be 94 million tons for the minus-3/8" and 38 million tons for the minus-200 mesh fractions.

These results contain some interesting information. The quantity of minus-3/8" produced per year or in stockpiles is 2.5 times greater than the quantity of minus-200 mesh fines produced. Not surprisingly, a portion of the minus-3/8" material is more readily marketed with nearly eighty percent of current production being sold. Producers report a need for research and marketing assistance in disposing of the minus-200 mesh fines. This may arise from the fact that the minus-200 fraction is accumulating in stockpiles and ponds about fifty percent faster than the minus-3/8" material. The greater moisture contents associated with the very fine fraction materials can result in increased volumes and other problems of storage and handling. In addition, the cost of dewatering fines is under evaluation nationwide by producers who are applying improved mineral processing technologies as the cost effectiveness of these treatments increases.

The data in the information booklets can lead to other interpretations that are important in quantifying the magnitude of the byproduct fines problem in Florida. One of these is based on the history of crushed stone production in Florida. Assuming that the respondents are a representative cross-section of Florida producers and their applied technologies, the weighted averages derived from their input can be applied to the production data for the past 20 years to estimate the quantities of fines that have been produced. In addition, statistical models can be developed to project these numbers into the future. Thus, data analysis methods can be used to estimate the past and future production of fines, allowing a quantitative understanding of the magnitude of this problem.

The U.S. Geological Survey, in cooperation with the Florida Geological Survey, and the former U. S. Bureau of Mines compile statistics on annual crushed stone production for Florida (see Fig. 1-1). Adding the weighted average for minus-3/8" (14.9 percent) and for the minus-200 mesh (14.2 percent) results in 29.1 percent as the total of byproduct fines produced. The total stone production from 1971-2000 was about 1,900 million tons. Multiplying the cumulative production times the weighted average of byproduct fines shows that the total produced over this period is about 550 million tons (i.e., 19 million tons/year) with nearly equal amounts of minus-3/8" and minus-200 mesh materials. This calculation does not agree with the respondent's data which show more than twice as much minus-3/8" material stockpiled (75 million tons) as minus-200 mesh material (33 million tons). This disparity may result from changes in technology that have reduced the quantity of minus-200 mesh material being produced. The types of stones being mined and milled also may be factors in changing this distribution. The 1997 ICAR study (Hudson et al., 1997) showed that the average production of minus-3/8" material was 22.4 percent of annual production for limestone and dolomite producers. For minus-200 mesh materials from limestone and dolomite producers in the same study, the figure was 3.6 percent. Stockpiles of minus-3/8" and minus-200 mesh fines reported in the ICAR study were 8.18 million and 7.55 million tons, respectively, for limestone and dolomite producers that reported.

In 1971, Florida produced 39 million tons of crushed stone. In 2000, that figure had grown to 93 million tons. Production increased by 54 million tons over a 29-year period (i.e., an average increase of 1.86 million tons/year). Assuming this growth rate will be sustained and using the weighted average production of byproduct fines, future production of fines can be estimated as follows:

$$APF_i = (CP_i + (n \times API)) \times WA$$

where,

APF_i = annual production of fines (in million tons - Mt)
CP_i = year specific annual stone production (in million tons - Mt)
n = number of year in the future
WA = weighted average of byproduct fines production (29.1 percent for this study)
API = annual production increase (1.86 million tons/year for the 29 year period used in this study.

Using this relationship, the annual production of fines for the year 2005 from 2000 data would be:

$$API_{2005} = ((93\text{ Mt}) + (5 \times 1.86)) \times 29.1\% = 29.8\text{ Mt}$$

Similarly, for the year 2010 from 2000 data:

$$API_{2010} = ((93\text{ Mt}) + (10 \times 1.86)) \times 29.1\% = 32.5\text{ Mt}$$

Applying these relationships to project future production shows that annual production will rise from 93 million tons per year in 2000 to about 112 million tons in 2010. The cumulative tonnage of byproduct fines that would be produced during this ten-year period will be about 300 million tons (154 million tons of minus-3/8" material and 146 million of minus-200 mesh).

Marketing of fines met with variable success depending on the product. For the minus-3/8" material, 16.8 million tons were sold and represented 78 percent of the materials made (in one case 100 percent) and an average of 24 percent of the total stone processed. For the minus-200 mesh fraction, 2.1 million tons were sold representing only 34.4 percent of the product made and 3.2 percent of the stone mined. These figures are quite comparable with the data from the 1997 ICAR study (Hudson et al., 1997) that showed 82.2 percent of minus-3/8" limestone and dolomite and 37.5 percent of the minus-200 mesh were sold by the companies they surveyed. These differences in sales rates impact the proportions of size fractions accumulated in stockpiles where the minus-200 mesh fraction is growing faster than the minus-3/8" material.

CHARACTERISTICS OF FLORIDA BYPRODUCT FINES

In the state of Florida, byproduct fines (both coarse and fine categories) from the coarse aggregate industry are deposited in abandoned quarry pits, collected in stockpiles, and sold as agricultural additives (Aglime) (Figs. 1-2 through 1-5). The byproduct fines vary in age, and as such their condition, often being overgrown by years, if not decades, of vegetation, and represent a major waste storage problem for the Florida aggregate industry. Being that aggregate mining takes place in a variety of locations around the state, which are, in turn, characterized by different geologic formations, often with variations in lithology from location to location (Fig. 1-6), the byproduct fines produced vary, as a result, in their moisture content, particle size characteristics (gradation), and mineralogy. As shown by Stokowski (1993), within source variation of these materials may be just as important as source to source variation, resulting in unique physical, mineralogical, and chemical properties along each point in the coarse aggregate production process. As a result, both source to source and within source variance in material properties are important considerations in the evaluation of potential markets of byproduct fines. Two types of byproduct fines are normally produced during coarse aggregate processing; primary fines and secondary fines. Primary fines (minus-3/8") originate during primary crushing and sizing/washing of aggregate raw materials prior to processing by the commercial products plant (Fig. 1-7). These materials are commonly discarded as waste, while the plus-3/8" material is further crushed and sized/washed to produce commercial coarse aggregate products. Byproduct fines produced during this latter stage of processing are termed secondary fines, and are either discarded as waste, or further processed into fines products (Fig. 1-7). For the purpose of this study, two size fractions of both primary and secondary fines were examined, the coarse fraction (minus-3/8" by plus-200 mesh) including screenings (minus-4 mesh by plus-40 mesh), and the fine fraction (minus-200 mesh). Samples taken for the study are coded in accordance with these size fractions, with identification labels (X-X-X) listing the quarry code (letter A-G), fines type (1 = fine, 2 = coarse, and 3 = screenings), and sample number.

Lithology of Selected Mines

Seven quarries were selected and sampled as part of this investigation to incorporate a variety of lithologies from around the state of Florida (Table 1-1). Lithologies include dolomitic limestone and dolomite from the Suwannee Limestone in Taylor County and the Avon Park Formation in Levy and Citrus counties, as well as limestone from the Suwannee and Ocala limestones in Hernando and Columbia counties and the Tamiami and Ft. Thompson formations in Lee County (Fig. 1-6). As the lithologies studied can be readily separated into dolomitic limestone/dolomite and limestone varieties, these two groups will be examined separately in some detail for both coarse (minus-3/8” by plus-200 mesh) and fine (minus-200 mesh) categories of fines. Analysis of screenings are included with the coarse category of fines as noted previously.

Figure 1-2. Fresh coarse (minus-3/8" by plus-200 mesh) limestone fines collecting in an abandoned mine pit in Hernando County, Florida.

Figure 1-3. Old coarse (minus-3/8" by plus-200 mesh) limestone fines overgrown by vegetation which have been stored in an abandoned mine pit in Hernando County, Florida.

Figure 1-4. Primary coarse (minus-3/8" by plus-200 mesh) dolomitic limestone/dolomite fines which have been stored in stockpiles in Levy County, Florida.

Figure 1-5. Secondary fine fraction (minus-200 mesh) dolomitic limestone/dolomite fines being pumped into dewatering pits for later excavation and sale as an agricultural additive.

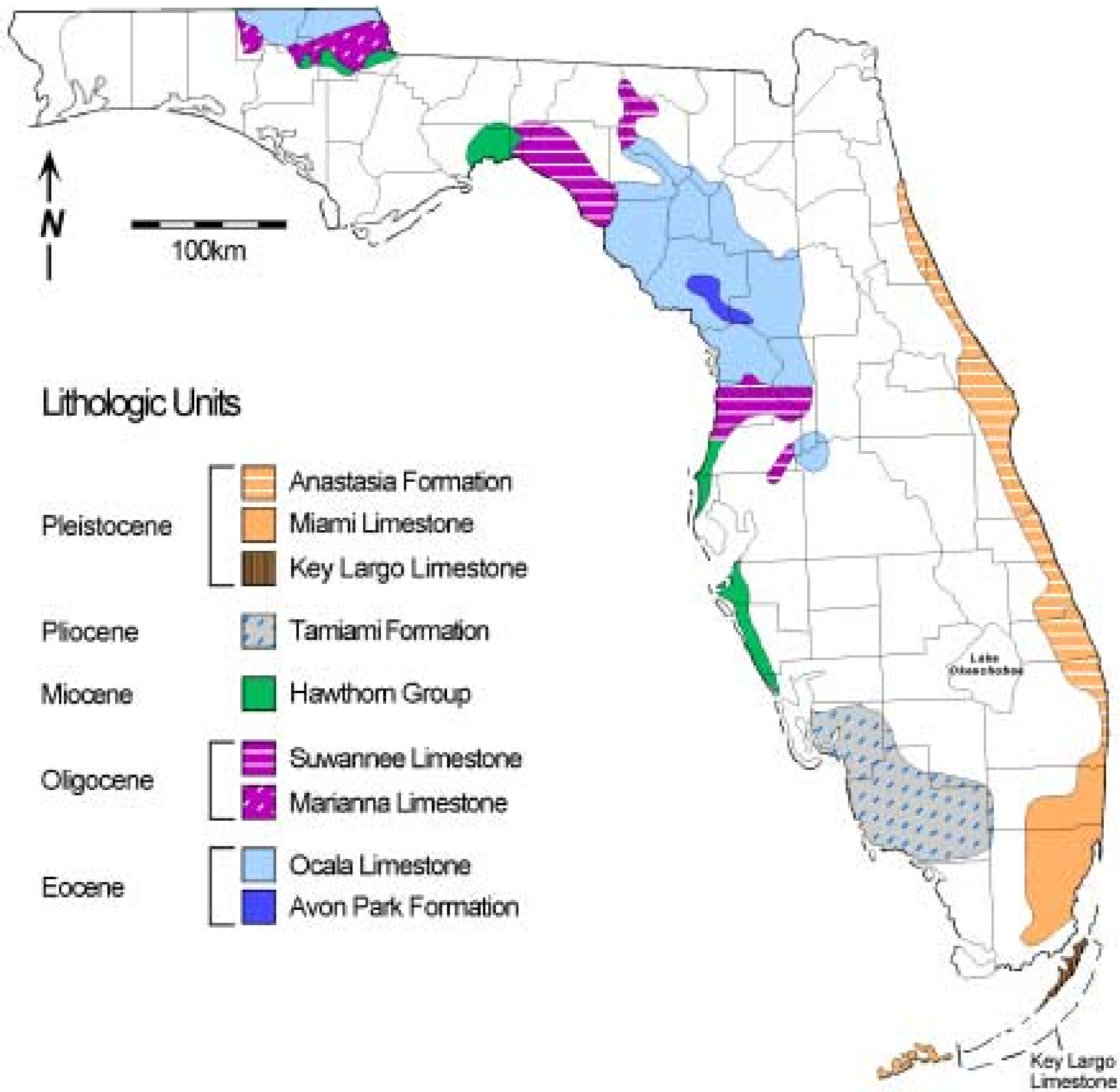

Figure 1-6. Carbonate rock (limestone and dolomitic limestone/dolomite) lithologies mined by the coarse aggregate industry in Florida.

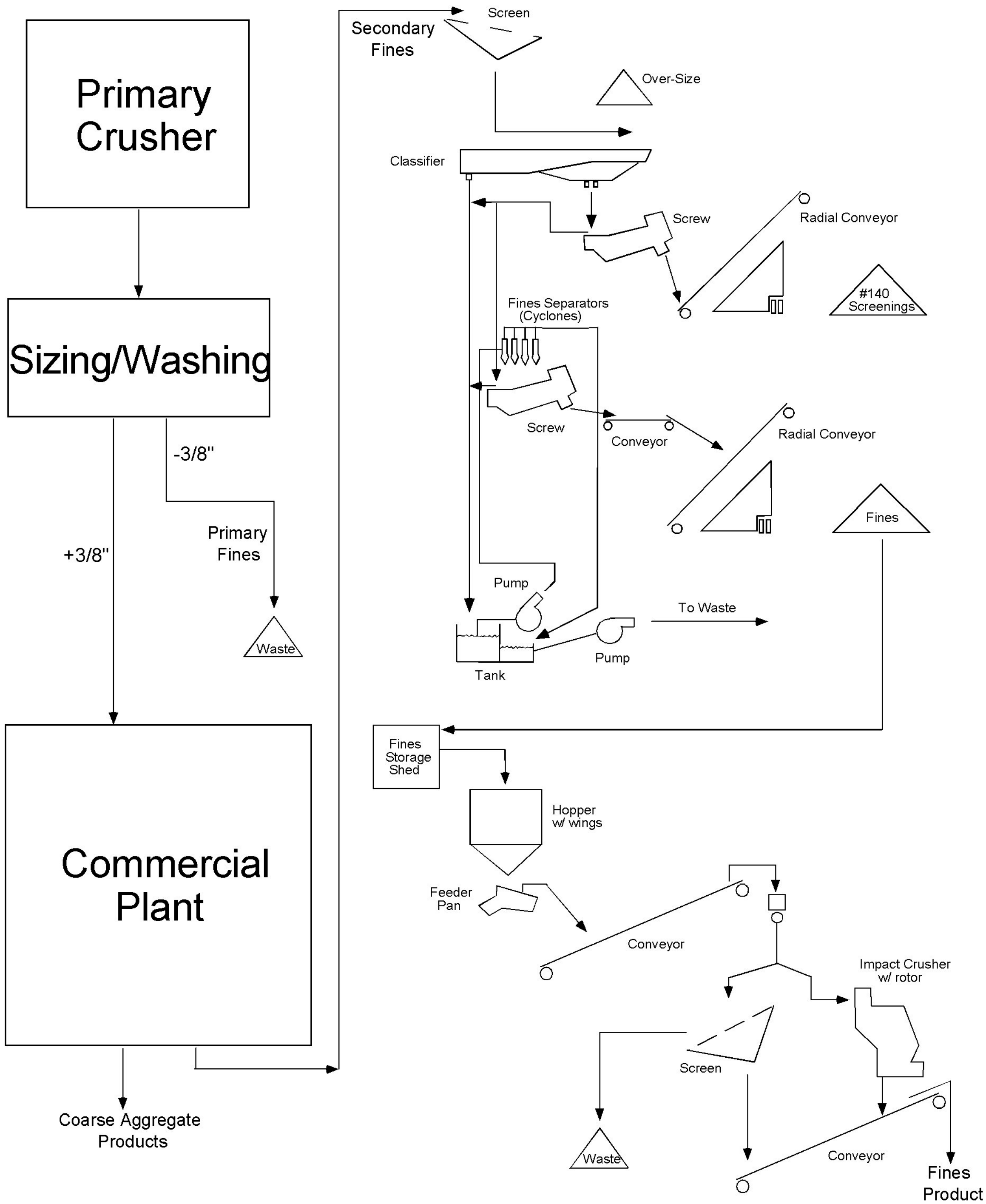

Figure 1-7. Example flow chart for byproduct fines production and disposal in Florida.

Table 1-1. Lithology and representative mineralogy summary for the quarries selected as part of this study.

Quarry Code	Formation	Lithology	Location	Coarse Aggregate Mineralogy*		
				Calcite %	Dolomite %	Quartz %
A	Suwannee Limestone	Dolomitic limestone/Dolomite	Taylor Co.	1	99	---
B	Suwannee Limestone	Dolomitic limestone/Dolomite	Taylor Co.	1	99	---
C	Avon Park Formation	Dolomitic limestone/Dolomite	Levy Co.	57	43	---
D	Avon Park Formation	Dolomitic limestone/Dolomite	Citrus Co.	57	43	---
E	Suwannee/Ocala Limestones	Limestone	Hernando Co.	94	---	6
F	Suwannee/Ocala Limestones	Limestone	Hernando Co.	94	---	6
G	Suwannee/Ocala Limestones	Limestone	Hernando Co.	94	---	6
H	Tamiami/Ft. Thompson Fms.	Limestone	Lee Co.	83	---	17
I	Ocala Limestone	Limestone	Columbia Co.	100	---	---

* Representative aggregate mineralogy based on previous studies (McClellan et al., 1990; Eades et al., 1997; McClellan et al., 2001).

Moisture Content/Acid Insoluble Content

Fines produced in Florida have inherently high moisture contents, resulting in problems associated with handling and sale of the material. Commonly, moisture contents average about 20 to 30 percent when fines are initially removed from a settlement pond and decrease to between 5 and 15 percent during stockpiling (Stokowski, 1992). Review of the data collected on samples for this study (Tables 1-2 and 1-3) show that mean (numerical average) moisture contents between the different lithologies are similar, but that within lithology moisture contents vary substantially, as reflected by standard deviation values. Dolomitic limestone/dolomite fines have a mean moisture content of 16.8 percent (N = 40, Max = 38.6%, Min = 5.7%, Std = 10.2%), limestone fines have a mean moisture content of 18.4 percent (N = 20, Max = 35.4%, Min = 1.1%, Std = 8.1%), and the total data set has a mean moisture content of 17.3 percent (N = 60, Max = 38.6%, Min = 1.1%, Std = 9.5%). The variation seen with this data is due, in great part, to the varying age of fines and storage methods (stockpile vs. quarry pit). Furthermore, particle-size differences among fines are likely to cause extreme differences in water storage and/or retention characteristics.

To transform wet fines into damp or dry products, producers can use several available methods including sand classifiers, flocculation and classifier tanks, dewater screens, cyclones, separators, belt presses, heat/rotary drying, and dragline/truck stockpiling. In Florida, most

Table 1-2. Moisture and acid insoluble content of dolomitic limestone/dolomite fines sampled for
the purpose of this study.

* Sample Code corresponds to quarry code, fines type (1 = minus-200 mesh (fine), 2 = minus-3/8"/plus-200 mesh
(coarse), and 3 = minu-4/plus-40 (screenings)), and sample number.

Sample Code*	Wet Sample Wt. (g)	Dry Sample Wt. (g)	Water Wt. (g)	Moist. %	Dry Sample Wt. (g)	Acid Insol. Wt. (g)	Acid Insol. %	Acid Insol. Mineralogy**
A-1-1	49.60	40.70	8.90	21.9	20.0	0.20	1.0	Q>>S>H
A-2-1	22.80	16.70	6.10	36.5	20.0	0.37	1.9	Q>>S>R
A-2-2	36.80	27.40	9.40	34.3	20.0	0.45	2.3	Q>>R
A-2-3	25.30	23.90	1.40	5.9	20.0	0.31	1.6	Q>>R
A-3-1	29.50	27.90	1.60	5.7	20.0	0.24	1.2	Q>>S
A-3-2	25.10	23.20	1.90	8.2	5.8	0.08	1.4	Q>>S>R>H
Mean				18.7			1.5	
Median				15.0			1.5	
STD				14.2			0.5	
B-2-1	47.38	42.23	5.15	12.2	20.0	0.20	1.0	Q>>K>S>P
B-2-2	66.14	50.22	15.92	31.7	20.0	0.13	0.7	Q>>S>P>R
B-2-3	60.48	50.75	9.73	19.2	20.0	0.20	1.0	P>Q
B-2-4	55.91	49.77	6.14	12.3	20.0	0.15	0.8	Q>>S>P
B-2-5	65.46	52.89	12.57	23.8	20.0	0.17	0.9	Q>>S>P
Mean				19.8			0.9	
Median				19.2			0.9	
STD				8.2			0.2	
C-1-1	16.73	12.44	4.29	34.5	20.0	0.84	4.2	Q>S
C-1-2	14.61	10.99	3.62	32.9	20.0	0.48	2.4	Q>S>P>R
C-1-3	19.37	14.47	4.90	33.9	10.0	0.56	5.6	Q>>S
C-1-4	17.98	15.83	2.15	13.6	20.0	0.38	1.9	Q>>S>P>R
C-1-5	21.69	19.53	2.16	11.1	20.0	0.77	3.9	Q>S>P>R
C-1-6	18.80	14.99	3.81	25.4	20.0	1.08	5.4	Q>S>P>R
C-1-7	21.70	19.25	2.45	12.7	20.0	0.45	2.3	Q>S>P>R
C-1-8	18.16	15.39	2.77	18.0	20.0	0.68	3.4	Q>S>P
C-2-1	25.56	20.29	5.27	26.0	20.0	0.71	3.6	S
C-2-2	23.86	21.24	2.62	12.3	10.0	0.53	5.3	Q>S
C-3-1	18.24	16.56	1.68	10.1	16.6	0.30	1.8	Q>S>P
Mean				21.0			3.6	
Median				18.0			3.6	
STD				9.8			1.4	
D-1-1	11.95	10.78	1.17	10.9	20.0	0.46	2.3	Q>S>G
D-1-2	11.86	8.56	3.30	38.6	20.0	0.23	1.2	S>Q>P>>G>R
D-1-3	10.55	9.00	1.55	17.2	20.0	0.20	1.0	Q>>P>S
D-2-1	11.11	9.81	1.30	13.3	20.0	0.72	3.6	Q>S
D-2-2	11.41	9.40	2.01	21.4	18.7	0.52	2.8	Q>>S>P
D-2-3	11.08	10.38	0.70	6.7	20.0	0.23	1.2	Q>>S>P
D-2-4	11.68	10.86	0.82	7.6	20.0	0.25	1.3	Q>>S>P
D-2-5	11.15	10.29	0.86	8.4	20.0	0.30	1.5	Q>>S>P>G
D-2-6	11.00	10.06	0.94	9.3	20.0	0.43	2.2	Q>>S>P>G
D-2-7	10.46	9.65	0.81	8.4	20.0	0.39	2.0	Q>S>P
D-2-8	10.17	9.31	0.86	9.2	20.0	0.55	2.8	Q>>S>P
D-2-9	10.59	9.73	0.86	8.8	20.0	0.42	2.1	Q>P>S
D-2-10	10.81	9.99	0.82	8.2	20.0	0.49	2.5	Q>P>S
D-2-11	10.60	9.75	0.85	8.7	20.0	0.38	1.9	Q>P>S
D-2-12	10.25	9.52	0.73	7.7	20.0	0.23	1.2	Q>>S>P>R
D-2-13	12.09	9.47	2.62	27.7	20.0	0.11	0.6	S>Q>P
D-3-1	11.19	10.49	0.70	6.7	13.5	0.15	1.1	S>Q>P>G
D-3-2	10.72	9.75	0.97	9.9	17.6	0.14	0.8	Q>P>S>G
Mean				12.7			1.8	
Median				9.0			1.7	
STD				8.5			0.8	

** Acid insoluble fraction mineralogy determined by XRD using ethylene glycol solvated oriented mounts (Q = quartz, S = smectite (clay), K = kaolinite (clay), P = pyrite, R = rutile, and G = geothite).

Table 1-3. Moisture and acid insoluble content of limestone fines sampled for the purpose of this study.

* Sample Code corresponds to quarry code, fines type (1 = minus-200 mesh (fine), 2 = minus-3/8"/plus-200 mesh

Sample Code*	Wet Sample Wt. (g)	Dry Sample Wt. (g)	Water Wt. (g)	Moist. %	Dry Sample Wt. (g)	Acid Insol. Wt. (g)	Acid Insol. %	Acid Insol. Mineralogy**
E-2-1	40.80	34.50	6.30	18.3	20.0	1.78	8.9	Q>S
E-2-2	37.90	30.80	7.10	23.1	20.0	2.06	10.3	Q>S
E-2-3	30.62	27.83	2.79	10.0	20.0	1.64	8.2	Q>S
E-2-4	31.90	26.10	5.80	22.2	20.0	0.41	2.1	Q>S>P
Mean				18.4			7.4	
Median				20.2			8.6	
STD				6.0			3.6	
F-1-1	17.39	13.95	3.44	24.7	20.0	2.03	10.2	Q>>S
F-1-2	14.69	11.59	3.10	26.7	20.0	0.86	4.3	Q>>S
F-1-3	18.23	13.46	4.77	35.4	20.0	1.54	7.7	Q>S>>P>R
F-1-4	17.39	15.17	2.22	14.6	20.0	1.32	6.6	Q>S
F-1-5	18.33	14.82	3.51	23.7	20.0	0.93	4.7	Q>>S
F-1-6	15.58	12.08	3.50	29.0	20.0	1.26	6.3	Q>>S
F-2-1	20.00	19.78	0.22	1.1	20.0	2.16	10.8	Q>S>>R
F-2-2	16.30	13.90	2.40	17.3	20.0	1.72	8.6	Q>S>>R
Mean				21.6			7.4	
Median				24.2			7.2	
STD				10.5			2.4	
G-2-1	21.30	18.70	2.60	13.9	20.0	4.75	23.8	Q>>S
G-2-2	32.90	28.20	4.70	16.7	20.0	3.82	19.1	Q>S
G-2-3	32.70	30.10	2.60	8.6	20.0	3.61	18.1	Q>S
G-2-4	23.70	21.10	2.60	12.3	20.0	4.23	21.2	Q>>S
G-2-5	32.50	26.80	5.70	21.3	16.0	2.75	17.2	Q>>S
G-2-6	28.50	23.60	4.90	20.8	20.0	2.54	12.7	Q>>S
G-2-7	52.30	43.10	9.20	21.3	20.0	1.86	9.3	Q>>S
G-3-1	29.40	27.30	2.10	7.7	20.0	3.66	18.3	Q>>S
Mean				15.3			17.4	
Median				15.3			18.2	
STD				5.6			4.6	

(coarse), and 3 = minu-4/plus-40 (screenings)), and sample number.

** Acid insoluble fraction mineralogy determined by XRD using ethylene glycol solvated oriented mounts (Q = quartz, S = smectite (clay), K = kaolinite (clay), P = pyrite, R = rutile, and G = geothite).

byproduct fines are allowed to dewater slowly in stockpiles or are left wet and discharged to waste ponds, dependent on the economic situation of the operation and/or the producer's perceived market for fines products. An exception to this was the use of dewatering hydrocyclones by some producers, which were being used to produce a more rapidly dewatered Aglime product.

Hydrocyclone technology has been around for many years, and although relatively inefficient in terms of classification, they are very economical to install. With byproduct fines produced in Florida, dewatering is a major problem for hydrocyclones, particularly with solids that are minus-270 mesh. However, systems which combine hydrocyclones with dual motor, high frequency, dewatering screens can give both maximum recovery and dewatering capabilities of plus-400 mesh materials (Baxter, 1996). Some type of thickener or clarifier is required if removal of minus-400 mesh material is desired.

The acid insoluble fraction of each sample of byproduct fines was determined after digestion in 2N HCl, and then analyzed by x-ray diffraction (XRD) to determine mineralogy. The resulting data (Tables 1-2 and 1-3) illustrate that the limestone fines possess much greater acid insoluble contents with a mean of 11.4 percent (N = 20, Max = 23.8%, Min = 2.1%, Std =

6.1%) as compared to the dolomitic limestone/dolomite fines which have a mean value of 2.1 percent (N = 40, Max = 5.6%, Min = 0.6%, Std = 1.3%). The total data set has a mean acid insoluble content of 5.2 percent (N = 60, Max = 23.8%, Min = 0.6%, Std = 5.7%). Data sets exhibit a wide variation in values, resulting in the large standard deviations observed (Tables 1-2 and 1-3).

XRD analyses of the acid insoluble residues show them to be composed of varying mixtures of quartz, clay (smectite and/or kaolinite), pyrite (FeS_2), rutile (TiO_2), and goethite (FeOOH). The most common component is, by far, quartz, which is common in many of the limestone fines studied, as well as the parent lithologies (Table 1-1). Clays, particularly smectite, also are common in the byproduct fines; a result of relative clay concentration in the fine fraction, and the stratigraphic relationship of many of the lithologies studied to smectite-rich Hawthorn Group sediments in quarry pits. This may be an observation of concern when examining the potential applications for byproduct fines, as smectite clays possess shrink/swell characteristics, which have been identified as deleterious in many construction applications.

Gradation

The most characteristic property of byproduct fines is their fine grading. The finest materials produced at an aggregate plant, gradation of fines often varies within and between quarries in response to hydraulic fractionation, the lithology being mined, the type of products being produced, and plant design (Stokowski, 1992). Processing of carbonate rocks (limestone and dolomite) produces fines that are finer than those derived from granites or natural sand and gravel, commonly as a result of material hardness and the fine size of constituent minerals.

Fines were wet sieved according to standard methods in order to determine sample gradation. The resulting data (Tables 1-4 and 1-5) shows that gradation varies significantly among samples from individual quarries as indicated by large standard deviation values, particularly quarries for which both coarse and fine category fines were examined (A, C, D, and F). Gradation variations also are significant between different quarries. Furthermore, it is evident from the results that both coarse (minus-3/8” by plus-200 mesh) and fine category (minus-200 mesh) fines contain materials that fall outside the described particle-size ranges for each.

Mean particle-size gradations and gradation moment statistics were calculated for both dolomitic limestone/dolomite and limestone lithologies for each category of fines (Table 1-6, Figs. 1-8 and 1-9). The resulting data show that fine category (minus-200 mesh) limestone fines tend to possess a particle-size distribution with slightly more minus-325 material than that found with dolomitic limestone/dolomite lithologies, and that coarse limestone fines tend to possess more material at both extremes of the particle-size distribution, resulting in a lower concentration of material in the intermediate sieve intervals. Both lithologies of fine category fines are strongly coarse skewed (< -0.30), possessing skewness values for dolomitic limestone/dolomite and limestone lithologies of –1.87ϕ (N = 12) and –2.63ϕ (N = 6), respectively, indicative of excess coarse particles in the sample population. Oppositely, coarse fines are strongly fine skewed (>

+0.30), possessing excess fine particles (dolomitic limestone/dolomite = 0.73ϕ, N = 28; limestone = 1.09ϕ, N = 14).

Table 1-4. Gradation data for dolomitic limestone/dolomite byproduct fines.

Sample Code	Gradation (% Retained)					
	+40	-40/+60	-60/+100	-100/+200	-200/+325	-325
A-1-1	1	1	1	13	9	76
A-2-1	0	3	60	30	6	1
A-2-2	20	32	31	13	2	1
A-2-3	0	0	31	62	5	1
A-3-1	99	0	0	0	0	0
A-3-2	89	7	1	1	1	1
Mean	35	7	21	20	4	13
Median	10	2	16	13	4	1
STD	47	12	24	23	3	31
B-2-1	10	5	19	39	16	10
B-2-2	2	2	5	58	15	18
B-2-3	8	12	17	32	16	14
B-2-4	22	14	18	23	14	9
B-2-5	17	17	18	24	12	13
Mean	12	10	15	35	15	13
Median	10	12	18	32	15	13
STD	8	6	6	14	2	4
C-1-1	0	1	8	13	1	76
C-1-2	1	3	5	8	27	56
C-1-3	0	0	0	2	27	71
C-1-4	3	5	11	28	19	34
C-1-5	0	0	6	8	29	56
C-1-6	2	2	6	18	22	51
C-1-7	0	0	1	10	13	75
C-1-8	0	0	3	6	7	84
C-2-1	8	19	31	17	18	8
C-2-2	14	9	13	21	16	26
C-3-1	86	7	2	1	1	3
Mean	10	4	8	12	16	49
Median	1	2	6	10	18	56
STD	26	6	9	8	10	28
D-1-1	4	2	4	24	39	28
D-1-2	0	0	0	0	0	100
D-1-3	0	1	3	28	37	32
D-2-1	18	60	8	8	6	0
D-2-2	56	17	11	7	3	5
D-2-3	62	17	11	4	5	0
D-2-4	56	13	9	6	7	9
D-2-5	62	12	8	7	5	6
D-2-6	52	15	12	8	6	7
D-2-7	45	14	14	11	9	7
D-2-8	46	16	17	13	6	1
D-2-9	55	13	13	6	6	8
D-2-10	54	17	12	6	4	7
D-2-11	63	14	9	4	3	6
D-2-12	59	20	12	4	2	2
D-2-13	53	12	9	14	0	11
D-3-1	83	7	4	2	1	3
D-3-2	89	7	2	1	0	1
Mean	48	14	9	8	8	13
Median	54	14	9	6	5	6
STD	26	13	5	7	11	23

Table 1-5. Gradation data for limestone byproduct fines.

Sample Code	Gradation (% Retained)					
	+40	-40/+60	-60/+100	-100/+200	-200/+325	-325
E-2-1	63	11	8	8	3	6
E-2-2	57	25	8	5	2	4
E-2-3	69	14	6	4	1	5
E-2-4	21	37	21	12	0	8
Mean	53	22	11	7	1	6
Median	60	20	8	6	1	6
STD	21	12	7	4	1	2
F-1-1	0	0	1	3	0	95
F-1-2	0	0	0	4	6	89
F-1-3	0	0	4	22	27	47
F-1-4	6	6	6	18	17	48
F-1-5	1	1	2	6	7	82
F-1-6	0	0	0	1	2	97
F-2-1	5	17	27	30	11	11
F-2-2	33	21	18	15	4	10
Mean	6	6	7	12	9	60
Median	1	1	3	10	7	65
STD	11	8	10	10	9	36
G-2-1	63	8	8	10	5	6
G-2-2	49	16	13	10	4	9
G-2-3	46	23	16	7	2	6
G-2-4	23	20	17	14	0	26
G-2-5	18	26	30	17	2	7
G-2-6	44	21	14	14	3	5
G-2-7	79	11	5	3	1	2
G-3-1	91	4	1	1	0	2
Mean	52	16	13	9	2	8
Median	47	18	13	10	2	6
STD	25	8	9	6	2	8

Table 1-6. Mean byproduct fines gradation data and moment statistics for distributions.

* includes screenings (minus-4 mesh by plus-40 mesh).

Sample Catagories	Lithology	N	Mean Gradation (% Retained)					
			+40	-40/+60	-60/+100	-100/+200	-200/+325	-325
Fine:	Dol. Ls./Dol.	12	1	1	4	13	19	62
(-200 mesh)	Limestone	6	1	1	2	9	10	76
	All samples	18	1	1	3	12	16	66
Coarse:*	Dol. Ls./Dol.	28	44	14	14	15	7	6
(-3/8"/+200)	Limestone	14	47	18	14	11	3	8
	All samples	42	45	15	14	14	5	7

Sample Catagories	Lithology	N	Gradation Moment Statistics**				
			Mean (phi)	Mean (mm)	Std. Dev. (phi)	Skewness (phi)	Kurtosis (phi)
Fine:	Dol. Ls./Dol.	12	4.27	0.052	0.76	-1.87	6.62
(-200 mesh)	Limestone	6	4.42	0.047	0.72	-2.63	10.22
	All samples	18	4.32	0.050	0.75	-2.09	7.54
Coarse:*	Dol. Ls./Dol.	28	2.11	0.231	1.23	0.73	2.23
(-3/8"/+200)	Limestone	14	1.96	0.257	1.18	1.09	3.06
	All samples	42	2.06	0.240	1.22	0.84	2.46

** ϕ (phi) = $-3.322 \log_{10} S$, where S is the grain size in mm.

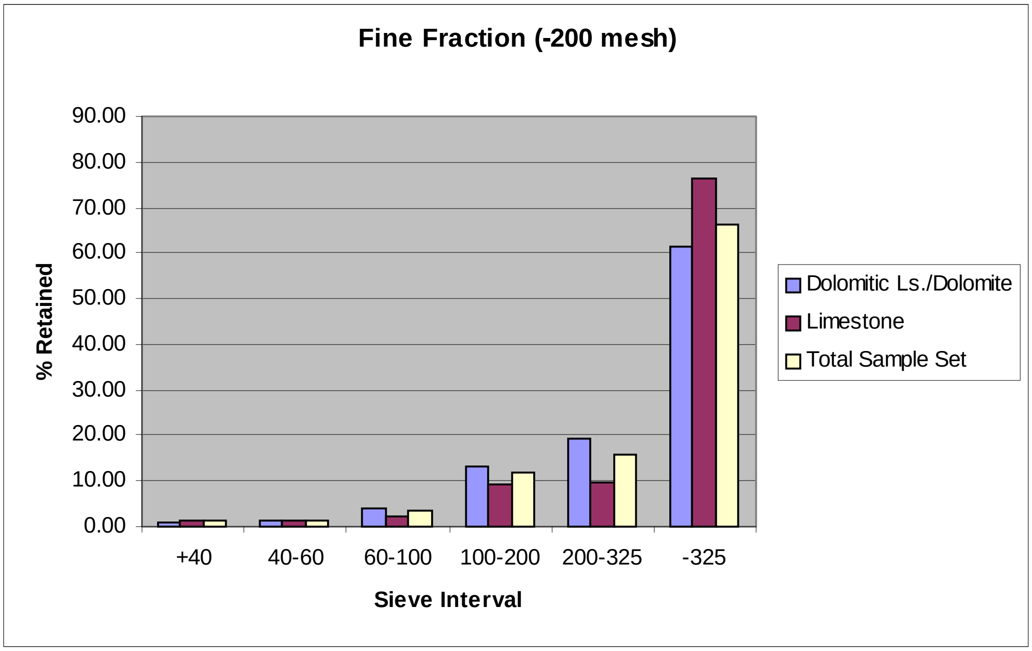

Figure 1-8. Mean particle-size distributions for fine category (minus-200 mesh) byproduct fines.

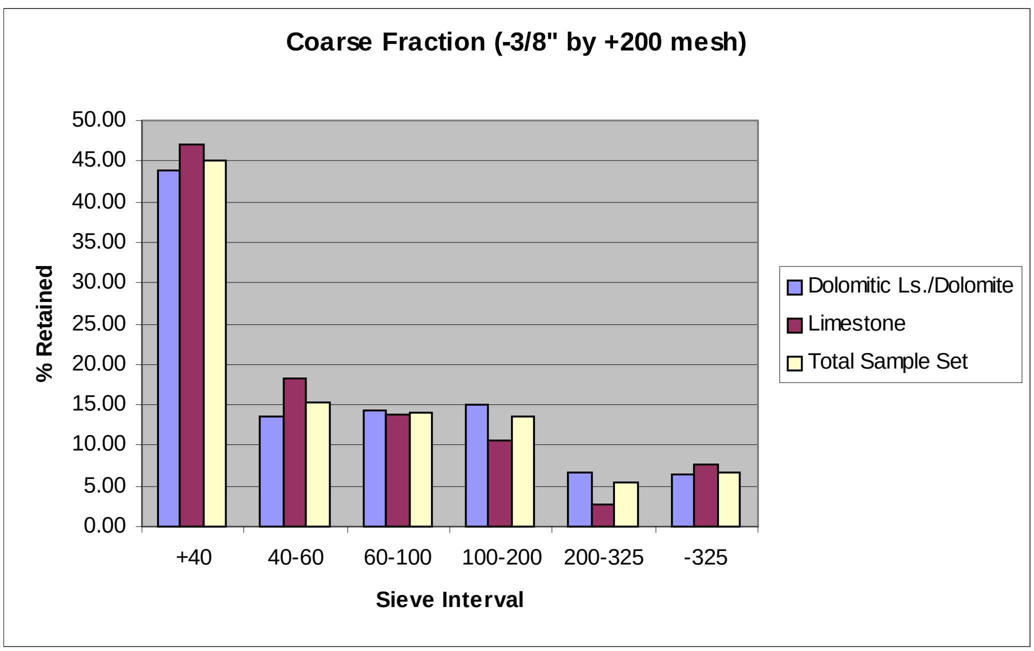

Figure 1-9. Mean particle-size distributions for coarse category (minus-3/8” by plus-200 mesh) byproduct fines.

As much of the byproduct fines material studied was minus-200 mesh (75 µm), alternative techniques to wet sieving were investigated to examine the byproduct fines. The nature of the minus-200 mesh fraction is of importance, in part, because of the presence of clays observed in the acid insoluble residue of most of the byproduct fines analyzed. Deleterious materials, such as clay, can have a major impact on construction materials that contain them. As an example, clays can greatly degrading the performance of concrete mixes through interfering with the bonding between aggregate and cement components.

Two techniques were evaluated, for this study, to characterize the minus-200 mesh fines component; the hydrometer method defined by ASTM D422 as the standard for measuring particle sizes smaller than 75 µm, and the sand equivalent test (ASTM D2419) which is used to indicate the relative proportions of clay-like or plastic fines and dust in granular soils and minus-4 mesh (4.75mm) fine aggregates. The latter test assigns an empirical value (SE) to the relative amount, fineness, and character of claylike material present in the test specimen, offering a means by which the clay content may be quantified. However, although clay content is commonly measured by the sand equivalent test, it can give misleading results if the fines are predominately minus-325 mesh dust of fractures, as would be expected with the byproduct fines sampled. Commonly used in Europe to identify deleterious fines and mineral fillers when clay content is the desired variable to be determined, the Methylene Blue Test is a more accurate alternative under these conditions, identifying clay minerals by measuring the surface activity of the fines via titration with methylene blue. However, for the purpose of this study, evaluation by the hydrometer method was deemed to be sufficient.

Hydrometer results (Table 1-7, Fig. 1-10) were consistent with gradation information derived by wet sieve analysis. The relative proportion of sand, silt, and clay, as well as the clay fraction (<2 µm)) mineralogy determined by XRD agreed well with previous results, taking into account byproduct fines lithology and the acid insoluble residue mineralogy previously noted in this study. From this data set, it is evident that a substantial clay-size fraction exists for many of the fines sampled, an important factor when considering potential application for these materials.

Table 1-7. Hydrometer results for select byproduct fines samples.

Sample Code	Grain-size Distribution			Clay Mineralogy
	Sand %	Silt %	Clay %	(< 2 micron)**
A-2-3	78.5	7.0	14.5	D>Q>S
B-2-2	56.5	38.3	5.1	D>>Q>S
C-1-7	26.4	52.2	21.4	D>>C>Q>S
D-1-3	10.8	70.5	18.7	D>>C>Q>S
E-2-4	84.9	9.6	5.6	C>>Q>S
F-2-2	67.7	22.1	10.3	C>>Q>S
G-2-6	79.1	9.7	11.1	C>>Q>S
H-1-1	41.5	43.8	14.7	C>>Q>S

** Clay mineralogy determined by XRD using ethylene glycol solvated oriented mounts (D = dolomite, C = calcite, Q = quartz, S = smectite (clay)).

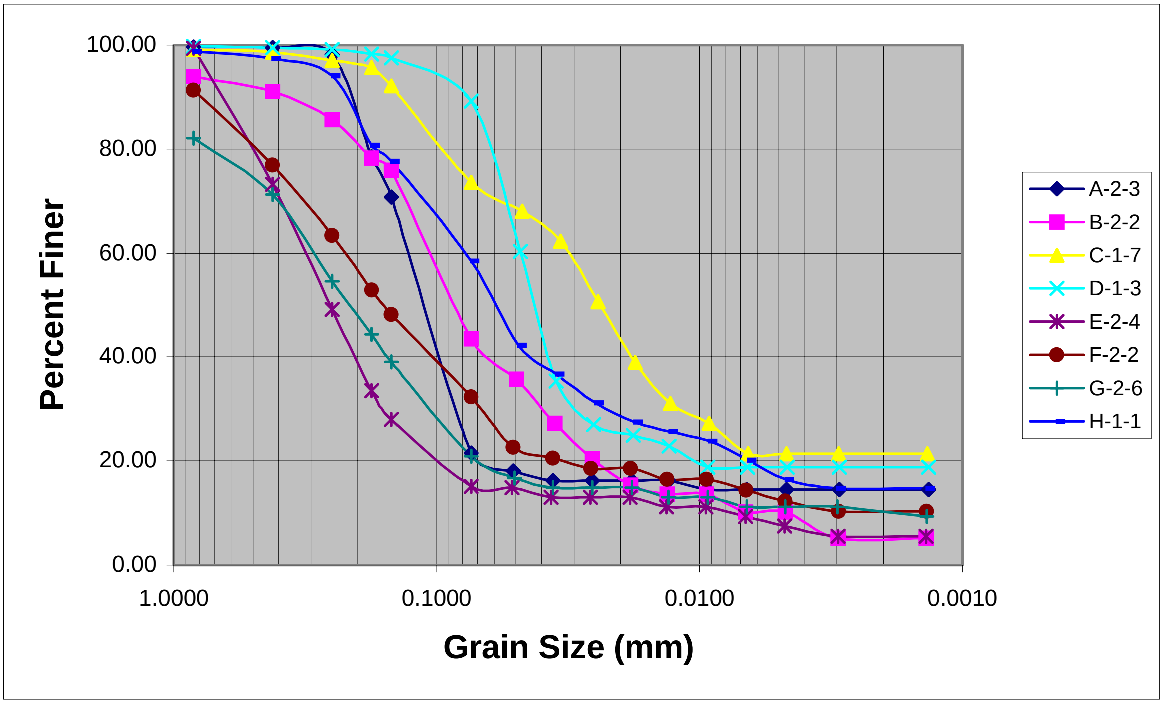

Figure 1-10. Grain-size distribution plot for byproduct fines evaluated by the hydrometer method.

Mineralogy

Byproduct fines often vary in mineralogy (and therefore chemistry) from coarse aggregate products and in response to variations in grading (Stokowski, 1992). This is particularly true for plants processing a mixture of limestone and dolomite. It has been shown by Stokowski (1992), that the finest sizes are enriched in $CaCO_3$, SiO_2, Al_2O_3, and Fe_2O_3 relative to $MgCO_3$, in response to the lower specific gravity and relative softness of calcite ($CaCO_3$) and enrichment of clay minerals (SiO_2, Al_2O_3, and Fe_2O_3). Mixed carbonate lithologies are common in Florida, relating directly to the reactivity of byproduct materials. For this reason, understanding how byproduct mineralogy varies among different lithologies and by particle-size within individual sources is required when assessing a potential byproduct fines application.

In order to evaluate the mineralogy of byproduct fines sampled for this study, and to compare the results to previously reported data on parent lithologies, XRD analyses were performed on the two finest sieve intervals collected during the gradation part of the study; the minus-200 mesh by plus-325 mesh (-200/+325) and the minus-325 mesh (-325) (Tables 1-8 and 1-9). The data collected includes quantitative mineralogy, the unit cell parameters a_0 and c_0 (calculated using a least-squares refinement), position of the $d_{(104)}$ reflection for both calcite and dolomite, and crystallite-size estimates (determined from $d_{(300)}$ values). In many cases, interferences or low concentrations prevented evaluation of all the variables of interest. For calcite and dolomite, a_0 and c_0 represent the axial dimensions of a rhombohedral unit cell, the

Sample Code	Size Fraction	Calcite %	Dolomite %	Quartz %	Rutile %	Zircon %	Calcite (d_{104}) Å	Calcite (a_0) Å	Calcite (c_0) Å	Dolomite (d_{104}) Å	Dolomite* (d_{300}) size Å	Dolomite (a_0) Å	Dolomite (c_0) Å
A-1-1	-325	17.0	83.0	0.5	0.0	0.0	3.0314	4.982	17.037	2.8951	507	4.817	16.088
A-2-1	-325	6.0	94.0	0.0	0.0	0.0	3.0313	4.985	17.033	2.8952	581	4.817	16.088
A-2-2	-325	7.0	93.0	0.5	0.0	0.0	3.0316	4.980	17.035	2.8969	498	4.817	16.080
A-2-3	-325	5.0	91.0	5.0	0.0	0.0	3.0315	4.981	17.044	2.8969	568	4.818	16.075
A-3-2	-325	18.0	81.0	0.5	0.0	0.0	3.0335	4.987	17.052	2.8952	767	4.818	16.086
	Mean	10.6	88.4	1.3	0.0	0.0	3.0319	4.983	17.040	2.8959	584	4.817	16.083
	Median	7.0	91.0	0.5	0.0	0.0	3.0315	4.982	17.037	2.8952	568	4.817	16.086
	STD	6.3	6.0	2.1	0.0	0.0	0.0009	0.003	0.008	0.0010	108	0.001	0.006
A-1-1	-200/+325	4.0	96.0	0.5	0.0	0.0	3.0332	4.983	17.034	2.8970	497	4.818	16.097
A-2-1	-200/+325	3.0	97.0	0.0	0.0	0.0	3.0313	4.982	17.033	2.8968	475	4.819	16.078
A-2-2	-200/+325	3.0	96.0	1.0	0.0	0.0	3.0297	4.987	17.031	2.8968	650	4.819	16.088
A-2-3	-200/+325	1.0	98.0	0.5	0.0	0.0	3.0334			2.8969	506	4.819	16.084
A-3-1	-200/+325	4.0	96.0	0.0	0.0	0.0	3.0317	4.984	17.028	2.8970	513	4.819	16.087
A-3-2	-200/+325	4.0	96.0	0.5	0.0	0.0	3.0316	4.984	17.037	2.8969	582	4.819	16.083
	Mean	3.2	96.5	0.4	0.0	0.0	3.0318	4.984	17.033	2.8969	537	4.819	16.086
	Median	3.5	96.0	0.5	0.0	0.0	3.0317	4.984	17.033	2.8969	510	4.819	16.086
	STD	1.2	0.8	0.4	0.0	0.0	0.0014	0.002	0.003	0.0001	66	0.000	0.006
A Total	**Mean**	6.5	92.8	0.8	0.0	0.0	3.0318	4.984	17.036	2.8964	559	4.818	16.085
	Median	4.0	96.0	0.5	0.0	0.0	3.0316	4.984	17.035	2.8969	513	4.818	16.086
	STD	5.6	5.7	1.4	0.0	0.0	0.0011	0.002	0.007	0.0008	87	0.001	0.006
B-2-1	-325	9.0	90.0	0.5	0.0	0.0	3.0316	4.985	17.056	2.8934	408	4.817	16.077
B-2-2	-325	5.0	95.0	1.0	0.0	0.0	3.0334	4.985	17.045	2.8951	442	4.817	16.088
B-2-3	-325	7.0	92.0	1.0	0.0	0.0	3.0334	4.983	17.041	2.8952	535	4.817	16.092
B-2-4	-325	8.0	91.0	1.0	0.0	0.0	3.0334	4.983	17.046	2.8970	479	4.818	16.100
B-2-5	-325	8.0	92.0	0.5	0.0	0.0	3.0335	4.986	17.039	2.8970	469	4.819	16.090
	Mean	7.4	92.0	0.8	0.0	0.0	3.0331	4.984	17.045	2.8955	467	4.818	16.089
	Median	8.0	92.0	1.0	0.0	0.0	3.0334	4.985	17.045	2.8952	469	4.817	16.090
	STD	1.5	1.9	0.3	0.0	0.0	0.0008	0.001	0.007	0.0015	47	0.001	0.008
B-2-1	-200/+325	2.0	98.0	0.5	0.0	0.0	3.0352	4.984	17.042	2.8952	426	4.817	16.082
B-2-2	-200/+325	2.0	98.0	0.5	0.0	0.0	3.0353	4.984	17.044	2.8953	612	4.818	16.075
B-2-3	-200/+325	2.0	98.0	0.5	0.0	0.0	3.0315	4.982	17.044	2.8952	442	4.817	16.080
B-2-4	-200/+325	2.0	98.0	0.5	0.0	0.0	3.0315			2.8970	590	4.818	16.093
B-2-5	-200/+325	4.0	96.0	0.0	0.0	0.0	3.0334	4.983	17.043	2.8969	460	4.818	16.089
	Mean	2.4	97.6	0.4	0.0	0.0	3.0334	4.983	17.043	2.8959	506	4.818	16.084
	Median	2.0	98.0	0.5	0.0	0.0	3.0334	4.984	17.044	2.8953	460	4.818	16.082
	STD	0.9	0.9	0.2	0.0	0.0	0.0019	0.001	0.001	0.0009	88	0.001	0.007
B Total	**Mean**	4.9	94.8	0.6	0.0	0.0	3.0332	4.984	17.044	2.8957	486	4.818	16.087
	Median	4.5	95.5	0.5	0.0	0.0	3.0334	4.984	17.044	2.8953	465	4.818	16.089
	STD	2.9	3.3	0.3	0.0	0.0	0.0014	0.001	0.005	0.0012	70	0.001	0.008
C-1-1	-325	8.0	92.0	0.5	0.0	0.0	3.0296	4.983	17.024	2.8988	564	4.821	16.114
C-1-2	-325	11.0	89.0	0.0	0.0	0.0	3.0314	4.984	17.030	2.8988	440	4.821	16.101
C-1-4	-325	6.0	94.0	0.5	0.0	0.0	3.0332	4.982	17.038	2.9006	944	4.821	16.112
C-1-5	-325	16.0	84.0	0.5	0.0	0.0	3.0296	4.985	17.026	2.9005	693	4.822	16.111
C-1-6	-325	16.0	84.0	0.5	0.0	0.0	3.0315	4.985	17.031	2.9006	685	4.822	16.100
C-1-7	-325	7.0	93.0	0.5	0.0	0.0	3.0315	4.983	17.037	2.9005	1000	4.821	16.106
C-1-8	-325	18.0	82.0	0.0	0.0	0.0	3.0296	4.983	17.038	2.9005	872	4.822	16.116
C-2-1	-325	9.0	91.0	0.0	0.0	0.0	3.0294	4.981	17.027	2.9005	1000	4.821	16.107
C-2-2	-325	4.0	96.0	0.5	0.0	0.0	3.0313	4.983	17.032	2.8988	536	4.821	16.105
C-3-1	-325	10.0	90.0	0.0	0.0	0.0	3.0314	4.983	17.042	2.8988	1000	4.821	16.104
	Mean	10.5	89.5	0.3	0.0	0.0	3.0309	4.983	17.033	2.8998	773	4.821	16.108
	Median	9.5	90.5	0.5	0.0	0.0	3.0314	4.983	17.032	2.9005	783	4.821	16.107
	STD	4.7	4.7	0.3	0.0	0.0	0.0012	0.001	0.006	0.0009	216	0.000	0.005
C-1-1	-200/+325	13.0	87.0	0.0	0.0	0.0	3.0314	4.982	17.039	2.8988	745	4.821	16.117
C-1-2	-200/+325	3.0	97.0	0.0	0.0	0.0	3.0312	4.982	17.032	2.8987	396	4.821	16.097
C-1-3	-200/+325	4.0	95.0	0.5	0.0	0.0	3.0314	4.982	17.034	2.8988	519	4.821	16.093
C-1-4	-200/+325	3.0	97.0	0.0	0.0	0.0	3.0315	4.980	17.036	2.9006	786	4.821	16.098
C-1-5	-200/+325	5.0	95.0	0.0	0.0	0.0	3.0314	4.981	17.035	2.8989	669	4.821	16.108
C-1-6	-200/+325	11.0	89.0	0.5	0.0	0.0	3.0296	4.983	17.025	2.8988	525	4.822	16.101
C-1-7	-200/+325	8.0	92.0	0.5	0.0	0.0	3.0314	4.982	17.041	2.8989	1000	4.821	16.109
C-1-8	-200/+325	18.0	82.0	0.5	0.0	0.0	3.0314	4.983	17.029	2.8989	628	4.821	16.101
C-2-1	-200/+325	4.0	96.0	0.5	0.0	0.0	3.0314	4.983	17.024	2.9005	508	4.821	16.106
C-2-2	-200/+325	6.0	93.0	0.5	0.0	0.0	3.0296			2.8952		4.819	16.107
C-3-1	-200/+325	3.0	97.0	0.5	0.0	0.0	3.0312			2.8987	702	4.820	16.093
	Mean	7.1	92.7	0.3	0.0	0.0	3.0310	4.982	17.033	2.8988	648	4.821	16.103
	Median	5.0	95.0	0.5	0.0	0.0	3.0314	4.982	17.034	2.8988	649	4.821	16.101
	STD	4.9	4.9	0.3	0.0	0.0	0.0007	0.001	0.006	0.0014	174	0.001	0.007
C Total	**Mean**	8.7	91.2	0.3	0.0	0.0	3.0310	4.983	17.033	2.8993	711	4.821	16.105
	Median	8.0	92.0	0.5	0.0	0.0	3.0314	4.983	17.032	2.8989	689	4.821	16.106
	STD	5.0	5.0	0.2	0.0	0.0	0.0010	0.001	0.006	0.0013	201	0.001	0.007

Table 1-8. Summary of x-ray diffraction (XRD) data for dolomitic limestone/dolomite byproduct fines.

* Crystallite size values >1000 treated as equal to 1000.

Sample Code	Size Fraction	Calcite %	Dolomite %	Quartz %	Rutile %	Zircon %	Calcite (d_{104}) Å	Calcite (a_0) Å	Calcite (c_0) Å	Dolomite (d_{104}) Å	Dolomite* (d_{300}) size Å	Dolomite (a_0) Å	Dolomite (c_0) Å
D-1-1	-325	23.0	77.0	0.5	0.0	0.0	3.0333	4.983	17.040	2.8990	469	4.820	16.115
D-1-2	-325	21.0	79.0	0.0	0.0	0.0	3.0314	4.982	17.044	2.8988	801	4.821	16.106
D-1-3	-325	9.0	91.0	0.0	0.0	0.0	3.0315	4.982	17.040	2.8988	786	4.821	16.113
D-2-1	-325	3.0	96.0	2.0	0.0	0.0	3.0333	4.982	17.050	2.9005	463	4.822	16.111
D-2-2	-325	9.0	91.0	0.0	0.0	0.0	3.0315	4.983	17.021	2.9006	689	4.823	16.122
D-2-3	-325	2.0	96.0	3.0	0.0	0.0	3.0316			2.9005	1000	4.824	16.114
D-2-4	-325	8.0	92.0	0.0	0.0	0.0	3.0314	4.985	17.023	2.9005	743	4.823	16.113
D-2-5	-325	16.0	84.0	0.0	0.0	0.0	3.0296	4.984	17.021	2.9007	564	4.822	16.129
D-2-6	-325	8.0	92.0	0.5	0.0	0.0	3.0297	4.981	17.059	2.9006	634	4.822	16.153
D-2-7	-325	10.0	90.0	0.0	0.0	0.0	3.0313	4.982	17.030	2.8987	573	4.821	16.102
D-2-8	-325	14.0	86.0	0.5	0.0	0.0	3.0332	4.984	17.037	2.9006	1000	4.823	16.116
D-2-9	-325	15.0	85.0	0.0	0.0	0.0	3.0314	4.983	17.035	2.9006	496	4.823	16.112
D-2-11	-325	13.0	87.0	0.0	0.0	0.0	3.0314	4.986	17.027	2.8989	540	4.822	16.131
D-2-12	-325	26.0	73.0	0.5	0.0	0.0	3.0334	4.985	17.028	2.8990	811	4.820	16.152
D-2-13	-325	10.0	89.0	1.0	0.0	0.0	3.0315	4.984	17.033	2.8988	824	4.821	16.103
D-3-1	-325	18.0	82.0	0.0	0.0	0.0	3.0336	4.985	17.034	2.9006	1000	4.822	16.096
D-3-2	-325	20.0	80.0	0.5	0.0	0.0	3.0315	4.983	17.041	2.8988	1000	4.822	16.104
	Mean	13.2	86.5	0.5	0.0	0.0	3.0318	4.983	17.035	2.8998	729	4.822	16.117
	Median	13.0	87.0	0.0	0.0	0.0	3.0315	4.983	17.035	2.9005	743	4.822	16.113
	STD	6.8	6.6	0.8	0.0	0.0	0.0012	0.001	0.010	0.0009	195	0.001	0.016
D-1-1	-200/+325	10.0	90.0	1.0	0.0	0.0	3.0295	4.983	17.037	2.8988	753	4.820	16.128
D-1-3	-200/+325	3.0	97.0	0.0	0.0	0.0	3.0296			2.8988	1000	4.820	16.115
D-2-1	-200/+325	2.0	97.0	1.0	0.0	0.0	3.0314			2.9006	954	4.823	16.109
D-2-2	-200/+325	3.0	97.0	0.5	0.0	0.0	3.0296			2.8989	722	4.822	16.106
D-2-3	-200/+325	2.0	97.0	1.0	0.0	0.0	3.0316			2.9006	695	4.823	16.119
D-2-4	-200/+325	2.0	98.0	0.5	0.0	0.0	3.0376			2.9005	910	4.823	16.112
D-2-5	-200/+325	5.0	95.0	0.0	0.0	0.0	3.0295	4.982	17.036	2.8989	554	4.822	16.113
D-2-6	-200/+325	3.0	97.0	0.5	0.0	0.0	3.0336			2.8989	806	4.821	16.115
D-2-7	-200/+325	6.0	94.0	0.0	0.0	0.0	3.0314	4.985	17.018	2.8988	560	4.821	16.118
D-2-8	-200/+325	5.0	94.0	1.0	0.0	0.0	3.0314	4.984	17.018	2.8989	629	4.821	16.112
D-2-9	-200/+325	6.0	94.0	0.5	0.0	0.0	3.0315	4.984	17.038	2.9006	796	4.822	16.113
D-2-10	-200/+325	14.0	86.0	0.5	0.0	0.0	3.0314	4.984	17.035	2.9007	1000	4.822	16.117
D-2-11	-200/+325	7.0	92.0	1.0	0.0	0.0	3.0332	4.983	17.047	2.9005	901	4.822	16.114
D-2-12	-200/+325	12.0	87.0	1.0	0.0	0.0	3.0334	4.984	17.027	2.9006	1000	4.822	16.126
D-3-1	-200/+325	14.0	86.0	0.0	0.0	0.0	3.0335	4.982	17.044	2.8989	760	4.821	16.117
D-3-2	-200/+325	9.0	91.0	0.0	0.0	0.0	3.0316	4.983	17.030	2.9005	475	4.820	16.106
	Mean	6.4	93.3	0.5	0.0	0.0	3.0319	4.983	17.033	2.8997	782	4.822	16.115
	Median	5.5	94.0	0.5	0.0	0.0	3.0315	4.984	17.036	2.8997	778	4.822	16.115
	STD	4.2	4.2	0.4	0.0	0.0	0.0021	0.001	0.010	0.0009	170	0.001	0.006
D Total	**Mean**	9.9	89.8	0.5	0.0	0.0	3.0318	4.983	17.034	2.8997	755	4.822	16.116
	Median	9.0	91.0	0.5	0.0	0.0	3.0315	4.983	17.035	2.9005	760	4.822	16.114
	STD	6.6	6.5	0.7	0.0	0.0	0.0017	0.001	0.010	0.0009	183	0.001	0.012

Table 1-8. (cont.) Summary of x-ray diffraction (XRD) data for dolomitic limestone/dolomite byproduct fines.

* Crystallite size values >1000 treated as equal to 1000.

smallest volume within the three-dimensional repetitive pattern of a crystal that contains a complete sample of the atomic or molecular groups that compose a mineral.

In order to calculate the quantitative mineralogy of the fines samples, the matrix flushing method (Chung, 1974), using an α-alumina internal standard for pattern correction, was used. The matrix flushing method gives an exact relationship between x-ray intensity data and phase concentration, independent of matrix effects. Published reference intensity ratios for the major phases identified in the samples (calcite, dolomite, quartz, rutile, and zircon) were used to calculate mineral concentrations according to the following equation:

$$P = (X_s/K_s) \times (I_x/I_s)$$

where,

P = wt. fraction of phase x in the sample

X_s = wt. fraction of the internal standard in the sample

K_s = the reference intensity ratio of phase x
I_x = measured intensity of I_{100} line of phase x
I_s = measured intensity of I_{100} line of internal standard

Table 1-9. Summary of x-ray diffraction (XRD) data for limestone byproduct fines.
* Crystallite size values >1000 treated as equal to 1000.

Sample Code	Size Fraction	Calcite %	Dolomite %	Quartz %	Rutile %	Zircon %	Calcite (d_{104}) Å	Calcite* (d_{300}) size Å	Calcite (a_0) Å	Calcite (c_0) Å	Dolomite (d_{104}) Å
E-2-2	-325	95.0	1.0	4.0	0.0	0.0	3.0335	1000	4.987	17.053	2.9023
E-2-3	-325	91.0	3.0	7.0	0.0	0.0	3.0336	909	4.987	17.051	2.8973
E-2-4	-325	86.0	1.0	13.0	0.0	0.0	3.0336	940	4.987	17.056	2.9014
	Mean	90.7	1.7	8.0	0.0	0.0	3.0336	950	4.987	17.053	2.9003
	Median	91.0	1.0	7.0	0.0	0.0	3.0336	940	4.987	17.053	2.9014
	STD	4.5	1.2	4.6	0.0	0.0	0.0001	46	0.000	0.003	0.0027
E-2-1	-200/+325	79.0	6.0	15.0	0.0	0.0	3.0336	682	4.988	17.050	2.8972
E-2-2	-200/+325	71.0	3.0	26.0	0.0	0.0	3.0336	526	4.987	17.056	2.8989
E-2-3	-200/+325	75.0	3.0	22.0	0.5	0.0	3.0353	645	4.987	17.054	2.9022
E-2-4	-200/+325	73.0	3.0	24.0	0.0	0.0	3.0336	775	4.988	17.049	2.8985
	Mean	74.5	3.8	21.8	0.1	0.0	3.0340	657	4.988	17.052	2.8992
	Median	74.0	3.0	23.0	0.0	0.0	3.0336	664	4.988	17.052	2.8987
	STD	3.4	1.5	4.8	0.3	0.0	0.0008	103	0.001	0.003	0.0021
E Total	**Mean**	81.4	2.9	15.9	0.1	0.0	3.0338	782	4.987	17.053	2.8997
	Median	79.0	3.0	15.0	0.0	0.0	3.0336	775	4.987	17.053	2.8989
	STD	9.3	1.7	8.5	0.2	0.0	0.0006	175	0.000	0.003	0.0022
F-1-1	-325	94.0	4.0	2.0	0.0	0.0	3.0335	814	4.987	17.052	2.9040
F-1-2	-325	92.0	6.0	2.0	0.0	0.0	3.0334	788	4.986	17.051	2.8988
F-1-3	-325	88.0	9.0	3.0	0.0	0.0	3.0336	826	4.988	17.050	2.9006
F-1-4	-325	89.0	4.0	6.0	0.5	0.0	3.0335	906	4.987	17.052	2.8989
F-1-5	-325	91.0	7.0	2.0	0.0	0.0	3.0336	577	4.987	17.053	2.8988
F-1-6	-325	97.0	2.0	1.0	0.0	0.0	3.0316	863	4.986	17.047	2.8936
F-2-1	-325	76.0	17.0	7.0	0.0	0.0	3.0336	513	4.987	17.049	2.8972
F-2-2	-325	94.0	3.0	3.0	0.0	0.0	3.0335	610	4.988	17.049	2.9007
	Mean	90.1	6.5	3.3	0.1	0.0	3.0333	737	4.987	17.050	2.8991
	Median	91.5	5.0	2.5	0.0	0.0	3.0335	801	4.987	17.051	2.8989
	STD	6.4	4.8	2.1	0.2	0.0	0.0007	148	0.001	0.002	0.0030
F-1-1	-200/+325	76.0	10.0	13.0	1.0	0.0	3.0335	618	4.986	17.051	2.9003
F-1-2	-200/+325	58.0	31.0	11.0	0.0	0.0	3.0353	973	4.986	17.050	2.9006
F-1-3	-200/+325	69.0	6.0	26.0	0.0	0.0	3.0334	966	4.987	17.044	2.9023
F-1-4	-200/+325	81.0	13.0	6.0	0.0	0.0	3.0334	1000	4.987	17.056	2.8970
F-1-5	-200/+325	80.0	15.0	5.0	0.0	0.0	3.0354	955	4.987	17.053	2.9004
F-1-6	-200/+325	86.0	4.0	8.0	2.0	0.0	3.0353	772	4.987	17.049	2.9006
F-2-1	-200/+325	63.0	19.0	14.0	4.0	0.0	3.0353	840	4.988	17.052	2.9006
F-2-2	-200/+325	64.0	11.0	25.0	0.0	0.0	3.0336	861	4.987	17.057	2.9025
	Mean	72.1	13.6	13.5	0.9	0.0	3.0344	873	4.987	17.052	2.9005
	Median	72.5	12.0	12.0	0.0	0.0	3.0345	908	4.987	17.052	2.9006
	STD	10.0	8.5	8.1	1.5	0.0	0.0010	130	0.001	0.004	0.0017
F Total	**Mean**	81.1	10.1	8.4	0.5	0.0	3.0338	805	4.987	17.051	2.8998
	Median	83.5	8.0	6.0	0.0	0.0	3.0336	833	4.987	17.051	2.9005
	STD	12.4	7.6	7.8	1.1	0.0	0.0010	152	0.001	0.003	0.0025
G-2-1	-325	89.0	5.0	5.0	0.0	1.0	3.0336	596	4.986	17.050	2.8949
G-2-2	-325	98.0	1.0	2.0	0.0	0.0	3.0332	731	4.987	17.051	2.9006
G-2-3	-325	98.0	0.0	2.0	0.0	0.0	3.0352	548	4.987	17.054	
G-2-4	-325	73.0	1.0	20.0	1.0	5.0	3.0333	891	4.988	17.046	2.8986
G-2-5	-325	97.0	0.0	3.0	0.0	0.0	3.0334	894	4.987	17.049	
G-2-6	-325	80.0	7.0	12.0	1.0	1.0	3.0335	864	4.986	17.047	2.8987
G-2-7	-325	93.0	3.0	3.0	1.0	0.0	3.0353	577	4.987	17.049	2.8970
G-3-1	-325	93.0	2.0	5.0	0.5	0.0	3.0334	953	4.987	17.048	2.8969
	Mean	90.1	2.4	6.5	0.4	0.9	3.0339	757	4.987	17.049	2.8978
	Median	93.0	1.5	4.0	0.3	0.0	3.0335	798	4.987	17.049	2.8978
	STD	9.1	2.5	6.3	0.5	1.7	0.0009	164	0.001	0.002	0.0020
G-2-1	-200/+325	59.0	6.0	29.0	6.0	0.0	3.0335	1000	4.988	17.044	2.8971
G-2-2	-200/+325	77.0	2.0	20.0	1.0	0.0	3.0354	869	4.989	17.046	2.8973
G-2-3	-200/+325	79.0	1.0	20.0	0.0	0.0	3.0335	823	4.987	17.051	2.8965
G-2-4	-200/+325	61.0	2.0	37.0	0.5	0.0	3.0335	809	4.988	17.053	2.8953
G-2-5	-200/+325	61.0	1.0	36.0	1.0	0.0	3.0335	1000	4.987	17.052	2.8971
G-2-6	-200/+325	63.0	6.0	28.0	1.0	2.0	3.0335	1000	4.986	17.052	2.8990
G-2-7	-200/+325	79.0	2.0	18.0	1.0	0.0	3.0333	651	4.987	17.050	2.8970
G-3-1	-200/+325	55.0	4.0	41.0	0.0	0.0	3.0336	1000	4.988	17.050	2.8968
	Mean	66.8	3.0	28.6	1.3	0.3	3.0337	894	4.988	17.050	2.8970
	Median	62.0	2.0	28.5	1.0	0.0	3.0335	935	4.988	17.051	2.8971
	STD	9.9	2.1	8.8	1.9	0.7	0.0007	129	0.001	0.003	0.0010
G Total	**Mean**	78.4	2.7	17.6	0.9	0.6	3.0338	825	4.987	17.050	2.8973
	Median	79.0	2.0	19.0	0.8	0.0	3.0335	867	4.987	17.050	2.8971
	STD	15.2	2.2	13.6	1.4	1.3	0.0008	159	0.001	0.003	0.0015

Examination of the XRD data for the dolomitic limestone/dolomite byproduct fines (Table 1-8) shows that for the entire data set, the mean dolomite concentration is 91.3 percent (N = 75, Max = 98%, Min = 73%, Std = 5.8%) and the mean calcite concentration is 8.4 percent (N = 75, Max = 26%, Min = 1%, Std = 5.8%). Trace amounts of quartz are found in many samples (Max = 5%), but no rutile or zircon was found, likely the result of only a very minor detrital flux to these lithologies during deposition. Comparing individual quarries, B has the highest mean dolomite concentration of 94.8 percent (N = 10, Max = 98%, Min = 90%, Std = 3.3%), and correspondingly the lowest mean calcite concentration of 4.9 percent (N = 10, Max = 9%, Min = 2%, Std = 2.9%) as the two phases correlate negatively. The remainder of the quarries, in descending order of mean dolomite concentration are A (92.8%, N = 11, Max = 98%, Min = 81%, Std = 5.7%), C (91.2%, N = 21, Max = 97%, Min = 82%, Std = 5.0%), and D (89.8%, N = 33, Max = 98%, Min = 73%, Std = 6.5%).

Dolomite and calcite concentrations also differ between the two sieve intervals studied. For the entire dolomitic limestone/dolomite data set, mean dolomite concentrations are greater in the –200/+325 verses the –325, ranging from 94.2 percent (N = 38, Max = 98%, Min = 82%, Std = 4.1%) to 88.3 percent (N = 37, Max = 96%, Min = 73%, Std = 5.8%), respectively. The opposite is true for calcite concentrations. The same observations hold true, as well, for the individual quarries. The relative concentration of calcite in the –325 samples supports the conclusion of Stokowski (1992) previously noted in this section. Apparently, the lower specific gravity and relative softness of calcite, as well as a potentially finer grain-size for calcite mud in these lithologies, are the primary causes for the mineralogy difference.

Dolomite unit cell axial dimensions a_0 and c_0 from the dolomitic limestone/dolomite sample set exhibit a bimodal distribution consistent with spatial/formation differences. A review of the data (Table 1-8, Figs. 1-11 and 1-12) shows that quarries A and B possess similar mean a- (4.818Å for both) and c-values (16.085Å and 16.087Å, respectively), while quarries C and D exhibit a similar correlation (a-values = 4.821Å and 4.822Å, respectively; c-values = 16.105Å and 16.116Å, respectively). This relationship is further supported by correlations in mean dolomite crystallite-size values for quarries A and B (559Å and 486Å, respectively) and for quarries C and D (711Å and 755Å, respectively). These observed differences in the dolomite crystal chemistry between quarries A and B and quarries C and D are directly linked to the different geological formations associated with each group (Table 1-1). A and B, in Taylor County, are mining material from the Suwannee Limestone of Oligocene age, while C and D, in Levy County, are mining from the Eocene age Avon Park Formation.

The differences in the dolomite from the two formations in question can be due to a variety of variables, including cation order/disorder, structural defects, and cation substitution (Goldsmith and Graf, 1958a; b; Reeder and Sheppard, 1984). Primary among these variables is the substitution of excess Ca on Mg sites in a non-stoichiometric dolomite with a corresponding reduction in crystalline order (Malone et al., 1994; 1996). The unit cell manifestation of this is an increase in the unit cell parameters with a corresponding increase in cell volume and crystallite size. Other cation substitutions also can generate similar cell expansion, such as Fe substitution. In the case of these dolomites, quarry A and B dolomites possess a more contracted unit cell (smaller a- and c-values), corresponding to increased cation ordering, while quarry C and D dolomites are less ordered and likely more reactive (increased solubility, etc.).

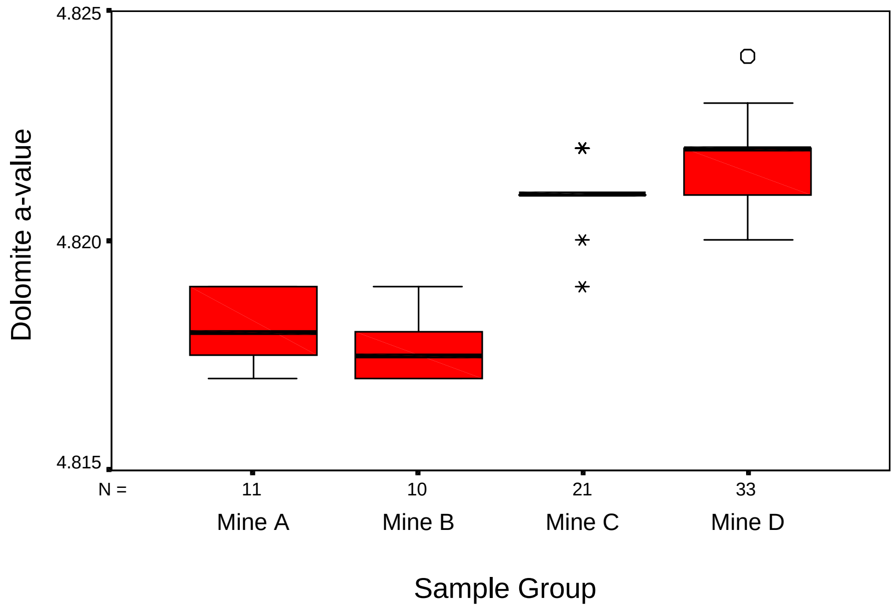

Figure 1-11. Box and whisker diagram of dolomite a-values for dolomitic limestone/dolomite fines (outlier samples are indicated by circles or asterisks).

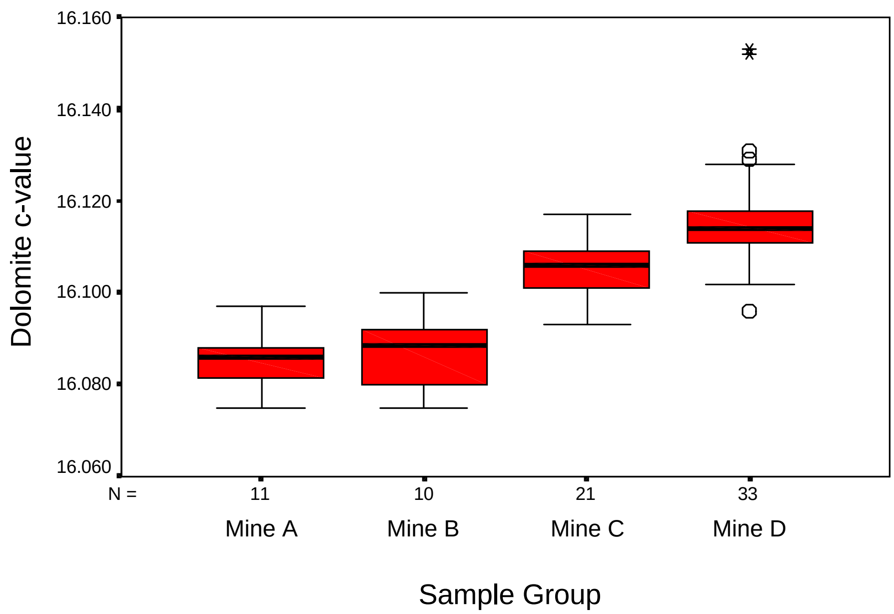

Figure 1-12. Box and whisker diagram of dolomite c-values for dolomitic limestone/dolomite fines (outlier samples are indicated by circles or asterisks).

Examination of the XRD data for the limestone byproduct fines (Table 1-9) shows that for the entire data set, the mean calcite concentration is 80.1 percent (N = 39, Max = 98%, Min = 55%, Std = 12.9%) and the mean dolomite concentration is 5.7 percent (N = 39, Max = 31%, Min = 0%, Std = 6.2%). Comparing individual quarries, E has the highest mean calcite concentration of 81.4 percent (n = 7, Max = 95%, Min = 71%, Std = 9.3%), followed by F at 81.1 percent (N = 16, Max = 97%, Min = 58%, Std = 12.4%) and G at 78.4 percent (N = 16, Max = 98%, Min = 55%, Std = 15.2%). Quarry F has, by far, the highest dolomite concentration of 10.1 percent (N = 16, Max = 31%, Min = 2%, Std = 7.6%) associated with the lowest quartz concentration.

Significant concentrations of quartz are found in most samples, with quarry G possessing the highest mean concentration of 17.6 percent (N = 16, Max = 41%, Min = 2%, Std = 13.6%), followed by E at 15.9 percent (N = 7, Max = 26%, Min = 4%, Std = 8.5) and F at 8.4 percent (N = 16, Max = 26%, Min = 1%, Std = 7.8). In all cases, quartz concentrations are significantly higher in the coarser –200/+325 fraction as compared to the finer –325 fraction. Rutile and zircon concentrations are sporadic in the limestone byproduct fines, but seem to be most prevalent in the quarry G samples, correlating well with the highest quartz values, and subsequently, the samples containing the greatest detrital flux.

Differences in calcite and dolomite concentration between the two sieve intervals studied follow the same trends observed with the dolomitic limestone/dolomite samples. For the entire limestone data set, mean calcite concentrations are greater in the –325 verses the –200/+325, ranging from 90.2 percent (N = 19, Max = 98, Min = 73, Std = 7.1) to 70.5 percent (N = 20, Max = 86, Min = 55, Std = 9.2), respectively. As was seen with the dolomitic limestone/dolomite samples, the opposite relationship is true for dolomite concentrations, and both observations hold for the individual quarries. Again, the distribution of calcite and dolomite within this sample set supports the previously noted observation of Stokowski (1992).

Based on a review of the data, calcite unit cell values from the limestone sample set seem to group together as a single population significantly different from the calcite unit cell values calculated for the dolomitic limestone/dolomite sample set (Tables 1-8 and 1-9; Figs. 1-13 and 1-14). The limestone fines possess mean calcite a- and c-values of 4.987Å (N = 39, Std = 0.001Å) and 17.051Å (N = 39, Std = 0.003Å), respectively, for the entire data set, and show no significant differences among the three quarries or the two sieve intervals. In comparison, the dolomitic limestone/dolomite samples possess mean calcite a- and c-values of 4.983Å (N = 64, Std = 0.002Å) and 17.036Å (N = 64, Std = 0.009Å), respectively, for the entire data set. Again, no differences were noted among the four quarries or the two sieve intervals. A comparison of the unit cell values for stoichiometric calcite (a = 4.989Å and c = 17.062Å) to those obtained for the two fines data sets indicates that the limestone fines calcite is very similar to stoichiometric calcite, whereas the calcite from the dolomitic limestone/dolomite samples has significantly larger unit cell dimensions, and correspondingly, a larger cell volume. This latter observation is consistent with Mg substitution for Ca in the calcite structure toward a metastable, high-Mg calcite composition (Goldsmith and Graf, 1958a), which would be characterized by greater chemical reactivity/instability.

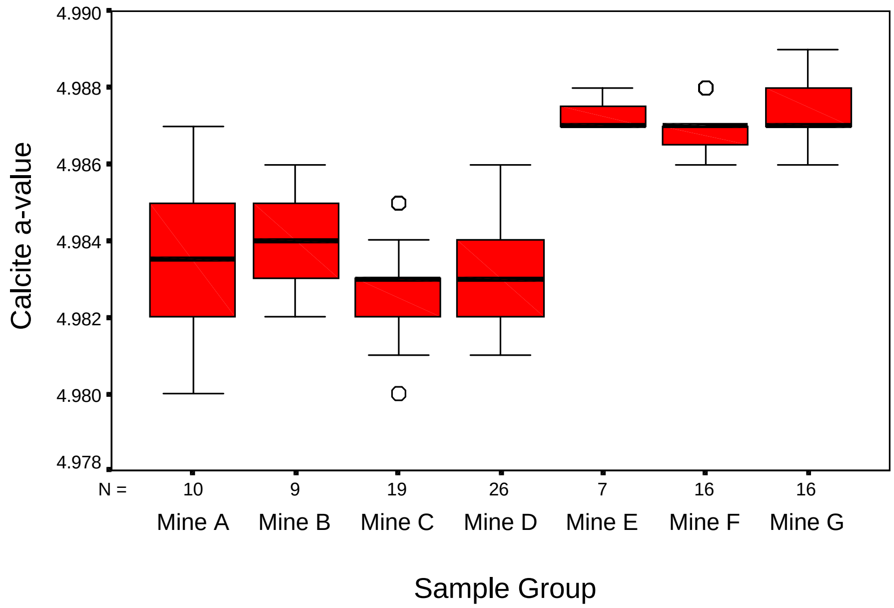

Figure 1-13. Box and whisker diagram of calcite a-values for dolomitic limestone/dolomite and limestone fines (outlier samples are indicated by circles or asterisks).

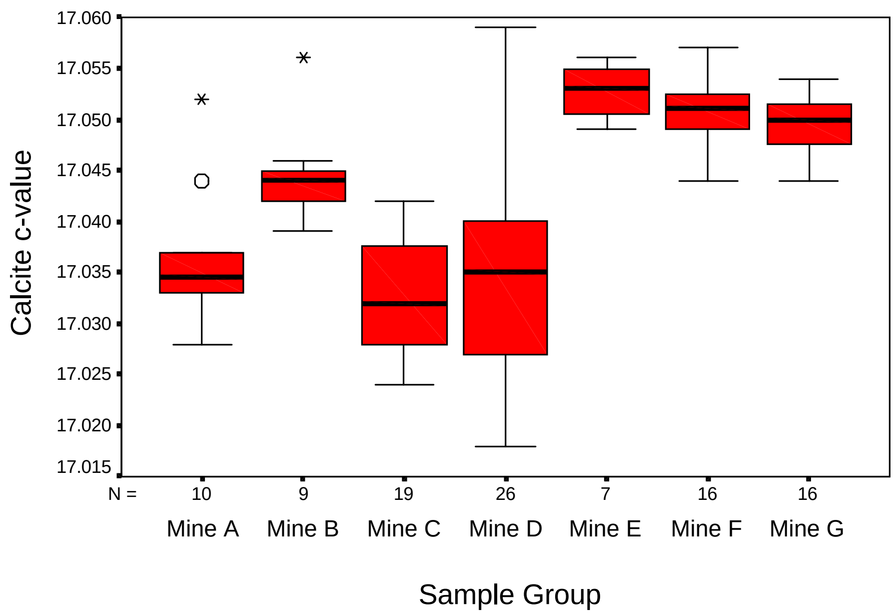

Figure 1-14. Box and whisker diagram of calcite c-values for dolomitic limestone/dolomite and limestone fines (outlier samples are indicated by circles or asterisks).

Differences in the position of the calcite and dolomite $d_{(104)}$ peak as measured by XRD have, for some time, also been used as an indicator of crystalline order and/or cation substitutions (Goldsmith and Graf, 1958a; b). The first of these, the calcite $d_{(104)}$ peak values are consistent with the previous observations differentiating between the two calcite populations (Tables 1-8 and 1-9; Fig. 1-15). A review of the data shows that for the entire dolomitic limestone/dolomite data set, the mean calcite $d_{(104)}$ is 3.0318Å (N = 75, Max = 3.0376Å, Min = 3.0294Å, Std = 0.0015Å), while for the entire limestone data set, the mean $d_{(104)}$ is 3.0338Å (N = 39, Max = 3.0354Å, Min = 3.0316Å, Std = 0.0008Å). Although not as definitive as the unit cell values, the peak shift toward a lower d-spacing does suggest that the calcite in the dolomitic limestone/dolomite fines are likely a more Mg substituted variety than that measured in the limestone fines. Such a trend is consistent with the work of Goldsmith and Graf (1958a; 1958b), and corresponds with the Mg substitution trend associated with the calcite-dolomite solid-solution series.

Although dolomite concentrations in the limestone fines were too small to give the XRD data resolution necessary for unit cell determinations, dolomite $d_{(104)}$ peak values were measured and compared to dolomite $d_{(104)}$ peak values from the dolomitic limestone/dolomite samples. As is the case with calcite, the dolomite $d_{(104)}$ peak position has long been used as an indicator of crystalline order and/or cation substitutions (Goldsmith and Graf, 1958a; b). The results indicate that the dolomite in the limestone fines is similar in crystal chemical character to the dolomite observed with quarries C and D mined from the Avon Park Formation (Tables 1-8 and 1-9; Fig. 1-16). For the entire limestone fines data set (quarries E, F, and G), the mean dolomite $d_{(104)}$ peak value is 2.8989Å (N = 37, Max = 2.9040Å, Min = 2.8936Å, Std = 0.0024Å), significantly larger than the stoichiometric dolomite $d_{(104)}$ peak value of 2.8880Å. This correlates well with the mean dolomite $d_{(104)}$ peak value of 2.8996Å (N = 54, Max = 2.9007Å, Min = 2.8952Å, Std = 0.0011Å) for dolomitic limestone/dolomite quarries C and D, but not with the mean value of 2.8961Å (N = 21, Max = 2.8970Å, Min = 2.8934Å, Std = 0.0011Å) from quarries A and B. As a result, it appears that the dolomite in the limestone fines (quarries E, F, and G) is very similar to that from quarries C and D, and therefore, is likely a less ordered and thereby more reactive dolomite phase than that measured from quarries A and B.

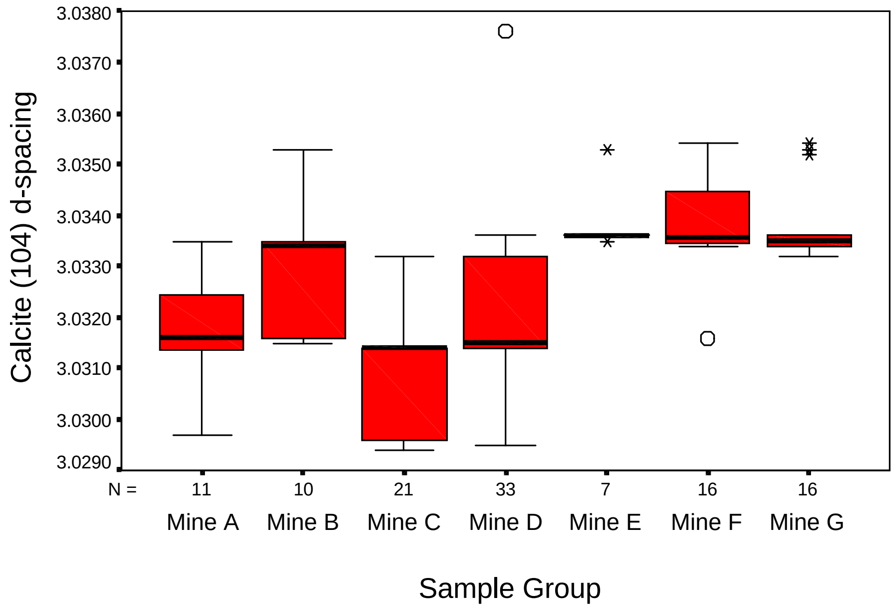

Figure 1-15. Box and whisker diagram of calcite $d_{(104)}$ values for dolomitic limestone/dolomite and limestone fines (outlier samples are indicated by circles or asterisks).

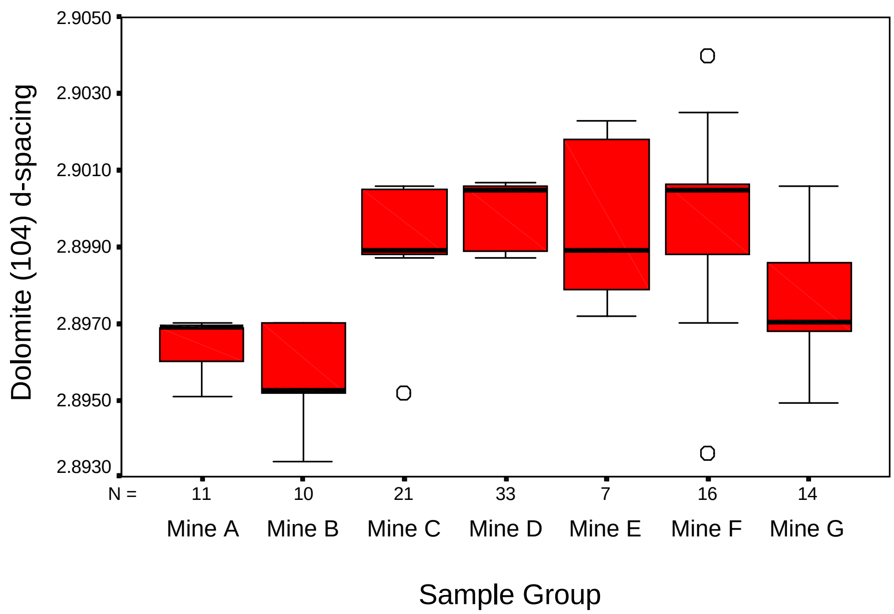

Figure 1-16. Box and whisker diagram of dolomite $d_{(104)}$ values for dolomitic limestone/dolomite

and limestone fines (outlier samples are indicated by circles or asterisks).

CONCLUSIONS AND RECOMMENDATIONS

Evaluation of the results from this section of the study, which focused on identifying the volume and characteristics of byproduct fines produced annually in the state, as well as estimating the quantity and characteristics of byproduct fines already stored at quarries, provided the following conclusions:

- Although the response to the questionnaire developed to quantify the byproduct fines problem in Florida was less than hoped for (40% of companies contacted responded), the data that was provided covered large geographic areas of the state and a significant proportion of total state production.

- Byproduct fines were classified into two categories; a coarse class (minus-3/8" by plus-200 mesh) that includes commercial "screenings", and a fine class (minus-200 mesh). The weighted average of coarse fines production was determined to be 14.9 percent of the total annual mine production in Florida, with the minus-200 fines possessing a 14.2 percent weighted average.

- Producers responding to the questionnaire tended to sell 78 percent of the coarse class of fines, but only 34 percent of the minus-200 mesh fines, and although the quantity of coarse fines produced annually was much greater than the minus-200 mesh fines, producers recognized the greater waste and storage problem represented by the fine class of fines. In fact, the projected cumulative production of fines from the year 2000 to 2010 is expected to be approximately 300 million tons (154 million tons of minus-3/8" material and 146 million tons of minus-200 mesh).

- Fines samples, divided into limestone and dolomitic limestone/dolomite lithologies were examined to determine moisture content, acid insoluble content, gradation, and mineralogy, in order to more thoroughly characterize the nature and variability of byproduct fines from around the state. As a result, the following observations were made:
    - Fines produced in Florida have inherently high and variable moisture contents, with dolomitic limestone/dolomite fines possessing a mean moisture content of 16.8 percent, limestone fines 18.4 percent, and the total data set 17.3 percent. Variations observed in the data are due, in great part, to varying ages of fines, storage methods (stockpile vs. quarry pit), and particle-size differences.
    - Acid insoluble contents show limestone fines to possess much greater values (mean = 11.4%) compared to dolomitic limestone/dolomite fines (mean = 2.1%).

Overwhelmingly, the acid insoluble fraction is dominated by quartz, followed by clays, pyrite, rutile, and goethite.

- Gradation analysis show that both the coarse and fine category of fines vary considerably in particle-size distribution. Fine category limestone fines tend to possess a particle-size distribution with slightly more minus-325 material than found in the equivalent dolomitic limestone/dolomite samples, while coarse limestone fines tend to contain more material at both extremes of the particle-size distribution. Hydrometer analysis were consistent with the gradation information derived by wet sieve analysis, and indicated a substantial clay-size fraction in many samples, an important observation when evaluating potential applications.
- X-ray diffraction (XRD) analyses performed on minus-200 mesh by plus-325 mesh and minus-325 mesh samples illustrated substantial mineralogical differences between dolomitic limestone/dolomite and limestone lithologies.
    - For the dolomitic limestone/dolomite fines, the mean dolomite concentration was 91.3 percent, with a mean calcite concentration of 8.4 percent. Only trace quantities of quartz were observed, suggesting a very low detrital flux. Calcite was more concentrated in the fine fraction (minus-325 mesh), an observation repeated in the limestone fines studied. Also, dolomite unit cell values and crystallite-size values indicate two dolomite populations, quarry A and B dolomite (Suwannee Limestone, Taylor County) corresponding to a contracted unit cell and greater cation ordering, and quarry C and D dolomite (Avon Park Fm., Levy County) characterized by less ordering and greater reactivity.
    - For the limestone fines, the mean calcite concentration was 80.1 percent, with a mean dolomite concentration of 5.7 percent. Significant quartz concentrations were found in most samples, with concentrations higher in the coarser fraction studied (minus-200 mesh by plus-325 mesh). Calcite unit cell values and $d_{(104)}$ peak positions from all the limestone fines grouped together in a population significantly different from the calcite observed in the dolomitic limestone/dolomite samples, which appeared to have greater Mg substitution for Ca in the calcite structure. Furthermore, dolomite $d_{(104)}$ peak positions determined from the limestone fines data show the small quantities of dolomite in these samples to be similar in crystal chemical character to dolomites from quarries C and D.

Summary of Conclusions

The accumulation of byproduct fines continues to be a major waste and storage problem for the coarse aggregate industry in Florida. Produced at rates as great as 300 million tons over the next 10 years, careful characterization is necessary in order to identify high volume economic application for these materials. Before products such as manufactured aggregate can be developed, producers must understand the differences in moisture content, acid insoluble content, gradation, and mineralogy encountered with the various lithologies mined in the state. These variables will, in turn, determine fines suitability for particular product applications.

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APPENDICES

APPENDIX A: EXAMPLE QUESTIONNAIRE USED FOR THE UF-FDOT BYPRODUCT FINES SURVEY

QUESTIONNAIRE FOR UF-FDOT SURVEY

I. Please provide us with the following:

A. The name and main office of your plant:

____
Name of company

____
Street address

____
City County/State Zip code

____
Telephone Fax

B. Mine name and location, or Florida Department of Transportation mine number:

____

____

C. Contact person for future communications:

Name: ____________________________
Title: ____________________________
Telephone: ____________________________
Fax: ____________________________

D. The name and main office address of your company:

____
Name of company

____
Street address

____
City County/State Zip code

____
Telephone Fax

II. Materials being produced.

A. What type of aggregate product do you process? Please check the appropriate box.

Limestone
Dolomite
Sand and Gravel
Other (please specify) ____________________________

B. What geologic formation(s) is/are being mined?

Ocala Ls	Suwannee Ls	Miami Ls
Tamiami Fm	Ft. Thompson	Key Largo
Anastasia Fm	Avon Park Fm	Citronelle/
Other	Unknown	Cypresshead

____
(please specify)

III. Total annual production of plant in tons:_______________________________

IV. What are your market areas within the state? (i.e., South Florida, Southwest Florida, West Central Florida, Northeast Florida, East Central Florida, Central Florida, etc.)

Areas:____

Areas:____

Out of state:____

Out of country:____

V. What kind of shipping facilities are available to you and what is your use?

A. Truck _______% of total
B. Rail _______% of total
C. Barge _______% of total
D. Ship _______% of total
E. Other _______% of total

VI. Please fill out the following:

	minus-3/8"	minus-200 mesh	other
Annual production			
Main mineral composition			
Quantity marketed annually (tons)			
Quantity of fines stockpiled (tons)			
Moisture content of fines (%) in-situ			
Stored above ground in piles			
Stored below grade in ponds			
Other type(s) of storage:			

VII. Which mesh sizes do you find most difficult to market?

#4 by #20	#20 by #100	#100 by #200	minus-200
Other (please specify):			

VII. Product marketing.

A. What are your **current** markets for the **minus-3/8"** mesh product? Please check all applicable boxes.

Proprietary Please do not disclose

1. Asphalt related uses such as slurry seal aggregate and mineral filler.
2. Agriculture related uses such as aglime, fertilizer filler, soil remineralization and livestock feed.
3. Use as fine aggregate in hot-mixed asphalt, concrete, and manufacturing concrete block and concrete pipe.
4. Environmental applications such as control of sulfur dioxide emissions, pond and watershed liming, acid mine drainage abatement, and landfill layers.
5. Miscellaneous applications such as industrial fillers, paint industry, etc.

6. Others (please specify)____

B. What do you foresee as **potential** markets for the **minus-3/8"** mesh product? Please check all applicable boxes.

Proprietary Please do not disclose

1. Cement treated subbases
2. Ready mixed flowable fills
3. Sandbags
4. Sandfilling applications
5. Solid waste landfills
6. Low cost masonry uses
7. Hazardous waste containment
8. Others (please specify):

IX. Product marketing of fines.

A. . What are your **current** markets for the **minus-#200** mesh product? Please check all applicable boxes.

Proprietary Please do not disclose

1. Asphalt related uses such as slurry seal aggregate and mineral filler.
2. Agriculture related uses such as aglime, fertilizer filler, soil remineralization and livestock feed.
3. Environmental applications such as control of sulfur dioxide emissions, pond and watershed liming, acid mine drainage abatement, and landfill layers.
4. Miscellaneous applications such as industrial fillers, paint industry, etc.

5. Others (please specify)____

B. What do you foresee as **potential** markets for the **minus-#200** mesh product? Please check all applicable boxes.

Proprietary Please do not disclose

1. Cement treated subbases
2. Ready mixed flowable fills
3. Sandbags
4. Sandfilling applications
5. Solid waste landfills
6. Low cost masonry uses
7. Wetland restoration
8. Hazardous waste containment
9. Others (please specify):

X. How do you currently dispose of your minus-#200 material?

Proprietary Please do not disclose

XI. Over the next decade, is the amount of fines stockpiled/produced at your plant likely to:

Increase Decrease Remain unchanged

XII. Please list the factors (e.g. specifications, regulations, lack of awareness on the part of producers, lack of research on potential uses, economics, transportation, etc.) which, in your opinion, are inhibiting the widespread use of fines for various applications.

____
____
____

XIII. Do you have the following details about your minus-3/8" and minus-#200 fines? Please mark the appropriate boxes.

	minus-3/8"	minus-200		minus-3/8"	minus-200
Bulk mineralogy			Gradation		
Elemental analysis			Permeability		
Plasticity index			pH		
Particle shape			Particle strength		
Loose unit weight			Compact unit weight		
Hydrometer analysis			Clay content, %		
Moisture content			Clay mineralogy		

XIV. Could the above details be made available for our data bank?

Yes No Some, but not all

XV. How would you classify the processing at your plant?

Dry Wet Both Other (please specify)

____

XVI. What steps are used in processing the products at this source (crushing, sizing, washing, etc.)? Fill in only those which are necessary to describe your process.

Step1___________
Step2___________
Step3___________
Step4___________

Step 5___________
Step 6___________
Step 7___________
Step 8___________

XVII. What kind of crushing equipment is used in each size reduction step? Please mark one box for each size reduction step.

Step	Proprietary	Jaw	Impact	Gyratory	Cone	Roll	Other	None
1								
2								
3								
4								
5								

XVIII. Which steps are open (no recirculating material) or closed (over-size recirculated) processes?

Proprietary Please do not disclose

Open Step: 1 2 3 4 5

Closed Step: 1.. 2.. 3.. 4.. 5..

XIX. What type of classification is used in sizing steps? Please mark one box for each sizing step.

Proprietary Please do not disclose

Stage	air	screening	hydraulic	cyclone	none	Other (specify)
1						____________
2						____________
3						____________
4						____________
5						____________

XX. Which steps are wet or dry classifications?

Proprietary Please do not disclose

Wet 1 2 3 4 5

Dry 1 2 3 4 5

XXI. Do you have any suggestions or clarifications regarding the contents or format of the proposed database? (Please feel free to attach a letter).

____

____

____

____

2 PART II: EVALUATION AND CHARACTERIZATION OF PRODUCTS

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James L. Eades
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College of Liberal Arts and Sciences
University of Florida

LIST OF FIGURES

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INTRODUCTION

At least 85 and up to 200 potential uses are known to exist for byproduct fines produced by the coarse aggregate industry (Stokowski, 1992; 1993), yet they remain a major waste problem. Current uses include additives to non-specification aggregate, applications as fill or daily cover for landfills, industrial mineral feedstocks, soil amendments, and acid neutralizers for wastes. For minus-3/8" by plus-200 mesh fines, current uses center around asphalt-related applications as slurry seal aggregate and mineral filler (Hudson et al., 1997). More limited applications are observed in agricultural-related uses (aglime, fertilizer filler, soil remineralization, and livestock feed) and as manufactured sand for concrete mixes. The use of minus-200 mesh fines is more limited, and presently directed toward agricultural-related applications, particularly aglime, and as mineral filler in asphalt (Hudson et al., 1997). As indicated previously for byproduct fines produced in Florida, the fine fraction (minus-200 mesh) lacks marketable applications (34.4 % sold annually), whereas the coarse fraction (minus-3/8" by plus-200 mesh) has seen greater success (78 % sold annually). As the FDOT is the focus of this study, this review of current and potential uses centers on construction applications, particularly for minus-200 mesh fines.

Product development must consider both market and plant location factors, as well as the needs of the coarse aggregate industry for a high volume, easily implemented product. Based on the available literature and patents reviewed, most of the work to date has revolved around agricultural limestone products (Able, 1995). The U.S. Bureau of Mines has even used a Geographic Information Systems (GIS) method to generate a spatial analysis on using byproduct fines in agricultural and forest soils (Kramer, 1996). Models of this type use input data which includes transportation infrastructure, locations of fines producing quarries, land-use data, transportation costs, unit value of the fines at the mine site, and the on-site use price to determine the economic feasibility of using fines dependent on distance from the quarries. It is likely that a similar technique could be employed for alternative applications in the construction industry.

This study aims to investigate a variety of uses for byproduct fines, focusing on construction applications attractive to the FDOT and the coarse aggregate industry as high volume and economical means of fines disposal. As byproduct fines, particularly the minus-200 mesh, are a major waste and storage problem for the coarse aggregate industry, development of a viable solution through alternative product application is of great importance.

OBJECTIVES AND SCOPE

As noted in Phase I of this report, the ultimate objective of this research project is to evaluate the nature of byproduct fines production in the state of Florida, with an emphasis on identifying high volume economic uses for these materials which are attractive to coarse aggregate producers in the state. The FDOT is the focus of this project, with the results aimed at enhancing the awareness of FDOT personnel to the geographic distribution, quantities, and properties of coarse aggregate byproducts that may be used as raw material for the production of manufactured aggregates and other secondary applications identified by this study. Phase II of the study is focused on the latter portion of the research objective; identifying high volume economic uses for byproduct fines, with the desire of increasing aggregate industry productivity, and extending the life of an important natural resource. The resulting goals aimed at accomplishing this objective are:

(1) to identify potential economic uses of the byproduct fines characterized in Phase I of the study
(2) to evaluate the economics of processing technologies that would be employed to produce high volume products produced with byproduct fines, emphasizing manufactured aggregates
(3) to evaluate test specimens produced with manufactured aggregates produced from byproduct fines of varying lithology

The first two goals of this phase of research were fulfilled through a review of the literature available on the use of byproduct fines, with an emphasis on identifying high volume uses feasible for the Florida aggregate industry, and through evaluation of economic data, particularly focused on technologies not widely employed in the state. The literature review focused on the published and unpublished literature on agglomeration and/or compaction of fines, as well as relevant computer programs that relate to the production of manufactured aggregate materials. However, other high volume uses, which might be of interest to the FDOT (backfill, flowable fill, and direct additives to concrete), were investigated as well. Subsequently, batch-scale pilot plant trials for producing granular limestone and dolomitic limestone/dolomite were performed on fines from three sites identified by the FDOT and representing different lithologies from around the state. The objectives of these batch-scale trials were:

- to produce a 0.42-9.5 mm granule from byproduct fines which have a mean particle size of 0.074 mm
- to produce a granule with low abrasion and high crush resistance in product handling
- to produce a granule which is insoluble when exposed to water
- to identify and test three possible binding agents for producing a granular limestone road aggregate

- produce a sufficient test quantity of each granular product to perform the necessary field trials
- to develop sufficient engineering data to scale-up the batch granulation process to the next development stage

Aspects of this portion of the study were carried out by Applied Chemical Technology, Inc., an expert in the area of process technology and economics.

The last goal of this phase of the research was accomplished by evaluating the use of granular aggregate produced from byproduct fines as a fine aggregate alternative in Portland cement concrete (PCC) mixes. Test cubes were prepared and evaluated for compressive strength after curing at room temperature for 3, 7, 14, and 28 days. The resulting data were compared to standard specimens prepared with Ottawa sand and FDOT construction grade sand, in order to ascertain the validity of this application for granular aggregate prepared with two different binders (sodium silicate and Portland cement)

EVALUATION OF POTENTIAL ECONOMIC USES

The use of quarry byproducts in the construction market has seen recent attention, as shown by two papers presented at the Third Annual Center of Aggregate Research Symposium. At this conference, Fowler (1995) of the American Limestone Company of Knoxville, Tennessee, discussed the company's marketing of fines as stone sand for use in ready mix concrete, precast concrete, concrete blocks, and masonry sand, while Wood and Mareck (1995) of Vulcan Materials Company, Birmingham, Alabama, discussed the recovery and use of limestone fines as mineral fillers in hot mix asphalt, stone sand, controlled low strength concrete (more commonly known as flowable fill), base, sub-base, and soil stabilization. In the ICAR study of byproduct fines, Hudson et al (1997) identified the following high volume potential uses for fines:

- Cement-treated subbase
- Ready-mix flowable fill
- Sandbags
- Sandfilling applications
- Solid waste landfills
- Low cost masonry uses
- Subsurface sewage disposal systems
- Wetland restoration

Given the interests of the FDOT, this study focuses on the potential use of fines in engineered backfills (flowable fill and backfill for mechanically stabilized earth), direct additives to concrete mixes (fillers and fine aggregate), and agglomeration of the minus-200 mesh fraction for use as fine aggregate in both flowable fill and concrete mixes.

Engineered Backfill

The use of byproduct limestone fines in ready mixed flowable fill (RFF), and as backfill material for mechanically stabilized earth (MSE) walls has shown promise in recent years. Investigations by the ICAR have identified flowable fills as a potential use for large volumes of byproduct fines. Estimates suggest that between 4 and 5 million tons of fines could be utilized annually in the Atlanta, Georgia area alone (Hudson et al., 1996). However, it should be noted that these fines are most likely granitic and in the coarser screenings size range. Either coarse fines (minus-3/8" by plus-200 mesh) or agglomerated fines produced from the minus-200 mesh fraction generated by the aggregate industry in Florida could be employed in much the same way in flowable fill mixes.

Flowable fills can potentially be used for a wide range of applications, including low strength backfills, slurry wall moisture barriers, vertical moisture barriers to maintain consistent moisture in swelling clays, foundation cushions, and pavement base and subbase layers. Important areas of research identified by the ICAR in the area of flowable fills include: (1) investigation of mechanical systems to effectively excavate and mix pond screenings with other components, (2) development of mix proportions for pond screenings, and (3) identification of high-potential uses (Hudson et al., 1996). Since flowable fills are a very low strength material (50 to 100 psi range), agglomerated fines (minus-200 mesh) would be ideal as a substitute for either natural or manufactured fine aggregate sands, likely increasing product workability.

The use of byproduct fines in MSE walls shows good strength properties, but questions remain concerning permeability, a problem that could be overcome with the addition of small amounts of larger grain sizes (Parker, 1996). The use of fines can result in MSE wall designs with a greater factor of safety using the same amount of reinforcement used with natural soil and/or sand, or can reduce the amount of reinforcement necessary for a given design (Parker, 1996). Agglomerated limestone and/or dolomitic fines might be applicable to this use as well, particularly due to both particle shape and material permeability. The rounded shape of pelletized material should not only increase the flowability of RFF, but also increase permeability as desired for MSE construction.

Direct Addition to Concrete Mixes

Both coarse (minus-3/8" by plus-200 mesh) and fine (minus-200 mesh) categories of byproduct fines have seen applications in asphaltic and Portland cement concrete (PCC) mixes either as fillers (minus-200 mesh) or fine aggregate (minus-3/8" by plus-200 mesh). Limestone fines have been used for over 20 years as a filler in asphalt and Portland cement, yet questions remain regarding their effect on concrete mix properties, including decreased strength with age and susceptibility to both rutting and carbonation (Ingram and Daugherty, 1991; Al-Suhaibani et al., 1992; Çelik and Marar, 1996; Uchikawa et al., 1996). In Brazil, research on the use of higher contents of crushed powder fill, in this case from basaltic rocks, in concrete mixes resulted in increased strength and reduced permeability (Oliveira et al., 1995). However, it appears that results vary, and that the effect of limestone fines as a filler in concrete mixes may be source specific, requiring detailed testing for individual quarries prior to use.

Only recently has the use of the coarse fraction (minus-3/8" by plus-200 mesh) as fine aggregate in concrete mixes been investigated. As this fraction is usually rich in filler sized (minus-200 mesh) particles, questions have been raised as to its suitability as a fine aggregate in concrete mixes. Historically, excess fines have been considered deleterious (particularly clays), and have been blamed for unstable asphalt mixes, and the inhibition of bonding between aggregates and the cementitious mortar (Dukatz, 1995). However, a study by Orr and Slattery (1993) found that crushed limestone fines containing up to at least 15 percent filler sized particles were suitable for use as fine aggregate in PCC mixes. Results indicated several advantages, including early increases in strength believed to result from the presence of crushed limestone particles in the fine aggregate size range causing accelerated cement hydration and the formation of additional gel products. However, reductions in strength after one year suggest that gel structure may alter with age.

Fines Agglomeration

Agglomeration methods can be used to convert limestone or dolomitic limestone/dolomite fines (particularly minus-200 mesh) into manufactured aggregate for use in concrete (asphaltic and Portland cement). However, a review of the available literature and patents show that the majority of the existing limestone agglomeration work has been geared toward production of agricultural products (Albert and Langford, 1993), and to ease fines handling for industrial purposes (Paul, 1993; Paul et al., 1993a; b). No literature was found that discussed the production of aggregate for concrete from limestone or dolomitic limestone/dolomite fines. Based on the needs of the FDOT and aggregate producers statewide, agglomeration was selected as the application of focus for continued evaluation.

Four agglomeration processes have been identified as potential processing methods for producing limestone and dolomitic limestone/dolomite aggregate from minus-200 mesh fines. The basic operating principles of these processes are covered in the following discussion, and are divided into two primary categories: Wet and Dry Processing. The wet processing methods are drum granulation and pan granulation. The dry processing methods are roll-press flaking and briquetting.

Wet Processing

Wet granulation processes, as the name implies, incorporate the use of moisture (water or a solvent) or a binder in solution to effect agglomeration. The basic principle of wet granulation is the application of a wetting agent (typically water or a water-based binder solution) onto a tumbling bed of fines. At equilibrium, the system is essentially at a point where the rate of input of raw material fines and solution to the tumbling bed is sufficient to yield a constant rate of the product granules from the tumbling bed. The granules of the desired size are separated by screening. The undersize fines and crushed oversize fines are returned to the tumbling bed as recycle material. Typically, the amount of moisture required to facilitate granulation is large enough to require the material to be dried before it can be stored.

The two methods of wet process granulation are: 1) drum granulation and, 2) pan granulation. Both of these granulation processes incorporate much of the same equipment and employ the same basic principles. The primary difference is the type of granulator that is used.

Drum Granulation

Literature and patents dealing with agglomeration of limestone include U.S. patent #4,954,134 (Harrison and Tittle, 1990). This patent details the use of a rotary drum agglomeration process to produce an agricultural limestone product. The process uses a mixture of ammonium lignosulfonate (48 % solids) and H_2O mixed 50/50 by weight as the binder. The final product is minus-6 by plus-16 mesh with 3 % to 6 % binder with a typical crush strength of 7 pounds for a Tyler #8 mesh granule. A second drum agglomeration process is detailed in U.S. patent #3,692,511 (Wilson and Southworth, 1967). This process was also developed for agricultural limestone. The process uses ammonium nitrate as the binder at a concentration of 2 to 18 %. No screen size or crush strength data was published in the patent.

The rotating cylinders of the drum granulator may incorporate various internal designs to facilitate mixing, lifting, or transport. In practically all cases, the drum is the point where the combination of dry and wet ingredients takes place. In some processing arrangements, a separate mixer prior to the drum granulator may be used to effect contact of the binder solution and the dry powder. The system includes surge bins for raw material powder and recycle, the granulator drum, binder addition equipment, drying, cooling, and sizing to separate the product sized material from oversize and undersize (Fig. 2-1).

The drum granulation process begins with the introduction of the limestone or dolomitic limestone/dolomite fines into the raw material surge bin. Recycled material (undersize and crushed oversize) and fines from the dust collection system are transported to the recycle surge bin to be introduced with the fresh raw material powder and binder solution to the granulator. The fresh feed (fines) and recycle are metered to the granulation drum by weigh feeders, typically belt or screw feeders. The binder solution is sprayed onto the rolling bed of fines and recycle in the

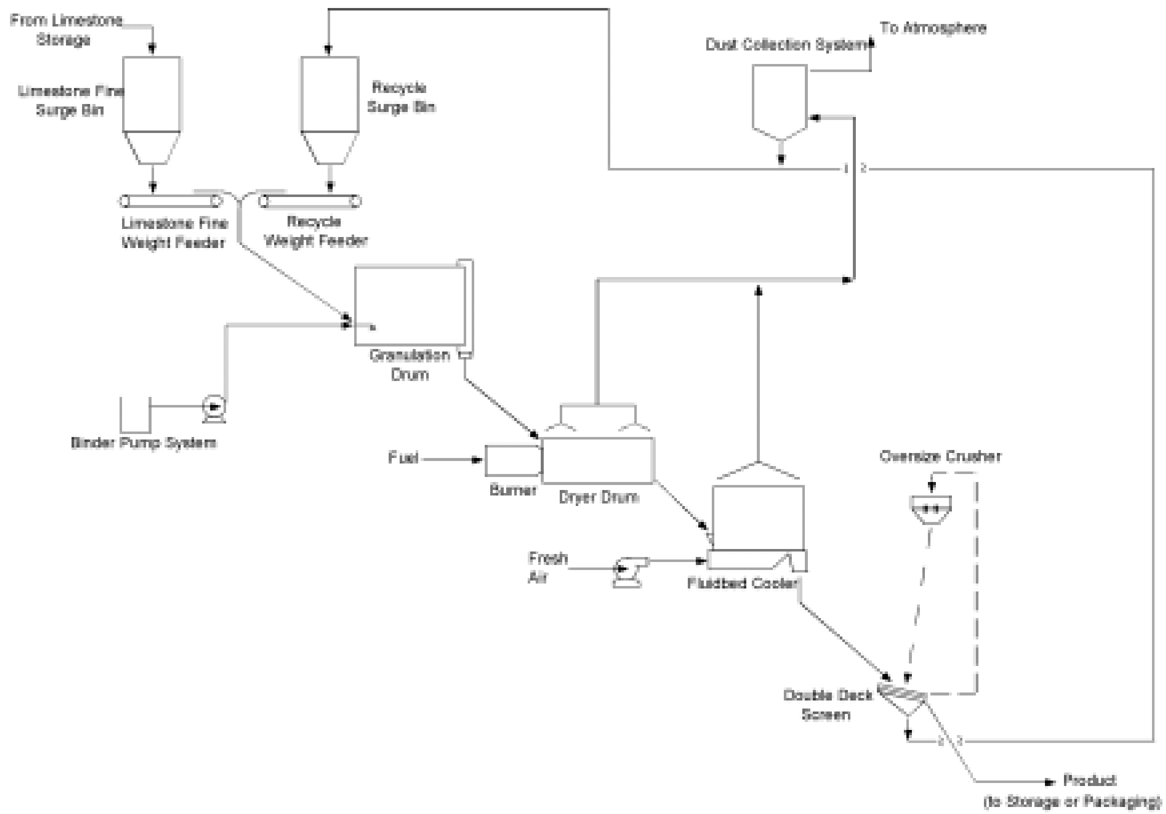


Figure 2-1. Byproduct fines drum granulation process flow sheet.

granulation drum. The water and binder, along with the rolling action of the drum, forms the limestone fines and recycle into granules by agglomeration and accretion. The concentration and quantity of binder is determined by the characteristics of each type of limestone.

Granular material formed in the drum typically contains from 4 % to 10 % moisture and displays a wet crush strength (tested by unconfined compression) of 1 to 2 pounds for the minus-7 by plus-8 mesh size fraction granules (~2.6 mm size). The "wet" granules are discharged from the granulation drum and transported to the drying step. Drying is normally accomplished in a rotary drum dryer which uses the rolling action of the granules during the drying step to further compact the material, hence imparting greater crush strength. The granules are dried to < 1.0% water. The drying step increases the crush strength to 3 to 10 pounds for the minus-7 by plus-8 mesh material and reduces caking should the material require storage prior to shipment.

The relatively hot material from the dryer is discharged into a cooler which reduces the temperature of the granules from 200ºF - 240ºF to < 120ºF for screening. Some processes use a rotary cooler for this service. Recent plants incorporate a fluid bed cooler due to the inherent increased efficiency of fluid bed units. The fluid bed cooler also serves to remove any surface dust from the granules, improving subsequent storage and handling properties of the materials. The dust is recovered in a bag filter or other dust collection device. The material is discharged from the fluid-bed cooler to a double-deck screen where the granular material is separated by size as desired. The oversize material goes to a crusher with the resulting partially crushed material conveyed back to the screen decks for re-screening. The undersize and fines from the dust collecting and crusher system are transported to the recycle feeder. The product is removed from the screen and is ready for storage or packaging.

The drum granulation flow diagram (Fig. 2-1) consists of the following four main systems:

- Raw Material Feed and Recycle Loop:
    - Limestone fines surge bin
    - Limestone fines weigh feeder
    - Recycle surge bin
    - Recycle weigh feeder
- Granulation Loop:
    - Binder solution pumping system
    - Drum granulator
- Drying/Cooling Loop:
    - Burner system, safety train & blower
    - Drying drum
    - Fluid-bed cooler & blower
    - Dust collection system
- Sizing Loop:
    - Double-deck screen
    - Oversize crusher

Pan Granulation

Two articles by Paul et al. (1993a;b) discuss using pilot-scale pan granulation equipment to produce limestone granules of approximately 0.5" diameter. Several binders were successfully used including: bentonite, soda ash, scrubber sludge, and lime. The pellets were recrushed to the minus-200 mesh fraction. The article states that more than 100 million short/tons of limestone are crushed to minus-200 mesh annually by consumers who require this fine size. The stone is typically crushed on-site due to the transportation problems of ultra-fine material. White Pelletizing Company operates two limestone 20 ton per hour granulation plants located in Castlewood and Paradise, Virginia. These plants use pan agglomeration technology to produce an agricultural limestone product with the following screen analysis before agglomeration: minus-8 mesh = 100%; minus-60 mesh = 80%; minus-100 mesh = 75%; minus-200 mesh = 55%. These plants use ammonium lignosulfonate as a binder to produce granules with a crush strength of 2.6 to 7.3 pounds for a Tyler #8 mesh granule.

The pan granulation process is similar to that previously described for drum granulation. The major difference in these two processes is the substitution of a granulation pan (disk pelletizer) for the granulation drum (Fig. 2-2). In this process, fines are fed into a surge bin.

Recycled material (undersize and crushed oversize) and dust from the dust collection system are transported to the recycle feeder. The fresh feed and recycle are fed to the pan granulator by the limestone fines and recycle weigh feeders in the same manner as to the granulation drum in the

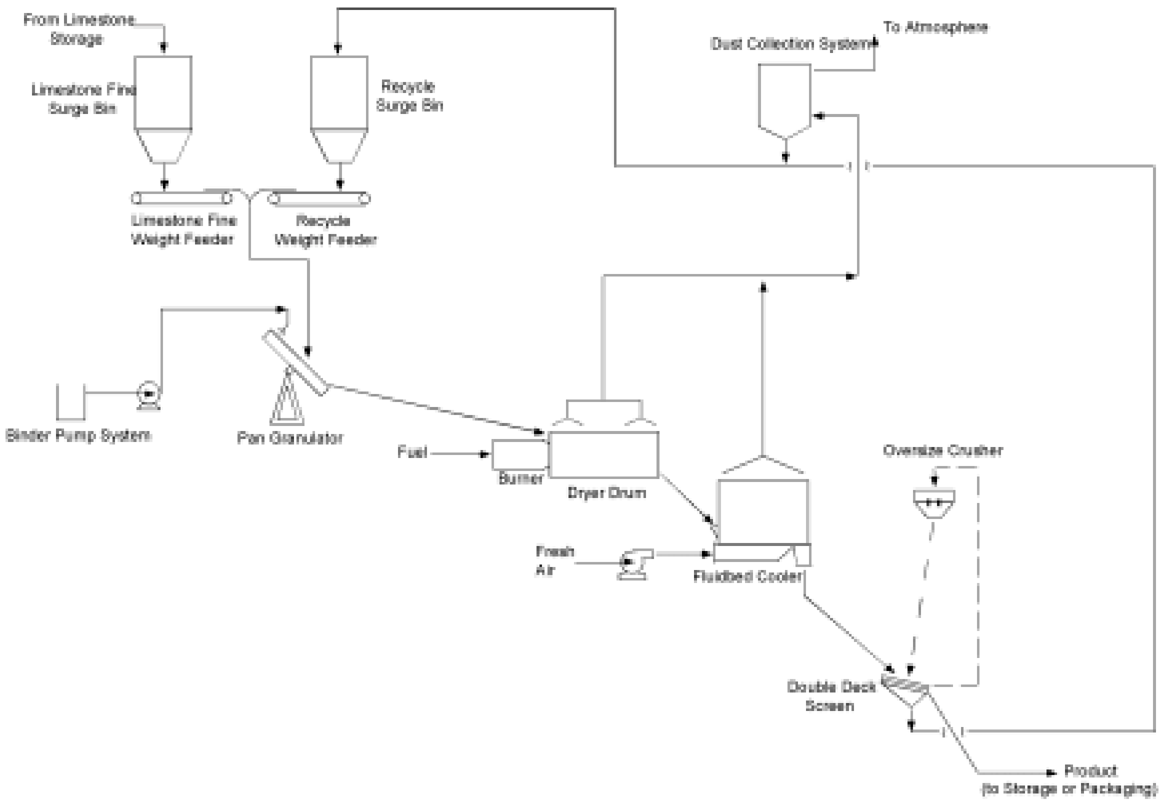


Figure 2-2. Byproduct fines pan granulation process flow sheet.

system previously described. The pan granulator operates as a positive displacement machine converting the raw material into wet pellets. During each revolution of the pan, the granules and fines are lifted to the maximum height and then fall/roll freely to the lower section of the rotating pan. In the bed of variously sized granules, large pellets circulate on the top of the smaller pellets over a relatively small area in the lower outside portion of the pan, close to the rim at the discharge point. Smaller pellets lifted under the larger pellets circulate over a wider area in the pan and become exposed to the water/binder spray and incoming fines and recycle being fed into the pan. Thus, the smaller pellets grow and are again lifted under the larger pellets. When the granules have "grown" sufficiently (size determined by pan angle, rotational speed, and fines and binder characteristics), the larger pellets discharge by centrifugal force over the lower edge of the pan. As with the drum granulation process, the granulated material is discharged from the granulation pan and transported to a drying drum. The granules are normally dried to < 1 % water. The dried granules have a crush strength in the range of 2.5 to 7 pounds for minus-7 by plus-8 material, marginally lower than that obtained using a drum granulation system.

Next, the dried material is discharged into a fluid-bed cooler which cools the material from 200ºF - 240ºF to < 120ºF for screening. The material is discharged from the fluid-bed cooler to the double-deck screen where the granular material is sized as desired. The oversize material flows to the crusher with the resulting partially crushed material flowing back to the screen decks for re-screening. The undersize and fines from the dust collecting and crusher system are transported to the recycle feeder. The product is removed from the screen and is ready for storage or packaging.

The pan granulation flow diagram (Fig. 2-2) consists of the following four main systems:

- Raw Material Feed and Recycle Loop:
    - Limestone fines surge bin
    - Limestone fines weigh feeder
    - Recycle weigh feeder
- Granulation Loop:
    - Binder solution pumping system
    - Pan granulator
- Drying/Cooling Loop:
    - Burner system, safety train & blower
    - Drying drum
    - Fluid-bed cooler & blower
    - Dust collection system
- Sizing Loop:
    - Double-deck screen
    - Oversize crusher

The following are potential modifications to both of these basic processes for granulation:

- Where storage space is available and a closely sized product is not desired, the wet pellets can be allowed to air dry in storage piles, in some cases, eliminating the drying, cooling, crushing, and screening steps. Increased product caking would probably be observed using this curing method.
- A product conditioner may be used to prevent caking. This can be accomplished by adding a conditioning drum with a conditioner feeding system. Several types of systems may be utilized including addition of parting agents such as clays or talc or spraying of an oil coating.
- Where a wide product size distribution is acceptable, the drum/pan systems can be run "open" sized with only the finest material being returned as recycle. This effectively increases production rates and would require smaller equipment with the reduced recycle loads.

Dry Processing

Literature from Ludman Machine Corporation (1997) outlines pilot plant test work using roll compaction and various binders to produce granular limestone and quick lime from fines. The basic principle of dry compaction is the application of pressure on a fine powder as it is forced between two counter-rotating rolls. The nip area between the rolls is the area where the compaction takes place. The effect of the applied pressure is to remove the air from the bulk powder and so densify the material.

The two methods of dry process compaction discussed are 1) roll-press flaking and, 2) roll-press briquetting. These processes incorporate much of the same type of feeding and sizing equipment used in the drum and pan granulation processes. Drying and cooling are not normally required. Both methods discussed use a similar roll-press and peripheral equipment. The roll-press machine consists of a set of two powered, counter-rotating rolls (the design of the rolls differ between the two processes and is discussed in detail later). One roll is typically mounted on fixed bearings; the other is floating in a slide arranged so that it can be forced toward the fixed roll by hydraulically actuated pistons acting on the bearing blocks. The material to be compacted is continuously fed into the nip of the rolls, usually by a screw feeder. Very high pressure is exerted on the material as it is drawn between the rolls thereby densifying the material by air removal, utilization of atomic attraction forces, and sometimes plastic deformation of the fines thereby yielding a hard sheet or briquettes (Paul et al., 1993a).

Roll-Press Flaking

Figure 2-3 is the flow diagram for the roll-press flaking process. In this process, the limestone and dolomitic limestone/dolomite fines and recycled materials are fed into surge bins in much the same manner as that described for the wet granulation processes. The fresh feed and recycle are fed to the roll-press compactor by weigh feeders. The proportion of fresh feed to recycle is kept constant; typically, a change in ratios of “fresh feed to recycle” will require adjustments to the roll-press. Fresh feed and recycle are mixed in a low intensity mixer and transported to the roll-press. In some processes, water or a binder solution is added to the material in the mixer. Normally, binders are not required for roll-press granulation.

The basic design of a roll-press flaker is two counter-rotating rolls. The rolls may be made of smooth cast iron or, in some cases, the rolls may be corrugated. The rolls form a sheet or flake of material which is processed further in size reducing equipment consisting of a flake breaker and comminution-type granulator to obtain a range of particle sizes from which the desired product size is separated by screening. Either gravity or screw feed is used to transport the material into the nip between the rollers. Due to the bridging tendency of limestone fines, a screw feeder is preferred.

The compacted flakes exiting from the roller press are pre-crushed by a flake breaker to provide proper feed size for the granulator. These pieces are transported by a belt to the granulator. The granulator consists of corrugated rolls with sharp edged corrugations designed to break the flakes in such a way to form a maximum percentage of the desired product size while keeping dust generation to a minimum. Oversize from the screen may be returned to the comminutor for additional processing with fines being returned to the recycle system.

The roll-press flaker flow diagram (Fig. 2-3) consists of the following three main systems:

- Feed and Recycle Loop:
    - Limestone fines surge bin
    - Limestone fines weigh feeder

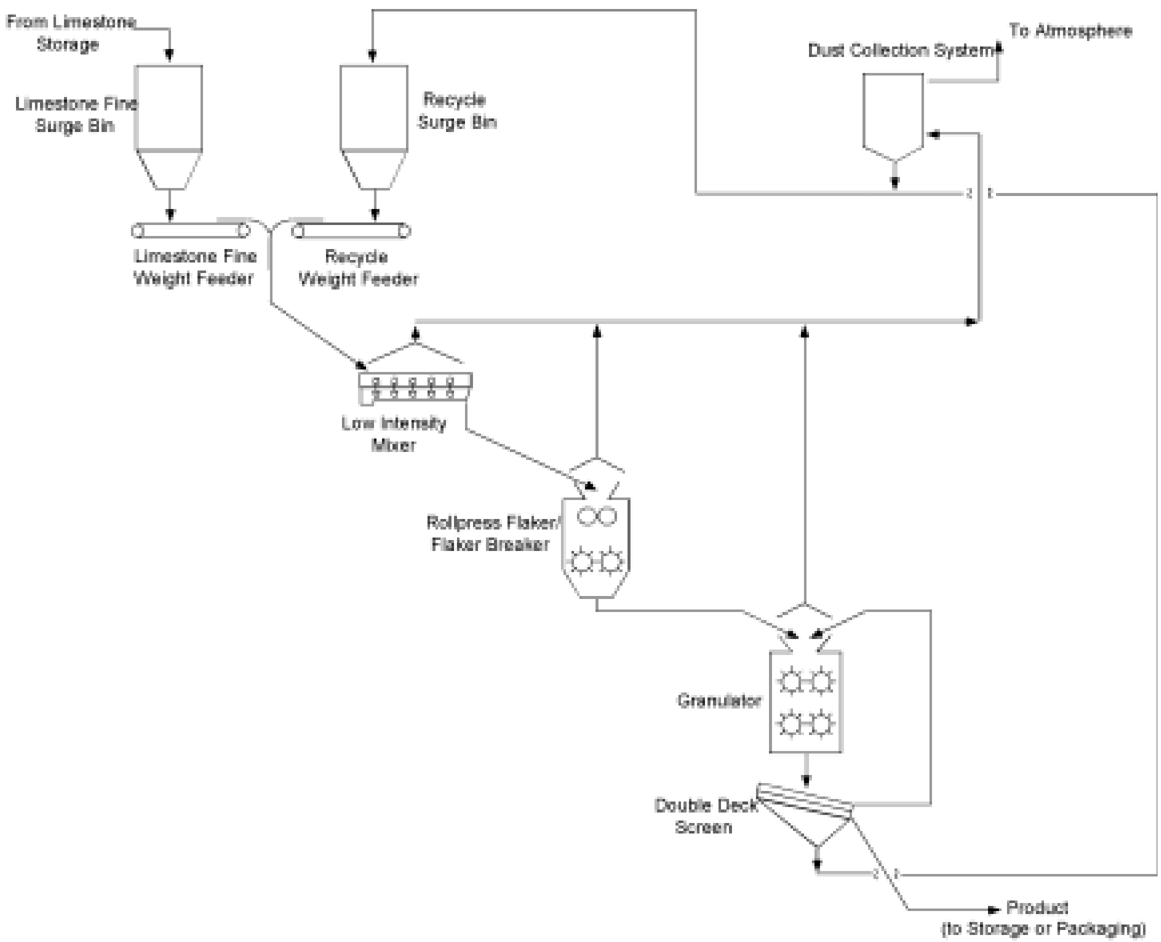


Figure 2-3. Byproduct fines roll-press flaking process flow sheet.

    - Recycle surge bin
    - Recycle weigh feeder
    - Low intensity mixer
- Compaction Loop:
    - Roll-press compactor
    - Flake breaker
- Granulation/Sizing Loop:
    - Granulator/Comminutor
    - Double-deck screen

Roll-Press Briquetting

The briquetting process is similar to the roll-press flaking process. The major difference is in the types of rolls used in the compaction machine. In this process, fines are fed into a surge bin. Recycle material (fines) and dust from the dust collection system are transported to a recycle bin (Fig. 2-4). As with the roll-press flaker, the fresh feed and recycle are fed to the process using weigh feeders. The proportion of fresh feed to recycle is kept constant. Fresh feed and recycle are mixed in a low intensity mixer and transported to the roll-press. The basic design of a roll-press briquetter is two counter-rotating rolls with half-briquette cavities formed into the circumference of each roll. The rolls are indexed so the cavities on the opposing rolls match to form whole cavities as both halves pass through the centerline.

A variable speed screw feeder forces material into the nip of the rolls (This method permits the feed rate to be adjusted to match the volumetric requirements of the roll cavity), filling the cavities just before they close. The pressure applied to the material by the closing cavities as they pass through the centerline compresses the material into a solid mass. As the cavity passes beyond the centerline, the two halves move apart allowing the formed briquettes to fall out of the briquette mold and discharge from the machine.

In briquetting, newly formed briquettes contain residual stresses upon emerging from the roll-press. As they “relax”, they gain hardness and strength. For that reason, the briquetting system includes a transfer conveyor of the appropriate length and speed to allow the briquettes to “relax” before they are screened for storage or packaging.

The roll-press briquetter flow diagram (Fig. 2-4) consists of the following three main systems:

- Feed and Recycle Loop:
  - Limestone fines surge bin
  - Limestone fines weigh feeder
  - Recycle surge bin
  - Recycle weigh feeder
  - Low intensity mixer
- Compaction Loop:
  - Roll-press briquetter
- Transfer/Sizing Loop:
  - Transfer “relaxing conveyor”
  - Single deck screen

The following are potential modifications to both these basic processes:

- Binding agents (typically lignosulfonates) may be introduced at the low intensity mixer prior to compaction.
- Due to the nature of the processes, the briquettes and granules may have thin edges or corners that break-off in handling to form fines. To overcome this problem, the material may be passed through a “tumbler” so that fragile edges are broken off and

can be removed by screening and recycled to the process along with undersize from the screen.

- A product conditioner may be used to prevent caking. This can be accomplished in a conditioning drum.

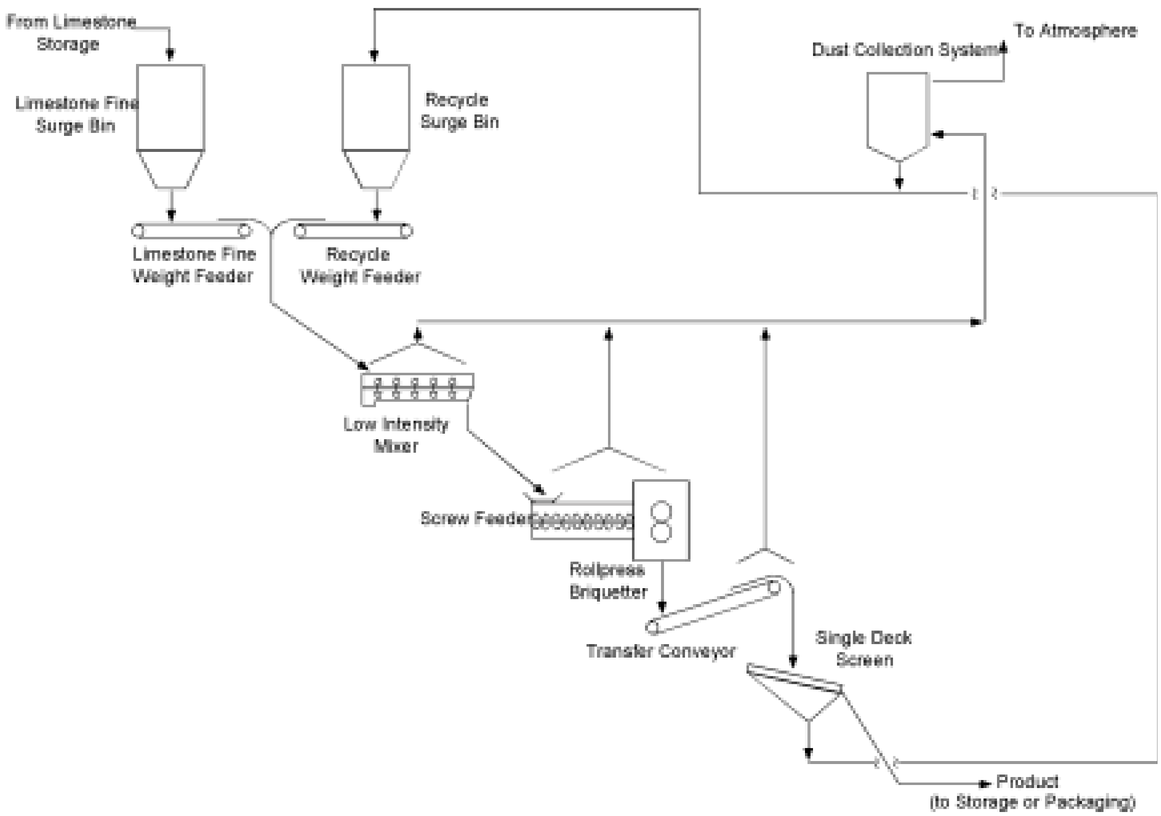


Figure 2-4. Byproduct fines roll-press briquetting process flow sheet.

Binders

With the assistance of Applied Chemical Technology, Inc., (ACT) and drawing from their more than 16 years experience in agglomeration process development, effective binders were selected to meet the specific application requirements identified by this study. ACT has used various lignosulfonates, molasses (and other sugar base extracts), starches, sodium silicate, clays, organic compounds, as well as acids, bases, and other reactive chemicals to agglomerate fine particles into granules. They also are experienced in the use of sintering and compacting as a means of binding fine particulates into granules.

One of the most critical points in the development of any agglomeration process is the proper selection of a binder. In order to form an agglomerate of sufficient strength to be produced from particulate matter, there must be adequate binding forces between the particles within the agglomerate. Agglomerate bonding may be divided into five major groups, with more than one mechanism possibly applying during a given size-enlargement operation.

Solid bridges can form between particles by the sintering of ores, the crystallization of dissolved substances during drying as in the granulation of fertilizers, and the hardening of bonding agents such as glue and resins. *Mobile liquid* binding produces cohesion through interfacial forces and capillary suction. Three states can be distinguished in an assembly of particles held together by a mobile liquid. Small amounts of liquid are held as discrete lens-shaped rings at the points of contact of the particles; this is the pendular state. As the liquid content increases, the rings coalesce and there is a continuous network of liquid interspersed with air; this is the funicular state. When all the pore spaces in the agglomerate are completely filled, the capillary state has been reached. When a mobile liquid bridge fails, it constricts and divides without fully exploiting the adhesion and cohesive forces in the bridge. By contrast, *immobile liquid bridges* formed from highly viscous materials such as asphalt or pitch fail by tearing apart the weakest bond. Then adhesion and/or cohesion forces are fully exploited, and binding ability is much larger. *Intermolecular and electrostatic forces* bond very fine particles without the presence of material bridges. Such bonding is responsible for the tendency of particles less than about 1 µm diameter to form agglomerates spontaneously under agitation. With larger particles, however, these short-range forces are insufficient to counterbalance the weight of the particle, and adhesion does not occur. *Mechanical interlocking* of particles may occur during the agitation or compression of, for example, fibrous particles, but it is probably only a minor contributor to agglomerate strength in most cases.

The most applicable binding mechanism(s) for the four processes discussed previously are solid bridges and mechanical interlocking. The solid bridge type binder would include sodium silicate, lignosulfonates, or the use of acids or bases. Mechanical interlocking would be the binding mechanism of the compaction process. Small amounts of binder are sometimes used in conjunction with compaction to further strengthen the particles.

Potential binders were investigated in the laboratory, allowing the evaluation of numerous binders without incurring substantial costs. Subsequently, the information gained during the lab-scale tests was used to develop a pilot test program to further develop and evaluate promising binder and agglomeration techniques involving the use of sodium silicate, portland cement, and calcium sulfate hemihydrate ($CaSO_4 \cdot \frac{1}{2}H_2O$).

Process Economics

The preliminary comparison of the economics of the wet agglomeration versus that of the dry compaction agglomeration for the production of limestone and dolomitic limestone/dolomite granules is based on the production of agricultural products. The estimate is based on a "battery limits" plant, which consists of the process equipment erected and ready to operate.

The installed cost of the battery limits plant comprises all direct and indirect costs. The direct costs represent the material and labor cost necessary for the installed process equipment with all auxiliary equipment and buildings that are needed for complete process operation. The indirect costs include the costs for design, engineering, supervision, construction overhead and expense, contractor's fee, and contingency. Because the battery limits process plant cost is the foundation of the total fixed capital, the technique for estimating process unit investments is discussed in detail in the following sections.

The fixed investment estimates were developed by estimating the delivered equipment costs and then estimating the other direct and indirect costs on the basis of the equipment costs. The equipment cost estimates were developed by ACT from the design and fabrication costs from projects with similar equipment and layouts. The cost of the compactor was provided by Cubman Machine Corporation. The cost estimates for equipment installation and other direct costs (labor and materials) were calculated as percentages of the equipment costs. The indirect cost items are estimated as percentages of the direct cost estimates. This fixed investment estimating technique is commonly used for preliminary estimates, particularly for comparative estimates.

Premises and Assumptions for Investment and Conversion Cost Estimates

The following premises and assumptions were made in the process of estimating investment and conversion costs for the economic comparison of wet agglomeration verses dry compaction agglomeration techniques:

- Cost estimates are for a U.S. Gulf Coast location and use a mid-1997 cost basis. Costs are shown in Tables 2-1 through 2-5.
- Plant design capacity for all designs is 11 tph, which corresponds to 78,800 tpy at 90 % capacity utilization for 300 days per year.
- The investment cost estimates (Table 2-2) are for battery limits granulation equipment installed at an existing plant requiring minor additions of auxiliary and support facilities. Allowances are not included for land, raw materials or product storage, handling, and bagging facilities.
- Allowances are not included for working capital or for interest and escalation during construction.
- The conversion cost estimates do not include the following: bagging cost, interest on working capital, and raw material and product handling costs, which are assumed to be the same for the compaction and wet granulation processes.
- Utilities costs are based on: electricity = $0.047/kWh; Fuel = $14/million kcal.
- Annual maintenance costs (material and labor) are based on 5 % of fixed investment for the wet granulation processes and 6 % of fixed investment for compaction processes.
- Annual insurance and taxes are based on 2 % of fixed investment.
- Operating labor and supervision costs are based on $240,000 and $400,000 per year for the compaction process and granulation process, respectively.
- Administration and general expenses are based on 100 % of operating labor and supervision.
- Fixed capital recovery costs are based on a capital recovery of 0.1314. This is the capital recovery factor corresponding to a 10 % interest rate and a 15-year period. The capital recovery covers depreciation and interest or return on investment.

Investment and Operating Costs

Data developed by ACT on investment costs for limestone granulation and compaction units (Table 2-1) show that the cost for a "wet" granulation process is slightly higher than that for a dry compaction unit. Most of the cost difference is due to greater costs for instrumentation, piping and ductwork, auxiliary facilities and buildings. The process equipment cost is essentially the same for both units (Tables 2-2 and 2-3) because the cost of the compactor and associated

Table 2-1. Direct and indirect fixed investment cost as percentages of process equipment cost.

% OF DELIVERED PROCESS EQUIPMENT COST	Compaction Process	Granulation Process
A) DIRECT COST		
1 - Delivered process equipment cost	100	100
2 - Process equipment installation	45	45
3 - Instrumentation (installed)	10	15
4 - Electrical equipment (installed)	15	10
5 - Piping/ducts (installed)	15	20
6 - Auxiliary facilities	10	15
7 - Building and structures (indoor plant types)	40	45
TOTAL DIRECT COST	235	250
% TOTAL OF DIRECT COST		
B) INDIRECT COST		
1 - Engineering and project management	10	10
2 - Construction overhead and expenses	8	8
3 - Preoperational and start-up	5	5
4 - Contractor's fee and contingency	18	18
TOTAL INDIRECT COST	41	41

Table 2-2. Fixed investment estimates for the dry compaction process.

MAJOR PREMISES:	1 - Cost basis: 1997 U.S.$ 2 - Location: U.S. Gulf Coast 3 - Plant capacity: 11 tph	
		FIXED INVESTMENT
A) Delivered process equipment cost		$1,400,000
B) Total direct cost (235% of item A)		$3,290,000
C) Total indirect cost (41% of item B)		$1,348,900
TOTAL FIXED INVESTMENT (B+C), rounded		$4,638,900

Table 2-3. Fixed investment estimates for the wet granulation process.

MAJOR PREMISES:	1 - Cost basis: 1997 U.S.$ 2 - Location: U.S. Gulf Coast 3 - Plant capacity: 11 tph	
		FIXED INVESTMENT
A) Delivered process equipment cost		$1,400,000
B) Total direct cost (250% of item A)		$3,500,000
C) Total indirect cost (41% of item B)		$1,435,000
TOTAL FIXED INVESTMENT (B+C), rounded		$4,935,000

Table 2-4. Conversion cost estimated for the dry compaction process.

MAJOR PREMISES:	1 - Cost basis: 1997 U.S.$ 2 - Location: U.S. Gulf Coast 3 - Plant capacity: 11 tph 4 - Operating rate: 78,800 tpy 5 - Bulk product	
Fixed Investment (FI), $ million: 4.64		
CONVERSION COST	**REQUIREMENT**	**$/mt**
A) VARIABLE COSTS		
1 - Electricity ($0.047/kWh)	30 Kwh	1.41
2 - Miscellaneous supplies		0.60
SUBTOTAL		2.01
	$/YEAR	**$/mt**
B) FIXED COSTS		
1 - Operating labor and supervision	240,000	
2 - Administrative and general expense	240,000	
3 - Maintenance (6% of FI)	278,000	
4 - Insurance and tax (2% of FI)	93,000	
5 - Fixed capital recovery (13.14% of FI)	610,000	
SUBTOTAL	1,461,000	18.54
TOTAL CONVERSION COST (rounded)		20.55

Table 2-5. Conversion cost estimated for the wet granulation process.

MAJOR PREMISES:	1 - Cost basis: 1997 U.S.$ 2 - Location: U.S. Gulf Coast 3 - Plant capacity: 11 tph 4 - Operating rate: 78,800 tpy 5 - Product moisture content (dryer inlet): 3-10% 6 - Bulk product	
Fixed Investment (FI), $ million: 4.94		
CONVERSION COST	**REQUIREMENT**	**$/mt**
A) VARIABLE COSTS		
1 - Electricity ($0.047/kWh)	20 Kwh	0.94
2 - Fuel (14/million kcal)		1.23
3 - Miscellaneous supplies		1.18
SUBTOTAL		3.35
	$/YEAR	**$/mt**
B) FIXED COSTS		
1 - Operating labor and supervision	400,000	
2 - Administrative and general expense	400,000	
3 - Maintenance (6% of FI)	296,000	
4 - Insurance and tax (2% of FI)	99,000	
5 - Fixed capital recovery (13.14% of FI)	648,000	
SUBTOTAL	1,843,000	23.39
TOTAL CONVERSION COST (rounded)		26.74

equipment for the compaction plant is about equal to the cost of the granulator and drying system in the granulation plant. Most of the peripheral equipment is about the same for each type of unit.

Tables 2-4 and 2-5 show the calculated conversion costs for both compaction and granulation. This analysis shows that the conversion costs including utilities, labor, maintenance, taxes, insurance, and capital recovery are about 30 % higher for wet granulation than for compaction. Given a yearly production of 78,800 tons, a savings of $488,000 per year would be realized in operating costs with the compaction plant. This would be an ongoing savings in addition to the estimated $296,000 savings in the investment cost for the compaction plant compared to wet granulation.

The investment costs for both wet and dry processing are quite similar. Other factors not considered in the investment estimate include:

- The effect of scale: Drum and pan granulation plants and operating costs are typically favorably affected by increasing plant size. The effects of scale are typically not as substantial in the dry/compaction granulation processing.
- Continuous versus interruptive use: Drum and pan granulation systems require a steady state of operation for optimum efficiency. They can sometimes require considerable time and material to start and stop. One advantage of the dry processing is easy start/stop operation allowing the plant to be operated more efficiently on an interruptive basis.
- Operator skill level: The dry/compaction processing typically requires lower skill levels to operate properly, due to the simplicity of the equipment. Typically fewer operators are required to operate a compaction plant versus a wet/agglomeration plant.

Summary

The four processing methods discussed for the agglomeration of minus-200 mesh (< 75 μm) limestone and dolomitic limestone/dolomite fines form the basis for most fine powder agglomeration processes found in industry today. These granulation methods may be useful in providing granules for use as aggregate in concrete. All four of the processes have/or are currently being used to produce agglomerated limestone for use as an agricultural liming agent. The key to successfully adapting any one of these processes to produce a granule suitable for aggregate use will be a the identification of a binder to provide a fines granule with adequate crush strength for use as concrete aggregate.

Due to variations in the physical and chemical make-up of the byproduct fines being evaluated, as well as the condition in which these wastes are stored (dry storage, pond fines, or uncovered stockpiles), variations in processing parameters from site to site are likely. The variation in physical and chemical make-up effects both wet and dry processing as shown by granulation pilot plant development tests conducted by Paul et al. (1993a). Ludman Machine Corporation (1997) sites variations in compactability of limestone fines based on calcium content, stating that high calcium limestone fines appear to compact more readily than dolomitic types. Parameters such as feed rates, temperatures, recycle ratios, percent and type of binder, and percent moisture required to agglomerate or compact may vary from site to site. Also, operational particulars such as rotational speed and retention times for drum granulators and roll separation force for compactors also can vary and cannot be determined without pilot plant studies.

PRODUCTION AND EVALUATION OF GRANULAR AGGREGATE FROM BYPRODUCT FINES

As a means of evaluating the production of granular aggregate from byproduct carbonate rock (limestone and dolomitic limestone/dolomite) fines, tests were performed on three of the raw materials described in Phase I (quarries D, H, and I; see Table 1-1) using three different binders. A "wet" processing method (similar to drum granulation) using a pug mill mixer/granulator was selected for fines agglomeration due to equipment availability/simplicity, positive economics, and ease of testing. One sample (D) also was processed using a "dry" compaction/extrusion technique (California Pellet Mill) for comparison to the granulation results. The materials tested are byproducts from several of the carbonate rock lithologies presently mined in the state for coarse aggregate materials. The three binders tested were sodium silicate, Portland cement, and calcium sulfate hemihydrate. A total of nine (9) 200 lb samples of aggregate granules were produced for field trials. The final objective was to produce granules that were hard, insoluble, and abrasion resistant, while beginning to identify the appropriate process parameters and equipment required for a large-scale granulation plant. This objective was to be achieved via the goals outlined at the beginning of this phase of the report.

Equipment Description for Batch Pilot Plant Tests

Pug Mill Mixer/Granulator:

- Purpose: To mix and granulate limestone fines
- Mixing Trough: 36" length, 13" width, 12" depth
- Two (2) - 2" diameter stainless steel shafts with 15 counter rotating blades per shaft
- Paddle Angle: 45º from vertical
- Discharge Dam: manual, adjustable
- Drive: 5 Hp, variable speed inverter
- Pug Mill Charge: 25-73 lbs
- Pug Mill Rotation: 104 rpm (powder mixing stage - forward direction)
  576 rpm (granulation stage - forward direction)
- Binder Addition: 3-7 lbs of 58% sodium silicate solution
  8-12 lbs of Portland Cement
  10 lbs of calcium sulfate hemihydrate
- Water Addition: 0-6000 ml
- Mixing/Granulation Time: 15-20 min

Vibrating Fluid-bed:

- Purpose: Dry granulated product to less than 2.0% moisture

- Fluidizing Plate Area: 2 ft^2
- Air Distribution Plate: 6.33% open area
- Combustion Chamber: Gas fired
- Maximum Bed Depth: 4"
- Operating Temperature: 276ºF inlet air
- Drying Time: 15-20 min

Drying Drum:

- Purpose: Dry granulated product and produce a round granule
- Drum Dimensions: 3' diameter, 7.5" width, 10" depth
- Drum Internals: (8) ¼" anti-slip rods (evenly spaced)
- Drum Drive: 2 Hp Baldor, variable speed
- Drying Temperature: 250ºF inlet air
- Drying Time: 15-20 min
- Combustion Chamber: gas fired
- Drum Speed: 12 rpm

California Pellet Mill:

- Purpose: Pelletize limestone fines
- Pelletizing Die: 15.5" diameter, 1.5" width
  3/16" openings for pellet extrusion
  4" diameter stationary rollers
- Drive: 30 Hp
- Pelletizing Die Speed: 132 rpm
- Pellet Size: cutting knives preset to produce 1" pellets

Process Descriptions

Sodium Silicate

PQ Corporation sodium silicate was the first binder used for the granulation of byproduct fines in the pugmill mixer/granulator. The sodium silicate solution was 58 percent. Additional water was added during the granulation of the byproduct fines to aid in granule formation and to achieve the proper consistency of powder to binder in the pugmill. Beaker studies were performed to determine the least amount of binder necessary for granule formation. Results for the tests using sodium silicate and the other binders are given in Table 2-6. Sodium concentrations were determined for the purpose of assessing the potential for alkali-silica reactivity (ASR) of the resulting products when used in concrete mixes.

<u>Quarry D</u>

The dolomitic limestone/dolomite fines from quarry D contained 17.5 percent moisture in the starting material. Seventy-three pounds of byproduct fines were added to the pugmill, while the paddles were rotating in the forward direction at a shaft speed of 104 rpm. Seven pounds of

58 percent sodium silicate solution along with 2000 ml of water was evenly poured over the fines. The mixture was granulated in the pugmill mixer/granulator until a desired particle size

Table 2-6. Granulated aggregate product evaluation.

Quarry Code	Agglomeration Method	% Binder	Crush Strength (lbs)	Drying Temp. (ºF)	% Na	% Moisture
Sodium Silicate						
D	Pug mill granulation	5.3	7.55	276	0.55	1.1
H	Pug mill granulation	6.5	6.98	276	0.44	1.9
I	Pug mill granulation	6.5	13.40	276	0.50	0.8
	Mean	6.1	9.3		0.5	1.3
	Median	6.5	7.6		0.5	1.1
	STD	0.7	3.6		0.1	0.6
Portland Cement						
D	Pug mill granulation	16.7	21.15	ambient	n/a	8.0
H	Pug mill granulation	11.7	2.36	ambient	n/a	0.6
I	Pug mill granulation	16.1	20.50	ambient	n/a	5.8
	Mean	14.8	14.7			4.8
	Median	16.1	20.5			5.8
	STD	2.7	10.7			3.8
Calcium Sulfate Hemihydrate						
H	Pug mill granulation	16.7	10.00	250	n/a	6.1
I	Pug mill granulation	16.7	10.00	250	n/a	5.0
	Mean	16.7	10.0			5.6
	Median	16.7	10.0			5.6
	STD	0.0	0.0			0.8
D	CA Pellet mill extrusion	5.0	6.00	ambient	n/a	n/a

distribution was determined by visual observation. The pugmill shaft speed during granulation was 576 rpm. The mixing time was approximately 20 minutes. When the granules reached a desired product size, the dam was lowered and rotation of the paddles reversed to discharge the material from the pugmill. The byproduct granules from the pugmill were hand fed into a vibratory fluid-bed. The granules were fluidized and dried at an inlet air temperature of 276ºF. Multiple passes through the fluid-bed were required for complete drying. The final product contained 1.1 percent moisture. The dry dolomitic limestone/dolomite granules were then screened to remove the undersize (0.71 mm) and the oversize (9.5 mm). The oversize was passed through a hammermill and re-screened. The undersize was combined with additional byproduct fines and recycled back to the pugmill for further granulation. The product contained approximately 5.3 percent sodium silicate binder. The average granule crush strength for the 2.3-3.6 mm particles was 7.55 lbs.

Quarry H

Quarry H byproduct limestone fines contained 15.2 percent moisture in the raw feed, therefore no additional water was added during the pugmill granulation test. Using the same process described for quarry D materials, fifty pounds of limestone fines were added to the pugmill along with six pounds of 58 percent sodium silicate solution. The mixture was granulated in the pugmill for approximately 15 minutes. When the granules reached a desired product size, they were discharged from the pugmill and hand fed into a vibratory fluid-bed. The final product contained a final moisture content of 1.9 percent and approximately 6.5 percent sodium silicate binder. The average granule crush strength for 2.3-3.6 mm particles was 6.98 lbs.

Quarry I

Quarry I byproduct limestone fines contained 0.2 percent moisture in the starting material. Using the same process described for quarry D materials, twenty-five pounds of byproduct fines were added to the pugmill along with three pounds of 58 percent sodium silicate solution and 2500 ml of water. The mixture was granulated in the pugmill for approximately 20 minutes. When the granules reached a desired product size, they were discharged from the pugmill and hand fed into a vibratory fluid-bed. The final product contained 0.8 percent moisture and approximately 6.5 percent binder. The average granule crush strength for 2.3-3.6 mm particles was 13.4 lbs.

Portland Cement

Portland cement was the second binder utilized for granulating byproduct fines. Water was added to the limestone fines and Portland cement mixture to begin granulation in the pugmill mixer/granulator. The ratio of Portland cement to byproduct fines was dependent upon the characteristics of the raw material. Beaker studies were performed to determine the least amount of binder necessary for granule formation. The moisture level and chemical composition of the limestone or dolomitic limestone/dolomite dictated the amount of Portland cement and water required to produce a desired product.

Quarry D

Sixty pounds of dolomitic limestone/dolomite fines along with 12 pounds of Portland cement were charged into the pugmill mixer/granulator. Operating conditions for the pugmill were the same as those used with testing of the sodium silicate binder. Approximately 4500 ml of water was evenly distributed over the powder mixture. The byproduct fines and Portland cement were granulated in the pugmill until a desired product distribution was determined by visual observation. The approximate time required to granulate the dolomite fines was 20 minutes. When the granules reached a desired product size, the dam was lowered and the paddle rotation reversed to discharge the material from the pugmill. Initial attempts to dry the limestone granules in the fluid-bed resulted in product degradation. This method of drying caused severe dust formation inside the dryer. Therefore, the limestone granules were spread onto sheets of plastic and allowed to cure under ambient conditions for 24 hours. The final product contained 8.0 percent moisture. As with the sodium silicate tests, the dry material was then screened to remove the undersize (0.71 mm) and the oversize (9.5 mm). The oversize material was passed through a hammermill and re-screened. The undersize material was combined with additional byproduct fines and Portland cement and recycled back to the pugmill for additional granulation. The product contained approximately 16.7 percent Portland cement. The average granule crush strength of 2.3-3.6 mm particles was 21.15 lbs.

Quarry H

Using the same process described for quarry D materials, sixty pounds of quarry H limestone fines along with 8 pounds of Portland cement and approximately 3000 ml of water were charged into the pugmill. The byproduct fines and Portland cement were granulated in the pugmill for approximately 20 minutes. When the granules reached a desired product size, they were discharged from the pugmill and allowed to cure under ambient conditions for 24 hours. The final product contained 0.6 percent moisture and approximately 11.7 percent Portland cement. The average granule crush strength of 2.3-3.6 mm particles was 2.36 lbs.

Quarry I

Using the same process described for quarry D materials, sixty pounds of quarry I limestone fines along with 11.5 pounds of Portland cement and approximately 5500 ml of water were added to the pugmill. The byproduct fines and Portland cement mixture were granulated in the pugmill for approximately 20 minutes. When the granules reached a desired product size, they were discharged from the pugmill and allowed to cure under ambient conditions for 24 hours. The final product contained 5.8 percent moisture and approximately 16.1 percent Portland cement. The average granule crush strength of 2.3-3.6 mm particles was 20.5 lbs.

Calcium Sulfate Hemihydrate

The final binder tested for granulation of limestone fines was calcium sulfate hemihydrate, also known as Plaster of Paris. Beaker studies were performed to determine the least amount of binder necessary for granule formation. These studies indicated a 5:1 ratio of limestone fines to calcium sulfate hemihydrate was required to granulate the materials. Water was added to the limestone fines and calcium sulfate hemihydrate mixture to begin the granulation step. Drying of the limestone granules was performed in a rotary drum dryer. The rolling action produced by the dryer improved the roundness of the particles. Quarry D fines were not tested with this binder.

Quarry H

Fifty pounds of quarry H limestone fines along with 10 pounds of calcium sulfate hemihydrate were charged into the pugmill mixer/granulator. Operating conditions for the pugmill were the same as those used with testing of the sodium silicate binder, except for a paddle speed of 380 rpm during granulation. As the paddle speed was increased to 380 rpm, 4000 ml of water was evenly distributed over the material. The byproduct fines and calcium sulfate hemihydrate were granulated in the pugmill until a desired particle size distribution was determined by visual observation. The approximate time required to granulate the quarry H limestone fines was 15 minutes. When the granules reached a desired product size, the pugmill dam was lowered to discharge the material. The granulated Aglime was hand charged to a rotary drum dryer. The granules were dried at 250ºF for 20 minutes using an air header, a blower, and a propane gas burner. The drum speed during the drying process was 12 rpm. Once the granule surface was dry, the material was removed from the drum dryer to prevent the evaporation of the water of hydration required to form calcium sulfate dihydrate crystals (gypsum). The interlocking of the calcium sulfate dihydrate crystals with the fines results in the formation of a hard granule. The byproduct granules were spread onto sheets of plastic and allowed to cure under ambient conditions for 24 hours. The final product contained 6.1 percent moisture. The quarry H fines product contained approximately 16.7 percent calcium sulfate. The average granule crush strength of 2.3-3.6 mm particles was 10.0 lbs.

Quarry I

Using the same process described for quarry H materials, fifty pounds of quarry I limestone fines along with 10 pounds of calcium sulfate hemihydrate and 6000 ml of water were charged into the pugmill. The byproduct fines and calcium sulfate hemihydrate were granulated in the pugmill for approximately 15 minutes. When the granules reached a desired product size, they were discharged from the pugmill and hand charged to the rotary drum dryer. After drying, the granules were allowed to cure under ambient conditions for 24 hours. The final product contained 5.0 percent moisture and approximately 16.7 percent calcium sulfate. The average granule crush strength of 2.3-3.6 mm particles was 10.0 lbs.

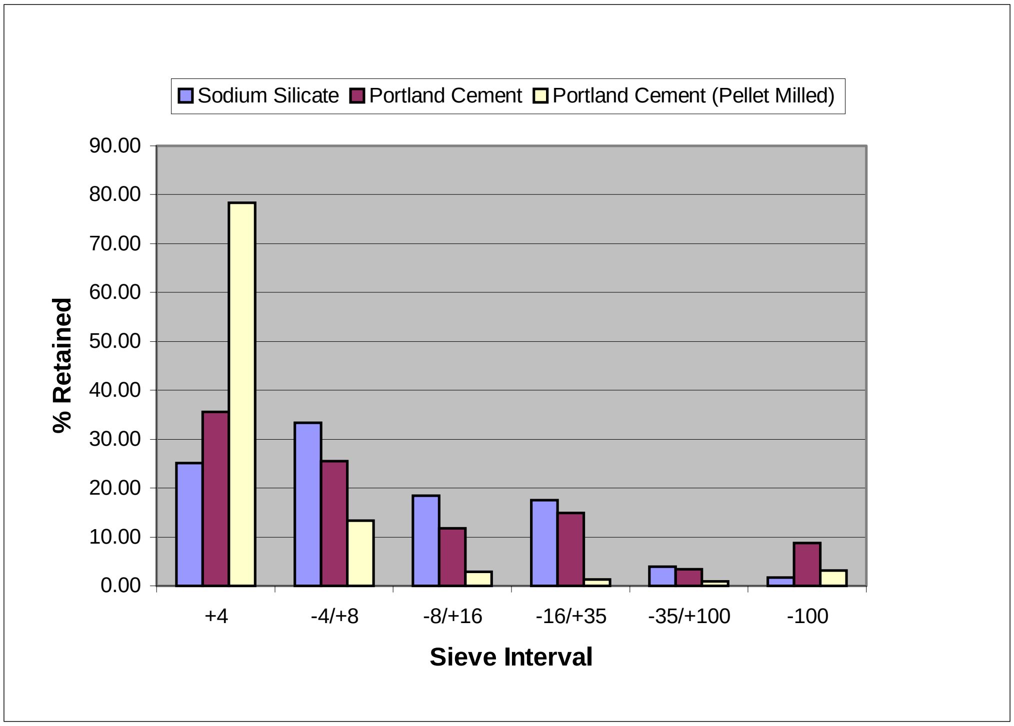
California Pellet Mill: Dolomite with Portland Cement Binder

Quarry D dolomitic limestone/dolomite fines also were tested in a California Pellet Mill. Fifty-seven pounds of dolomite fines and 3 pounds of Portland cement were combined in a 5-gallon bucket with 136 ml of water. A high-speed agitator was utilized to mix all of the raw materials. The material was fed into the California pellet mill. The mixture was extruded through 3/16" openings in the pellet mill die. The pellets were cut to a length of 1" and collected in a container located at the discharge chute of the pellet mill. The pellets contained approximately 5.0 percent Portland cement. The average granule crush strength of the pellets was 6.0 lbs. Tests on quarry H and I limestone fines were not attempted due to the processing problems encountered while testing the quarry D fines.

Discussion of Results

Plots showing the comparative particle-size distribution of the granulated products for each fines source tested are included for evaluation (Figs. 2-5 through 2-7), along with plots comparing the particle-size distribution of products made with sodium silicate (Fig. 2-8) and Portland cement (Fig. 2-9) binders. Review of these plots reveals that the granulated products produced with the calcium sulfate hemihydrate binder and the quarry D product produced with the California Pellet Mill all tended to produce far too much coarse (plus-4 mesh) material, inconsistent with desired product parameters. Sodium silicate and Portland cement binders performed better, producing a product with a more suitable particle-size distribution. A comparison of the sodium silicate and Portland cement products for all three sources (Figs. 2-8 and 2-9) indicates that the quarry D dolomitic limestone/dolomite fines consistently generated a coarser mean particle-size product for both binders in comparison to quarry H and I fines, which possess limestone lithologies.

The binding characteristics of the 58 percent sodium silicate solution produced a mean granule crush strength of 9.3 lbs for 2.3-3.6 mm particles, for all three materials tested. The binder content had a mean value of 6.1 percent, and ranged from 6.5 percent in quarry H and I limestone granules to 5.3percent in the quarry D dolomitic limestone/dolomite granules. The relationship that this might have with the observed differences in particle-size distribution is



uncertain. The process for producing granular limestone and dolomitic limestone/dolomite utilizing sodium silicate as a binder was operator friendly. Recycle addition to the pugmill reduced the processing time necessary for granulating the fines. Drying of the granules was achieved in a vibratory fluid-bed.

Figure 2-5. Particle-size distributions for quarry D granulation products.

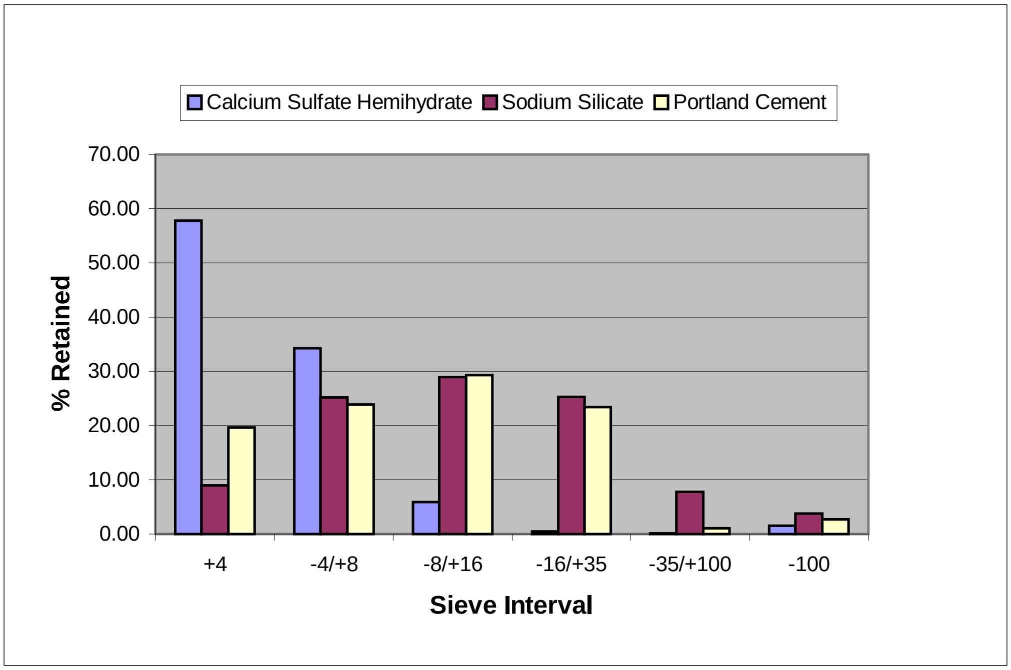
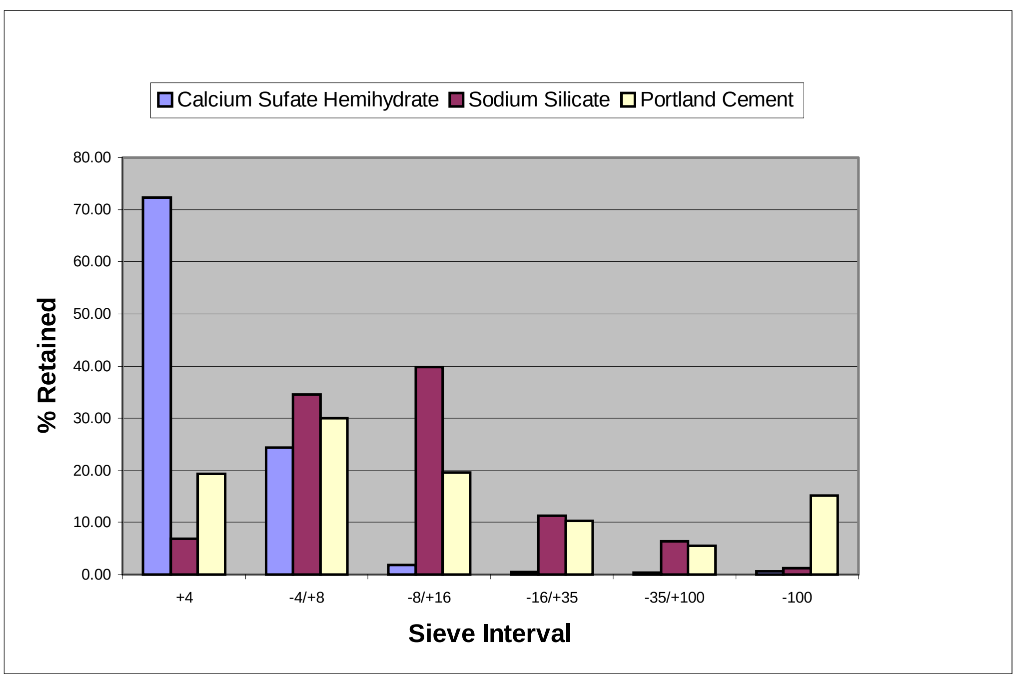


Figure 2-6. Particle-size distributions for quarry H granulation products.

Figure 2-7. Particle-size distributions for quarry I granulation products.



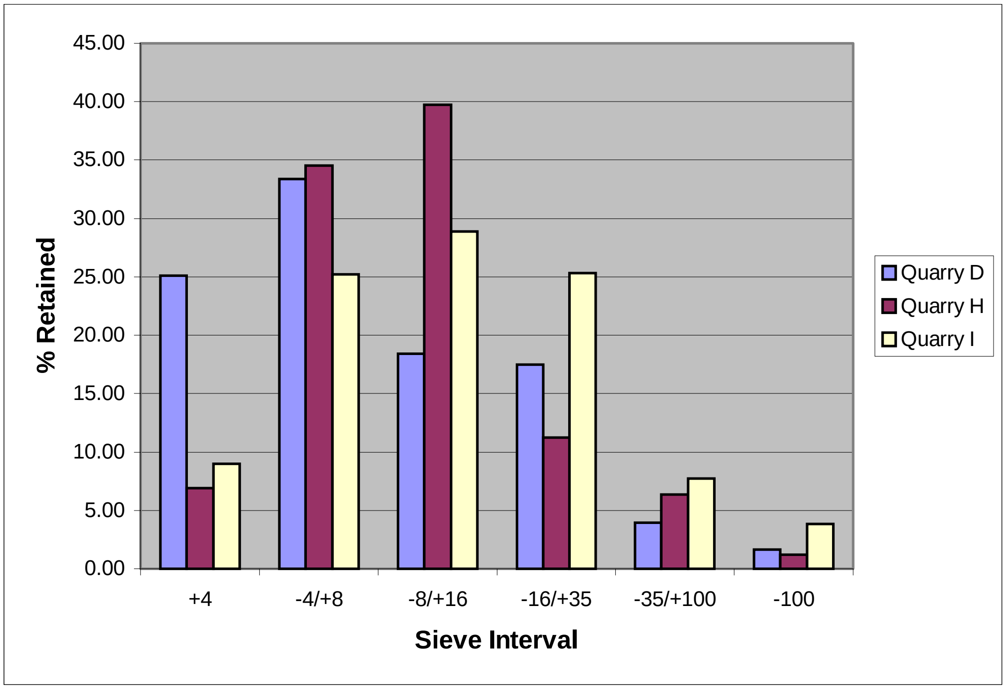


Figure 2-8. Particle-size distributions for products made with a sodium silicate binder.

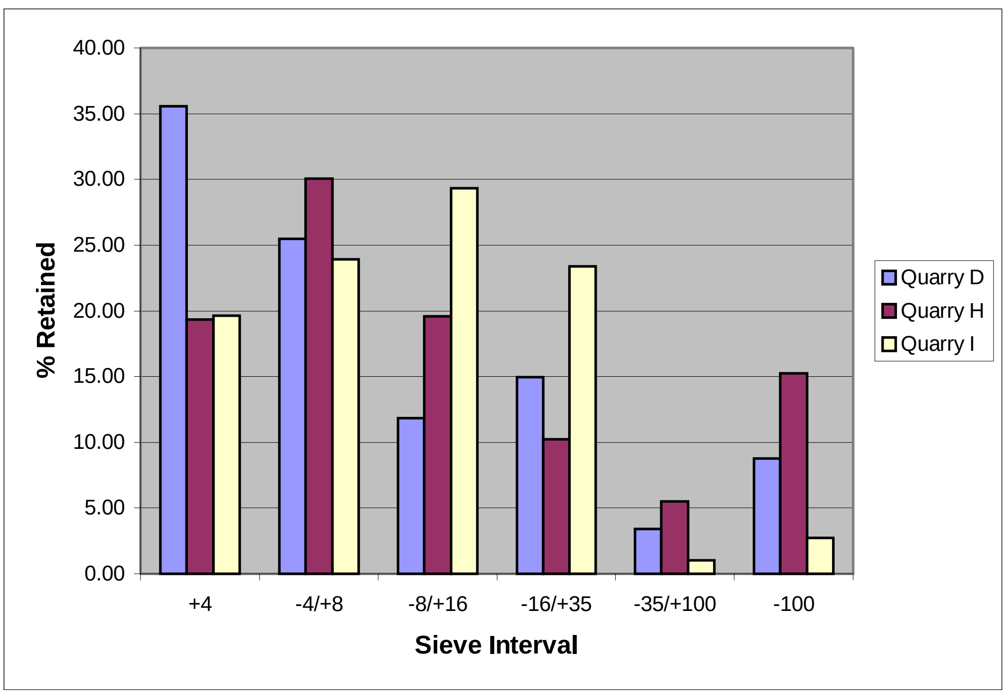


Figure 2-9. Particle-size distributions for products made with a Portland cement binder.

Granules produced in the pug mill with Portland cement produced a mean crush strength of 14.7 lbs for 2.3-3.6 mm particles, for all three materials. However, the mean crush strength for quarry H material was only 2.3 lbs. The cause for the low granule crush strength for this source was not investigated, and may have, in fact, been due to anomalous test results. The other two fines sources each possessed mean crush strengths > 20 lbs. Good granule formation was achieved using a mean binder content of 16.4 percent for quarries D and I, with quarry H (2.3 lbs crush strength) using only 11.7 percent binder during granule production. This difference in binder content may be the underlying cause of the low granule crush strength achieved for quarry H material, if the results are not anomalous. Initial attempts to dry the granules in the vibratory fluid-bed resulted in excessive dust formation. Therefore, granules produced in the pugmill were cured on sheets of plastic under ambient conditions for approximately 24 hours. The time required to dry the limestone granules could be affected by changes in temperature and humidity, as well as fines characteristics (e.g. lithology, clay content, etc.).

The granules made with calcium sulfate hemihydrate produced a mean crush strength of 10.0 lbs for the two materials tested (quarries H and I). The granules had 16.7 percent calcium sulfate hemihydrate for both materials. Beaker studies indicated that a lower percentage of calcium sulfate hemihydrate produced fragile granules. Higher percentages of calcium sulfate hemihydrate in the product improved granule crush strength in the beaker studies. A drum dryer, which improved product roundness, was utilized to dry the granules produced in the pugmill. There was no quarry D material left to perform a granulation test using calcium sulfate hemihydrate as a binder.

Quarry D byproduct fines were the only material processed in the California Pellet Mill. The dolomite pellets produced in the pellet mill contained 5 percent Portland cement as a binder. The crush strength of these pellets was 6 lbs. It was evident that the California Pellet Mill was not a feasible alternative for granulation of fines, due to several processing problems encountered during the test.

EVALUATION OF PORTLAND CEMENT CONCRETE CONTAINING GRANULAR AGGREGATE AS FINE AGGREGATE

Florida ranks fourth nationally in the use of recycled concrete as a coarse aggregate construction material. Other materials commonly investigated for use in the U.S. as either coarse or fine aggregate alternatives include industrial waste products and low grade natural materials produced as waste after mineral extraction, such as china clay waste, slate waste, and pulverized fuel ash (Butler and Harrisson, 1998). Given the geological character of materials mined in Florida by the coarse aggregate industry, and the waste and storage problems generated by byproduct fines, these materials, once granulated, would seem to be a suitable alternative aggregate resource. The potential for use of these materials could significantly extend the life of aggregate resources and the aggregate industry in Florida. However, granule production and subsequent application should take into account the size, shape, and moisture content of the final aggregate product, as all of these factors greatly influence the performance of materials in concrete mixes and the performance of the concrete product itself. Furthermore, given the use of sodium silicate as a binder in the granulation tests, the potential for alkali-silica reactivity (ASR) also must be taken into account with granular aggregate design.

In order to evaluate the viability of using granular aggregate produced from byproduct fines as a fine aggregate alternative in Portland cement concrete (PCC) mixes, 2"×2" concrete cubes (ASTM C109) prepared with granular aggregate produced from the three raw materials discussed previously (quarries D, H, and I), were tested for compressive strength and evaluated microscopically. Only the products prepared with sodium silicate and Portland cement as the binding agents were used in this portion of the study. The principal objective of this phase of testing was to evaluate the strength characteristics and physical make-up of the resulting concrete test mixes. Ottawa sand (ASTM C778) and FDOT construction grade sand for concrete and asphalt from the Florida Rock Industries, Inc. Grandin Sand Mine in Grandin, Florida, were used as comparative standards.

Test Specimen Analysis

The 2"×2" cubes used for compressive strength testing were prepared according to the procedures outlined in ASTM C109. Minus-4 mesh by plus-40 mesh sieve fractions were separated via sieving and collected for each granulated fines material. Samples were subsequently prepared with variable water to cement (W/C) ratios (values recommended by technicians with Florida Crushed Stone Company, Brooksville, FL) according to Table 2-7, dependent on voids volume. After preparation, the cubes were placed in a moist room for 24 hours in accordance with ASTM C511, and then cured for 3, 7, 14, or 28 days at room temperature. Cubes were prepared and tested in triplicate using a Tinius Olsen Testing Machine at the FDOT State Materials Office facility in Gainesville.

Table 2-7. Compressive strength data measured for Portland cement concrete test cubes containing granulated byproduct fines as an alternative aggregate.

Replicate	Time (days)	Sand Standards		Quarries (Sodium Silicate Binder)			Quarries (Portland Cement Binder)			Mean	STD
		Ottawa	FDOT	D	H	I	D	H	I		
W/C		0.485	0.459	0.832	0.709	0.770	0.747	0.692	0.681		
1	3	3413	3600	575	913	980	1738	2475	2500	2024	1156
	7	3538	5838	1213	1200	1025	3075	3375	3175	2805	1628
	14	4200	7288	1313	1498	1513	2763	4088	2913	3197	2003
	28	4763		1525	1313		2788	4050	3800	3040	1408
2	3	3450	3688	600	1038	890	1550	3263	2063	2068	1244
	7	3488	5888	988	888	1163	2450	3450	3313	2703	1706
	14	3713	7038	1138		1450		3950	2738	3338	1957
	28	4400	7063	1230	838	1550	3238	4100	2813	3154	2052
3	3	3475	4450	585	900	995	1913	2563	1975	2107	1343
	7	3338	5688	988	800	1075	2775	4088	3075	2728	1715
	14	4125	7700	1263	1163	1275	3113	4013	3188	3230	2184
	28	4188	7250	1540	763	1450	2925	4450	2888	3182	2100
Mean	3	3446	3913	587	950	955	1733	2767	2179		
	7	3454	5804	1063	963	1088	2767	3638	3188		
	14	4013	7342	1238	1330	1413	2938	4017	2946		
	28	4450	7156	1432	971	1500	2983	4200	3167		
STD	3	31	468	13	76	57	181	432	281		
	7	104	104	130	210	70	313	392	119		
	14	263	335	90	237	123	247	69	227		
	28	291	133	175	298	71	231	218	550		

Compressive Strength Results

Table 2-7 outlines the results from the compressive strength tests performed on the granulated byproduct fines and standard samples. Anomalous results were excluded. The table and a graph comparing the compressive strength results for each of the materials granulated with sodium silicate compares poorly with the standard test specimens (Fig. 2-10). The cubes made with FDOT sand exhibit the greatest mean compressive strength values (28-day = 7156 psi) followed by Ottawa sand (28-day = 4450 psi). The cubes prepared with the granulated fines produced with the sodium silicate binder all show very poor results, particularly quarry H. In fact, the standards are as much as 4 to 7 times stronger than the granular fines samples. The poor performance by the materials prepared with sodium silicate may have been the result of the binder exhibiting a degree of water solubility that was unexpected based on material design criteria. As a result, granule strength immediately deteriorated during preparation of the 2"×2" cubes. ASR also must be considered as a possibility as well.

The test specimens prepared with the granulated fines made with the Portland cement binder performed far better than the sodium silicate samples (Table 2-7; Fig. 2-11). However, the FDOT sand and Ottawa sand still exhibited the greatest strength values. In decreasing strength order, quarries H, I, and D exhibited mean 28-day strength values ranging from 4200 psi to 2983 psi, all far better than the strongest sodium silicate sample, quarry I (28-day = 1500 psi). In fact, the quarry H sample performed very well, exhibiting a mean 28-day strength of 4200 psi, a result within only 250 psi of the Ottawa sand sample.

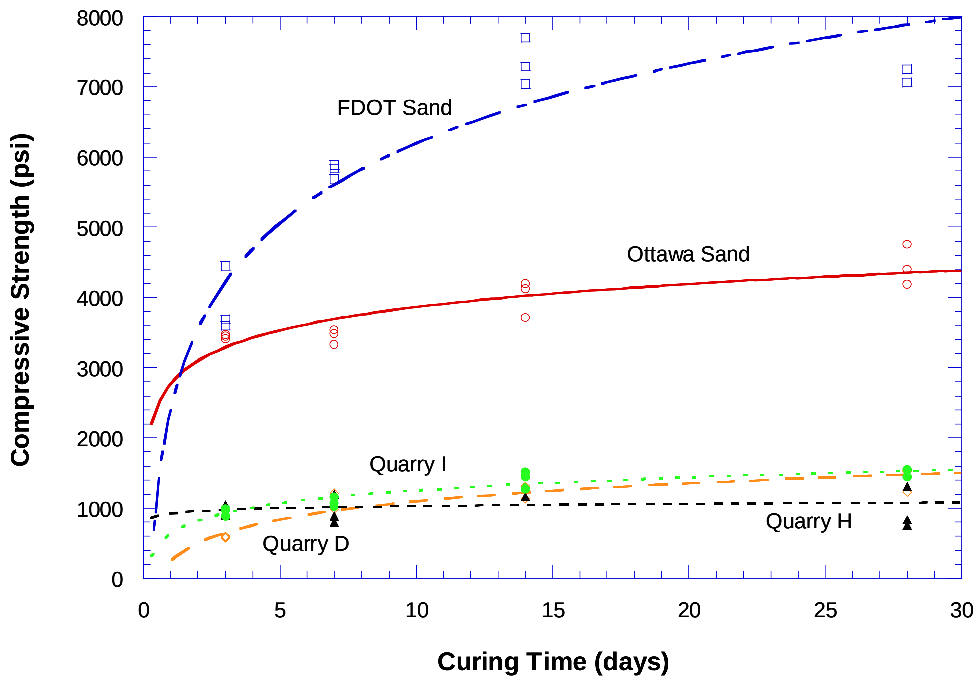


Figure 2-10. Compressive strength verses curing time for granules made with a sodium silicate binder compared to the Ottawa sand and FDOT sand standards.

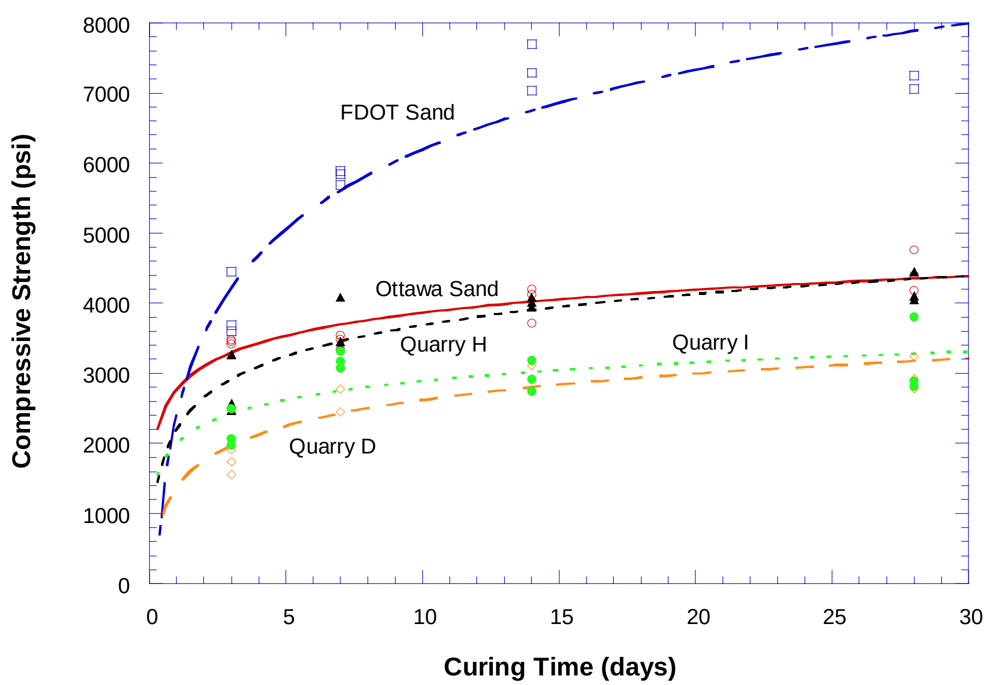


Figure 2-11. Compressive strength verses curing time for granules made with a Portland cement binder compared to the Ottawa sand and FDOT sand standards.

Scanning Electron Microscope Imaging

Scanning electron microscope (SEM) images were collected from polished thin sections of each of the types of 2"×2" test specimens studied (Fig. 2-12). The microtextural observations noted in these images were consistent with the compressive strength test results discussed previously. Figure 2-12 illustrates a representative selection of these textures.

The first two images illustrate the bonding characteristics and grain shapes encountered with the two standard materials analyzed for comparison to the manufactured aggregate samples. Figure 2-12A shows the FDOT sand to be composed of sub-angular to angular grains of varying size that seem to show better grain-paste bonding characteristics than the more rounded grains associated with the Ottawa sand (Fig. 2-12B). In fact, a crack is seen to propagate through the cement paste and along the grain border in the Ottawa sand image, illustrating the relative weakness of the grain-paste bond as compared to grain strength. Quartz grains usually possess this characteristic, resulting in bonding strength being the strength-limiting factor. It is likely that the more angular shape seen with the FDOT sand improves grain-paste bonding strength, and may be responsible for the greater compressive strength values collected for this material.

The next two images (Fig. 2-12C and 2-12D) illustrate the bonding and particle strength characteristics reflected in the compressive strength values obtained for the materials prepared with Portland cement. Figure 2-12C is an image taken from a test specimen made with quarry H granulated fines. This material exhibited the greatest strength values of any of the granulated byproduct specimens (nearly equal to Ottawa sand), as is reflected in the good bonding characteristics seen between grain boundaries and the cement paste. Furthermore the grains themselves appear to be well agglomerated, showing little evidence of either primary or secondary porosity development which might have affected granule strength. Figure 2-12D is taken from a test specimen prepared with quarry I granules. This material failed to possess strength characteristics as good as the quarry H specimens (also true for quarry D), yet exceeded all of the test specimens prepared with sodium silicate bonded granules. The difference between this specimen and the quarry H image are obvious. The grain-paste bond is far more inconsistent and porous as is the grain itself. In other images collected from this material, cracking is observed to propagate both along grain-paste boundaries and directly through grains, illustrating the weakness of these grains in comparison to the quartz grain standards studied.

Figures 2-12E (quarry D) and Figure 2-12F (quarry H) illustrate the microtexture observed in the cubes produced with granulated fines made with the sodium silicate binder. Consistent with the poor strength values collected on these specimens, both images clearly indicate poor grain-paste bonding (Fig. 2-12E) and poor grain strength (Fig. 2-12F). In fact, the last of the two images seems to show the grain cracking throughout, as if spalling apart within the concrete cube. The high secondary porosity development within the grains themselves, as well as the high primary porosity caused by poor initial agglomeration are primary causes for the poor performance of this test material.

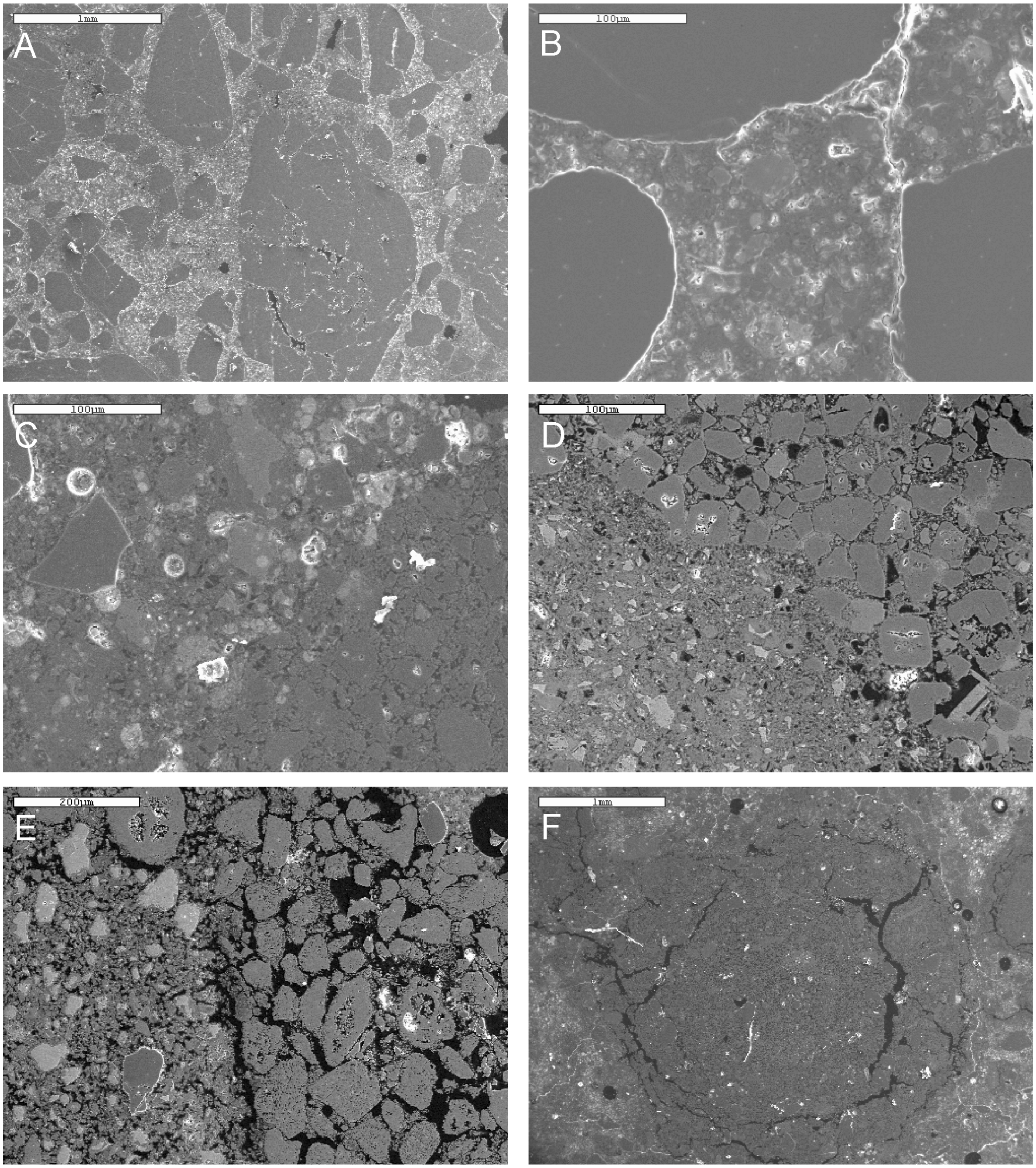


Figure 2-12. SEM images of representative 2"×2" concrete cubes tested for compressive strength: A) FDOT sand, B) Ottawa sand, C) quarry H (Portland cement binder), D) quarry I (Portland cement binder), E) quarry D (sodium silicate binder), F) quarry H (sodium silicate binder).

CONCLUSIONS AND RECOMMENDATIONS

Evaluation of the results from this section of the study, which focused on identifying potential economic uses for byproduct fines and the technical evaluation of a granular manufactured aggregate product, provided the following conclusions:

- A variety of high volume, economic applications exist for the use of byproduct fines currently underutilized in Florida, and a major waste and storage problem for coarse aggregate producers in the state. Of these, fines application in engineered backfill, particularly in ready mixed flowable fill (RFF) due to low manufactured aggregate strength, direct addition to concrete mixes, and incorporation in Portland cement concrete (PCC) as a fine aggregate replacement (both coarse fines (minus-4 by plus-40) and granulated fines) have great potential, and deserve further investigation.

- Both wet (drum granulation and pan granulation) and dry (roll-press flaking and roll-press briquetting) means of fines agglomeration are capable of producing manufactured granules that could be incorporated into PCC mixes as a fine aggregate alternative. Of these, wet processing appears to be slightly more expensive, but is favored, as it is more likely to produce a granulated product with the compressive strength requirements suitable to application to PCC. This result is expected due to the ability of wet processing to more thoroughly incorporate the binders proposed for granulation. A pug mill (similar to drum granulation) granulation process was ultimately employed due to mechanical simplicity, cost effectiveness (economics), and equipment availability.

- Three binders (sodium silicate, Portland cement, and calcium sulfate hemihydrate) were investigated, which seemed appropriate for granulation, considering the application to PCC. Of these, Portland cement consistently produced the granules with the greatest crushing strength, although quarry H materials seem to produce anomalous results in this test. The crushing strengths observed for sodium silicate were not as high as expected, and concerns were raised with respect to the sodium content creating ASR reaction problems with incorporation in PCC. Calcium sulfate hemihydrate consistently produced very coarse granules inconsistent with desired product particle-size requirements.

- An attempt to produce quarry D granules (Portland cement binder) with a California Pellet Mill were unsuccessful due to processing problems and the production of coarse granules inconsistent with particle-size guidelines.

- Evaluation of PCC containing the granulated fines produced mixed results:
    - Samples using sodium silicate as the binder performed poorly when tested for compressive strength. In fact, standards containing Ottawa sand and FDOT sand

possessed strength values as much as 4 to 7 times that of the granulated fines samples. Several factors appeared to play a role in these poor results.

  - The sodium silicate binder showed unexpected solubility when mixed with the cement to make the test cubes. It was originally assumed that the sodium silicate would react with soluble calcium from the limestones and dolomitic limestones/dolomites during wet processing to form a water insoluble compound. However, it appears that calcite is too insoluble under the test conditions applied, resulting in the solubility of the binder and poor strength characteristics of the granules.
  - Binder concentration also is a concern, as SEM images reveal that granules appear poorly agglomerated in many cases. This is true for samples employing a Portland cement binder, as well.
  - Samples prepared with a Portland cement binder performed far better than the sodium silicate test specimens. Quarry H samples performed the best, exhibiting a mean 28-day compressive strength value of 4200 psi, within 250 psi of the Ottawa sand sample. This material could be used as an alternative fine aggregate in PCC mixes, based on these results.

- A reevaluation of the binders used in granulation of the byproduct fines could improve granule strength and the PCC text results. The solubility characteristics of sodium silicate would require more careful control, perhaps through the addition of a soluble calcium source (e.g. $CaCl_2$) during wet processing to generate the insolubility required for the product. Also, binder concentration may require reevaluation, as well, for granules employing both sodium silicate and Portland cement as the binder.

Summary of Conclusions

Many factors remain to be evaluated concerning the application of granulated byproduct fines as a manufactured aggregate. Among these factors are the importance of granule shape and gradation, as well as the role of the W/C ratio and granule sodium content on the ultimate performance of construction materials produced with them. However, the potential for use of granulated fines in either PCC or even RFF have been identified, and deserve further evaluation by the FDOT as a means of extending the life span of both aggregate resources and the aggregate industry in Florida while solving a major waste and storage problem for the aggregate industry.

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