To the Graduate Council.

I am submitting herewith a thesis written by Nabeel Hafiz Abusadah entitled "Evaluation of The Quinetics Colloidal Mill in Converting Type I Portland Cement Into Microfine Cement." I have examined the final copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Civil Engineering.

N. Mike Jackson, Major Professor

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and recommend its acceptance:

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Associate Vice Chancellor and
Dean of The Graduate School

EVALUATION OF THE QUINETICS COLLOIDAL MILL DEVICE IN CONVERTING TYPE I PORTLAND CEMENT INTO MICROFINE CEMENT

A Thesis
Presented for the
Master of Science
Degree
The University of Tennessee, Knoxville

Nabeel Hafiz Abusadah
May 2000

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DEDICATION

This thesis is dedicated to my children

Yasmin N. Abusadah

Yousef N. Abusadah

Yacob N. Abusadah

Do your best, and make your mother and me proud of you all.

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The preparation of this thesis necessitated spending many hours at night and on weekends away from my family. Realizing that this lost time cannot be recovered, I wish to thank my wife and children for their understanding and support.

I humbly acknowledge that any contribution I have made is only through gifts granted by our Creator almighty.

ABSTRACT

A proprietary colloidal mill device was evaluated in the Civil Engineering laboratory at the University of Tennessee with respect to its ability to reduce the mean particle size of Type I Portland cement to sizes typical of micro-fine cement.

The Coulter Multisizer II equipment was used for the evaluation of the particle size of the slurry produced by the colloidal mill. The main reason for electing the use of Coulter Multisizer II was that results obtained during base line testing demonstrated that this equipment is very reliable. The other factor that is equally important is the speed in obtaining test results with the Coulter Multisizer II as compared to other available methods.

The effect of the particle size reduction (after mill process) on cement-slurry viscosity was studied by performing flow cone test. It was observed that improved dispersion has the effect of reducing the viscosity of the water-cement mix.

Also the effect of the particle size reduction (after mill process) on the compressive strength of sand-cement grout made from the resulting cement was also studied by forming grout cubes, which were tested for compressive strength in the laboratory.

Test results indicate that the subject mill is ineffective in reducing the mean particle size of the Type I Portland cement. In addition, the compressive strength tests showed no improvement in the strength of the grout, which was another compelling piece of evidence that there was no appreciable particle size reduction.

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CHAPTER 1.0

INTRODUCTION

1.1 Background

A proprietary colloidal mill device, developed by Quinetics of San Antonio, Texas, has been proposed for the manufacture of Microfine cement. The Quinetics colloidal mill device functions by directing two streams of cement slurry at one another at a high velocity. This high velocity collision is supposed to result in a significant reduction in particle size. A schematic diagram of the Quinetics colloidal mill device is presented in Figure 1, and a photograph of the device is presented as Figure 2.

Because of the significant difference in cost for Microfine cement versus Type I Portland cement (approximately $0.06 per pound for typical Type I Portland cement versus $0.60 per pound for commercially available Microfine cement), Rembco Engineering Corporation recognized the potential market value of the Quinetics colloidal mill to the grouting and construction industry. Rembco Engineering Corporation

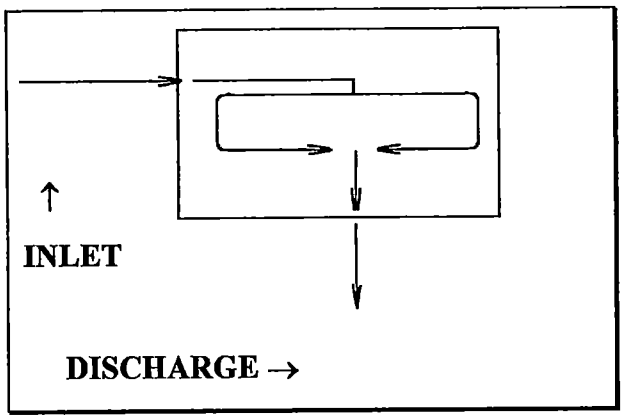


Figure 1. Schematic Diagram of the Quinetics Colloidal Mill Device.

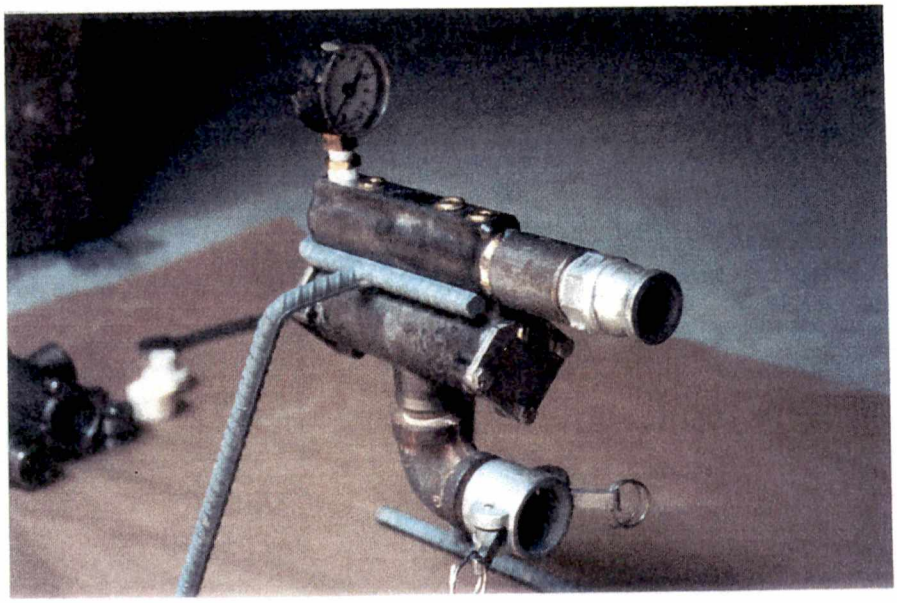


Figure 2. Proprietary Colloidal Mill Device

obtained the rights to use this proprietary device in hopes of converting the less expensive Type I Portland cement into Microfine cement. Subsequently, Rembco Engineering Corporation entered into a research contract with the University of Tennessee to evaluate this proposed process.

1.2 Objective

The objective of this research effort was the evaluation of the mean particle size of Portland cement slurry produced by the Quinetics colloidal mill device. To achieve this objective, the mean particle size of the product of the colloidal mill was compared with slurry made from typical Type I Portland cement and slurry made with commercially available Microfine cement.

1.3 Components of Study

This study consisted of six primary components. The general steps taken to achieve the objectives of this study were as follows:

1. Literature Review;
2. Preliminary Testing (Modified Hydrometer Test);
3. Base Line Testing;
4. Advanced Testing (Coulter Multisizer II),
5. Documentation and Analysis of Test Results; and
6. Conclusions and Recommendations.

CHAPTER 2.0

LITERATURE REVIEW

2.1 Introduction

Microfine cement is an extremely fine cementitious grouting material that is used in geotechnical and environmental applications to improve the stability and reduce the permeability of soil, rock and other substrates. It can also be described as a cement-based grouting material with at least 50% of particle sizes less than 4 μm. It exhibits excellent permeation, strength, and durability when compared with ordinary Type I Portland cement, which has a mean particle size of 22 μm (Clarke, 1984). Microfine cement is also known to be free of the toxic substances found in some solution grouts. The 4 μm particle size is small enough to penetrate voids and crevices where only chemical grouts could reach in the past, making it ideal for sealing fractured rock, porous soils, and damaged concrete. This type of cement is excellent for permeation grouting of dams, foundations, tunnels, and waste disposal sites. Selective examples that will be presented in this chapter include the use of Microfine cement for grouting, repairing of reinforced concrete, and hazardous waste stabilization.

2.2 Microfine Cement for Special Applications

Recent developments in the applications of Microfine cement have been documented by researchers in Japan, with special emphasis on the Japanese experience (Uchikawa, 1994). The Japanese note that the cementitious binder has been used as grout for reinforcing foundation rock and stopping water leakage in the construction of a tunnel, a dam, a high-rise building, and a subsurface structure. Production of Microfine

cement with uniform particle-size distributions has recently become possible by the remarkable progress of grinding technologies. As a result, reduction of viscosity and the improvement of permeation of Microfine cement suspensions have been achieved, and it has gained greater attention as a grouting material. Microfine cement is also attracting attention as a filler for repairing deteriorated parts and cracks in concrete structures.

2.2.1 Grouting Applications

Additional grinding of the cement particles, regardless of type, increases hydration rate and slurry viscosity (Uchikawa, 1994). The particle-size distribution of cement for grouting is optimized by removing the particle sizes of 10 μm or larger to improve permeation and by separating the 2 μm or smaller sized particles for inhibiting the increase of viscosity after mixing with water. A mixture composed of 20% to 30% of Microfine cement, and 70% to 80% blast furnace slag is typically used for grouting purposes in Japan.

Although cement contributes to the increase of suspension viscosity, the viscosity can be reduced by the addition of an admixture. For example, the suspension viscosity can be reduced to as low as 8 centipoises (cp) at a water-to-cement (w/c) ratio of 2.0 by the addition of an admixture. Microfine cement with a Blaine's specific surface area of 10,000 cm^2/g or larger can permeate a sand with mean particle diameters D_{50} on the order of 0.18 mm, while a grout with a mean size as large as ordinary Portland cement can only permeate a layer composed of larger sand particles.

Microfine cement based grout has been widely used for various Japanese construction projects. These projects include the building of the Nakayama Tunnel on

Joetsu Shinkansen, the building of tunnels for the construction of the Kashiwazaki Nuclear Power Plant for Tokyo Electric Power Co., and the improvement of load bearing capacity of soil in the construction of the Sayama Dam (Yoneda et al. 1993).

2.2.2 Concrete Repair

Cracking may compromise the structural integrity of concrete by reducing the yield strength, and durability. Repairing cracks in concrete structures is often required for maintaining function and service life. The properties of interest in the repair of concrete include viscosity, permeability, impregnation, shrinkage, strength, adhesion, and durability.

Cracks 0.1 to 0.5 mm wide are typically filled with an organic resin-based material. Although the resin-based grouting material has advantages, including superior impregnation properties and high adhesion, it also has shortcomings, including inapplicability to wet parts, much higher coefficient of thermal expansion than that of concrete, insufficient strength development at low temperatures, and higher cost. Using Microfine cement and adding polymer dispersants and blending components has developed new grouting materials with superior impregnation properties, high strength, and high durability. Blast-furnace slag is also frequently used as a blending component.

Styrene butadiene rubber (SBR) based polymers are most suitable for decreasing the viscosity. Adding an accelerator can control the variation of viscosity with time. It has been demonstrated that the strength, adhesion, and the shrinkage can be sustained by using Microfine cement (Wakasugi, et al. 1993).

2.2.3 Hazardous Waste Stabilization

In the summer of 1996, low pressure permeation grouting was performed inside parts of four shallow unlined disposal trenches at a low level radioactive waste burial site at the Oak Ridge National Laboratory. The project's goal was to trap and retain radioactive material during its natural degradation cycle and reduce the release of chemical contamination to a very small amount. This remediation option was chosen to eliminate excavation and minimize long term operating and maintenance costs. A unique grouting methodology was used to control interaction of wastes with natural runoff. A combination of Portland cement based- grouts, Microfine cement-based grouts, and acrylamide solution grouts were injected into driven steel pipes. Apparent Lugeon values were calculated during grouting and grout formulations were modified during injection to maximize permeation, achieve durability, and obtain a maximum reduction in permeability. About 520 m^3 (18,300 ft^3 or 137,600 gallons) of grout were placed. Assessment at the completion of grouting showed an average residual permeability of 10^{-6} cm/sec. An immediate decrease in the amount of released strontuim-90 (^{90}Sr) was discovered at adjacent monitoring wells and hydrologic monitoring has been established to determine the long-term effectiveness of the project. When monitoring wells directly below the grouted trenches were sampled, these specific areas showed a reduction of about 25% in the ^{90}Sr. (Berry and Narduzzo, 1997).

2.3 Comparison of Material Properties

2.3.1 Introduction

Material properties of interest in this study include particle size distribution, compressive strength, and flow characteristics. Compressive strength is influenced by cement type, the compound composition, and cement particle size. Usually compressive strength for grout is obtained from tests of standard 2-in. mortar cubes tested in accordance with ASTM C109. These cubes are made and cured in accordance with established procedures. ASTM C 150 sets only a minimum strength requirement that is well met by most manufacturers. As a result, two types of Portland cement meeting the same minimum requirements may not necessarily produce the same strength of mortar. In general, cement strengths (based on mortar-cube tests) cannot be used to conclusively predict grout strength due to differences in fine aggregate characteristics, grout mixtures, and construction procedures (Kosmatka, and Panarese, 1994).

2.3.2 Particle Size and Flow Characteristics

Typical gradation curves for the different types of cement were obtained through a literature review. Figure 3 Illustrates that Microfine cement (MC 100, MC 300, and MC 500) typically has the smallest particle size in relation to Portland cement, types I & III.

Figure 4 presents typical viscosity curves for Microfine versus Portland cement slurries. As presented, the water-to-cement ratio (w/c) and the cement type are determining factors in the flow characteristics of slurry.

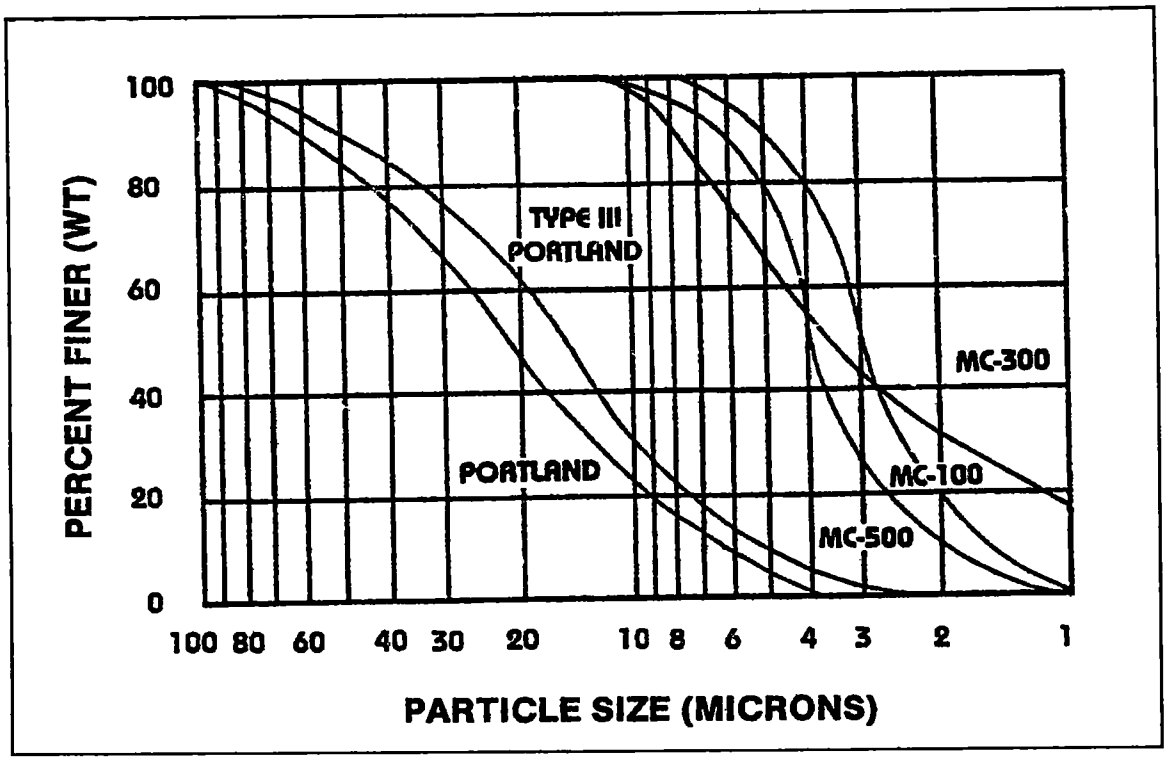


Figure 3. Typical Gradation Curves (Clarke, 1984)

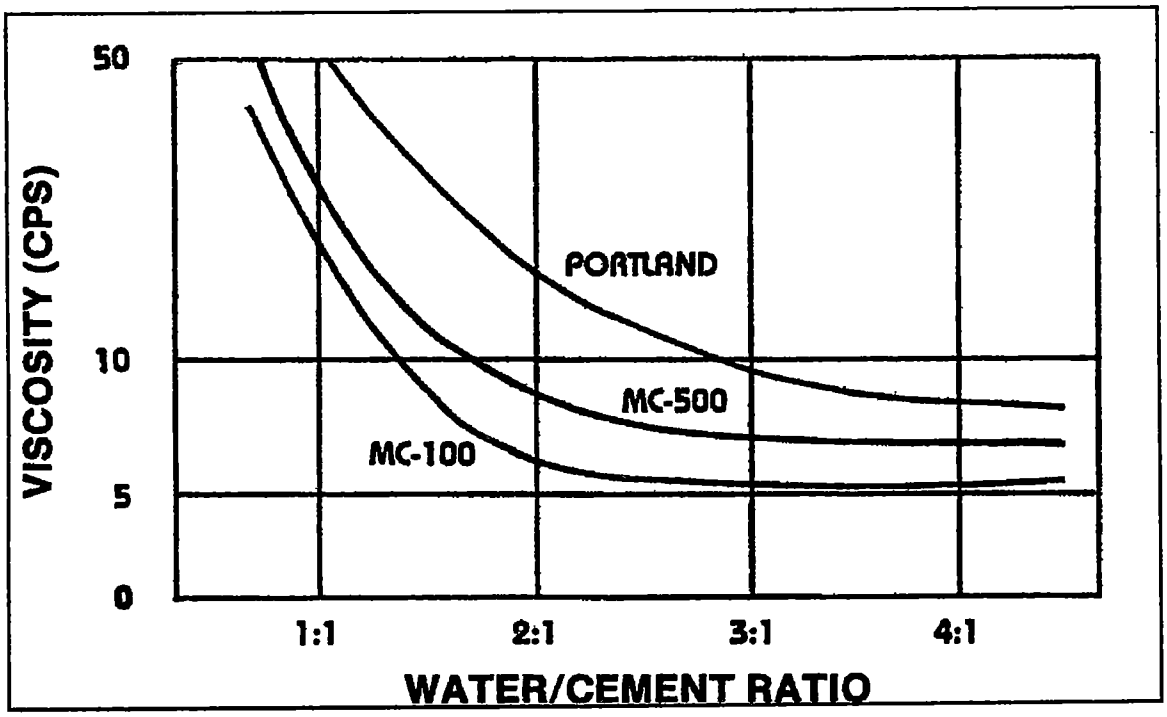


Figure 4. Typical Viscosity Curves (Clarke, 1984)

2.3.3 Physical Properties

Pertinent physical properties of Portland cement include fineness (particle size), specific gravity, unit weight, odor, and appearance. A brief description of each of these properties is provided below, and summarized in Table 1

The cement fineness affects the rate of heat released during hydration. Strength development is also accelerated by increased fineness. (Kosmatka, and Panarese, 1994). Specific gravity of a cement, which is determined by ASTM C188, is not a direct indication of the cement's quality; its principal use is in mixture-proportioning calculations. The physical properties of Microfine and typical Portland cement are summarized in Table 1.

2.3.4 Chemical Composition

The principal constituents of Portland cement are calcium silicates, ferro-aluminates and sulfates. Small amounts of alkalis, lime and chlorides are also present, together with trace amounts of chromium compounds.

Table 1. Physical Properties (Kosmatka, and Panarese, 1994), and (Clarke, 1984).

Cement Types		**Microfine cement**	**Type I Portland cement**
Specific gravity		3.00± 0.10	3.15
Unit weight (lb/cu. ft)		About 62	94
Odor		None	None
Fineness	Blaine Specific Area (cm2/g)	About 9000	3170
	Mean Particle	4μm	22μm

During the burning operation in the manufacture of Portland cement clinker, calcium oxide combines with the acidic components of the raw mix to form four principal compounds that make up 90% of cement by weight. Gypsum and other materials are also present. The primary compounds, their chemical formulas, and abbreviations are presented in Table 2.

The approximate percentage of each compound can be calculated from a chemical analysis of the cement. X-ray diffraction techniques may be used to more accurately determine compound percentages. Table 3 below summarizes the typical chemical composition of each type of cement evaluated in this study.

Table 2. Principal Compounds of Portland Cement

Primary Compounds	**Chemical Formulas**	**Abbreviations**
Tricalcium silicate	$3CaO \cdot SiO_2$	C_3S
Dicalcium silicate	$2CaO \cdot SiO_2$	C_2S
Tricalcuim aluminate	$3CaO.Al_2O_3$	C_3A
Tetracalcium aluminoferrite	$4CaO.Al_2O_3.Fe_2O_3$	C_4AF

Table 3. Chemical Composition

Type of Cement	Chemical composition, %					
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3
Type I	20.9	5.2	2.3	64.4	2.8	2.9
Type III	21.3	5.1	2.3	64.9	3.0	3.1
Microfine	30.6	12.4	1.1	48.4	5.8	.8

2.3.5 Permeation

The relative permeation of Microfine cement is compared with that of Type I Portland cement in Figure 5 Microfine cement, because of its fineness, exhibits extremely good permeation, or penetration into cracks in rock, soil and concrete and provides efficient tightness combined with good durability.

2.3.6 Permeability

Permeability is a constant of proportionality related to how easily a fluid passes through a porous medium. The coefficient of permeability of a homogeneous, isotropic soil mass depends primarily on the following factors (Das, 1983):

1- The viscosity of the fluid.

2- The void ratio e of the soil.

3- The size and the shape of the individual particles.

4- The degree of saturation.

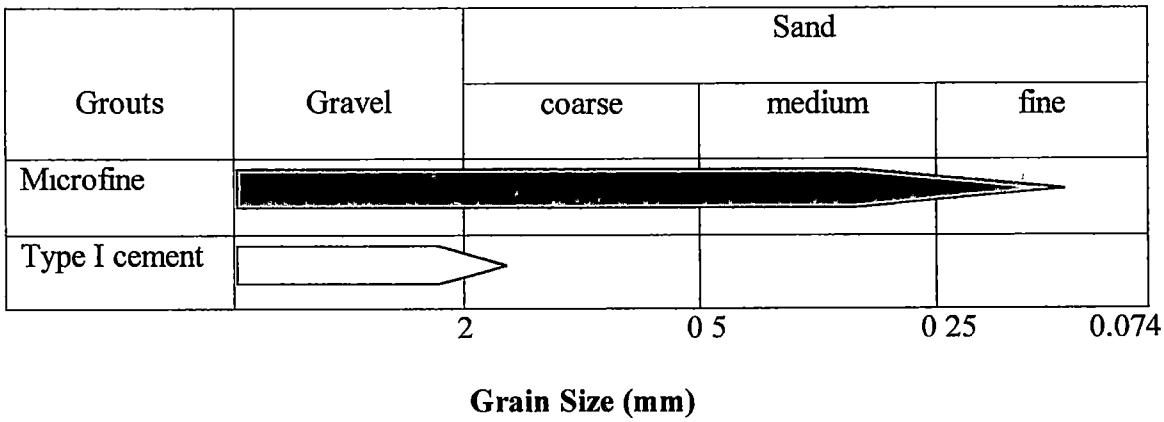


Figure 5. Relative Permeation of Different Cements (Clark, 1984)

The permeability of hardened Microfine cement is far less than that of Type I Portland cement, as illustrated in Figure 6. This low permeability can be attributed to the fact that the particle size of Microfine cement is less than that of Type I Portland cement.

2.4 Dispersion

2.4.1 Introduction

Dispersion may be defined as the spreading of marked fluid particles by the combined action of a nonuniform velocity distribution and diffusion (Streeter and Wylie, 1979). The mechanism of electrolytes as accelerators for the hardening of cement systems is highly complex and not fully understood. It should be noted that only a few researchers have examined the effects of electrolytes in water-cement dispersions (Sychev et al, 1979).

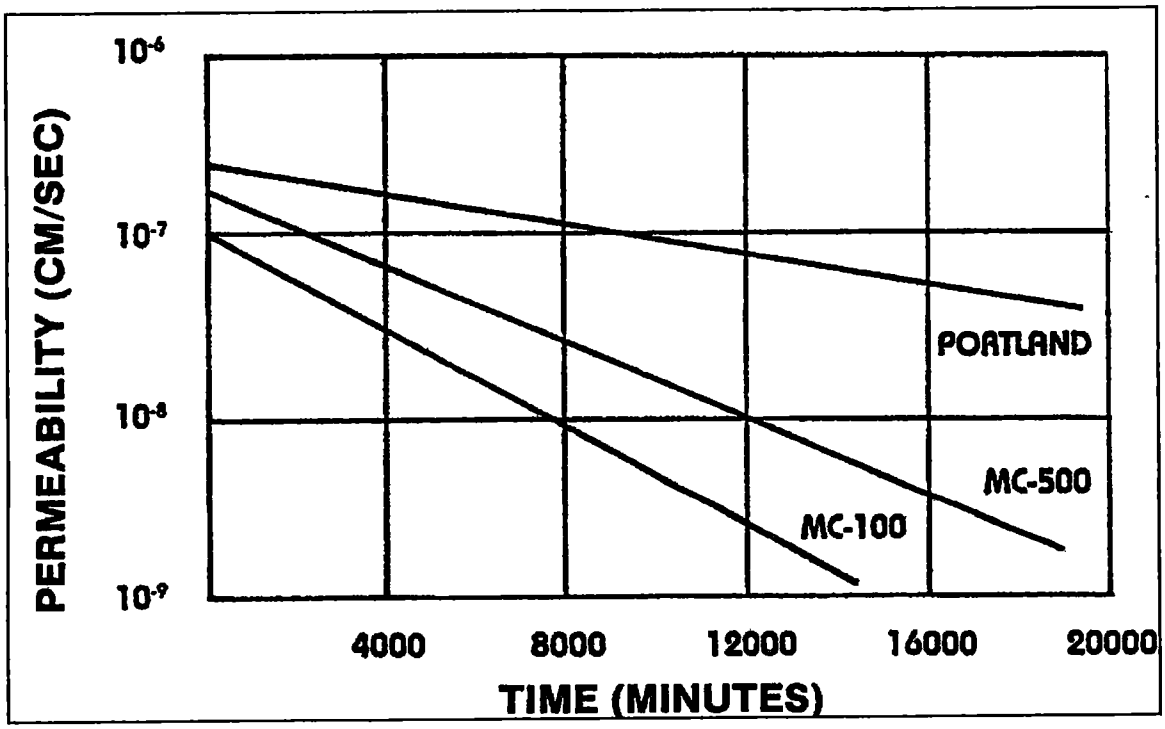


Figure 6. Permeability vs. Cement Types (Clarke, 1984)

2.4.2 Dispersion Mechanism

The rate of cement hydration is determined primarily by the rate of dissolution of the clinker minerals and the rate of their transfer into the liquid phase, which itself depends on the rate of adsorption of water molecules on the surface of the solid phase (Kapranov, 1976). It has been shown that adsorption of water molecules is energetically easier in the presence of unsaturated hydrogen bonds. Establishment of such hydrogen bonds is easily achieved since the structure of minerals in Portland cement clinker consist of ionic-covalent crystals containing in their lattices strongly electronegative oxygen atoms, which are active proton acceptors. Hydrogen bonds form between the hydrate complexes and the surface of the cement crystals, with subsequent dissociation of water and adsorption of H^+ and OH^- ions on the cement. This lowers substantially the bond energy of ions in the lattices and leads to intensified removal both of ions and ion pairs, and their transfer into the liquid phase.

2.4.3 Effects of Dispersions on the Property of the Cement Slurry

The decrease in the degree of hydroxylation of cement particle surface and the conversion of part of the physically adsorbed water to the free state are responsible for the acceleration of hydration. Simultaneously, the dispersity of the system is observed to increase (Sychev and Tsement, 1984).

The influence of electrolyte solutions on hydration in water-cement systems is put forward on the basis of analysis of the structure of the solution. It has been shown that the effect of added electrolytes results in acceleration of dissolution of cement minerals due

to an increase in the concentration of hydrogen bonds between the liquid and solid phases.

In summary, the process of dispersion results in the reduction of the bond between water and cement particles. As a result, a dispersed water-cement mix has lower resistance to flow. The viscosity of a water-cement mix is the measure of the resistance to flow; water-cement mix with lower viscosity has a lower resistance to flow. Therefore, improved dispersion has the effect of reducing the viscosity of the water-cement mix.

2.5 Coulter Multisizer II

2.5.1 Introduction

As a major part of this study an available Coulter Multisizer II machine at the University of Tennessee was used to evaluate the particle size distributions of the different cement types in dry powder and slurry forms. The following paragraphs provide a comprehensive description of this machine and its function, general errors in collecting data, operational procedures, and limitations encountered during testing.

2.5.2 Equipment Description and Function.

The Coulter Multisizer II, a particle size analyzer employing the electrical-zone method, is commonly used for measuring the number concentration and size distribution of particles or droplets suspended in an electrolyte. This machine works by drawing a known volume of suspension through an orifice, which is the only conducting path between two electrodes. A particle passing through the orifice will cause a pulse increase in resistance. Such pulses can then be counted, resulting in the estimation of the number

concentration of particles in the known volume of suspension. In addition the pulse magnitude distribution can thus measure the particle volumes distribution (Allen, 1990).

2.5.3 Coulter Multisizer II Component and Operating Steps

The Coulter Multisizer II system is divided into three separate units. The first is a PC that has special executing software; second is the Coulter Multisizer II machine (See Figure 7); and third is the Sampling Stand II (See Figure 8). This final unit, considered the main component of the system, determines the number and size of particles suspended in an electrically conductive liquid by forcing the suspension to flow through a small aperture having an immersed electrode on either side. Test samples are also introduced at this unit. (See Appendix A for specific steps to be followed in operating the Coulter Multisizer II system).

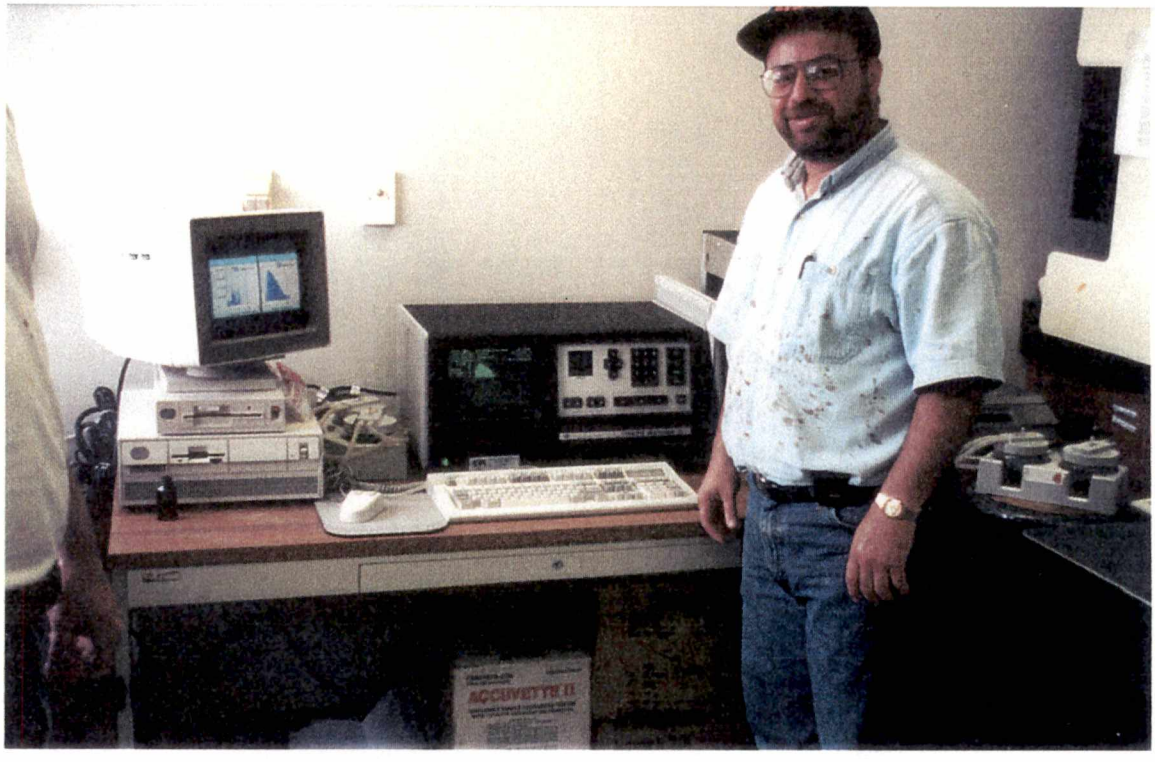


Figure 7. Coulter Multisizer II Unit.



Figure 8. Sampling Stand II

2.5.4 General Error

Particle size analyzers using the electrical-zone method assume that particles pass through an orifice tube one at a time. In fact, two or more particles will sometimes pass through the orifice tube roughly simultaneously. This is called coincidence, and is a source of error in the measurement from this machine. Coincidence reduces the observed number of particles and increases the observed average particle size. There will inevitably be a 'background' of unrelated particles (See Figure 9), which must be minimized by careful filtering and handling of the solutions.

Use of the machine relies on the assumption that particles pass through singly, so that each counted pulse corresponds to a single particle, and that the pulse magnitude

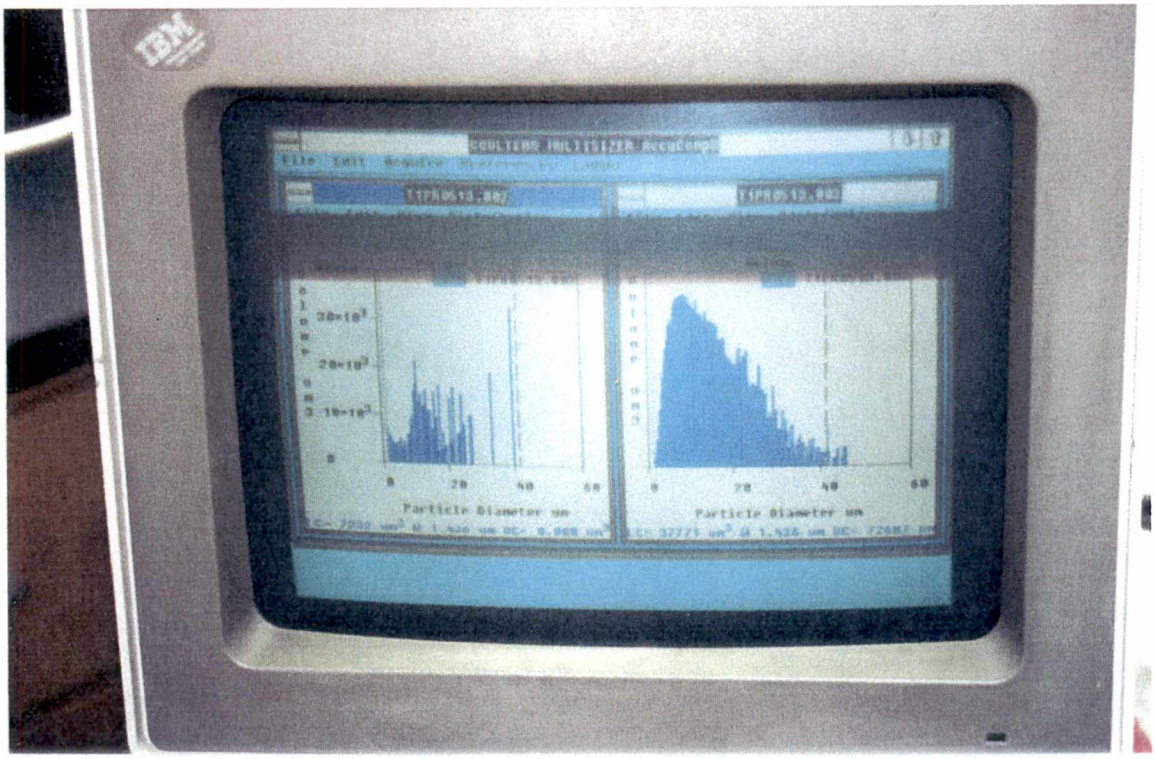


Figure 9. Opened Background and Sample Files Used in the Particle Size Analysis.

corresponds to a single particle size. However, if two or more particles happen to pass through the orifice in close proximity, the machine may interpret the resulting signal as a single particle. This is called primary coincidence (Wynn and Hounslow, 1997), and causes a reduction in the observed number of particles. If the particles pass through the orifice almost simultaneously, the resulting signal will be a single pulse, and the magnitude of the single pulse will equal the sum of the particles' volumes. This is called vertical interaction (Wynn and Hounslow, 1997); it does not affect the total particulate volume that is observed, but it does increase the observed mean size. The observed size distribution therefore tends to be flattened slightly and skewed to larger sizes.

Particles that pass through the orifice soon after other particles, or that pass through the orifice off-center or with a tumbling motion, may cause overlapping, or otherwise misshapen pulses. The analyzer's software must process these in some way; one is simply to ignore all misshapen pulses. The other is by employing extremely dilute suspension (Wynn and Hounslow, 1997). Machine manufacturers commonly recommend upper limits of number concentration to avoid a specified amount of coincidence.

CHAPTER 3.0

PRELIMINARY EVALUATION OF COLLOIDAL MILL – PHASE I

3.1 Introduction

The properties of the slurry produced from the colloidal mill were compared with slurries made from ordinary Type I Portland cement and commercially available Microfine cement.

3.2 Preliminary Base-Line Testing

Various laboratory-testing procedures were considered for the evaluation of the particle size of the slurry processed by the colloidal mill. ASTM Procedure D 422, Standard Specification for Particle Size Analysis of Soils (ASTM, 1998) was selected as a preliminary method to evaluate relative changes in particle size because of its low cost and availability and because more sophisticated than methods typically used for Portland cement require the test sample to be in the form of a dry powder prior to testing. The standard hydrometer test method was modified slightly to minimize hydration of the cement particles during testing. A chemical admixture was selected for use as a dispersing agent and to stop hydration of the cement slurry during testing. The chemical admixture used in this study is commercially available from Master Builders, Inc. and is marketed under the trade name Delvo. Delvo is marketed as a product that stops hydration for up to 3 days by forming a protective coating around the Portland cement particles (See Appendix B for Modified Hydrometer Testing Protocol).

In order to evaluate the effectiveness of the colloidal mill in reducing the size of typical Portland cement particles, it was necessary to know the particle size distribution

of the parent Portland cement. It was also desirable to know the particle size distribution of commercially available Microfine cement. Samples of Lonestar Type I Portland cement and Lehigh Portland Cement Company Microcem B Microfine cement were tested with a Coulter LS Particle Size Analyzer at Southdown's Victorville, California laboratory. The results of these analyses are summarized in Table 4.

3.3 Preliminary Laboratory Testing Program

Preliminary hydrometer tests (See Figure 10) were run on slurry samples of Lonestar Type I Portland cement to evaluate the effects of Delvo on the test results. The results of these modified hydrometer tests, with and without the use of Delvo, are

Table 4. Summary Results of Coulter LS Particle Size Analyses of Typical Type I and Microfine Cements.

Lonestar Type I Portland Cement		Microcem B Microfine Cement	
Particle Diameter, μm	**Percent Finer**	**Particle Diameter, μm**	**Percent Finer**
47.590	90.0	7.496	90.0
29.140	75.0	5.354	75.0
13.130	50.0	3.182	50.0
4.494	25.0	1.508	25.0
1.529	10.0	0.810	10.0

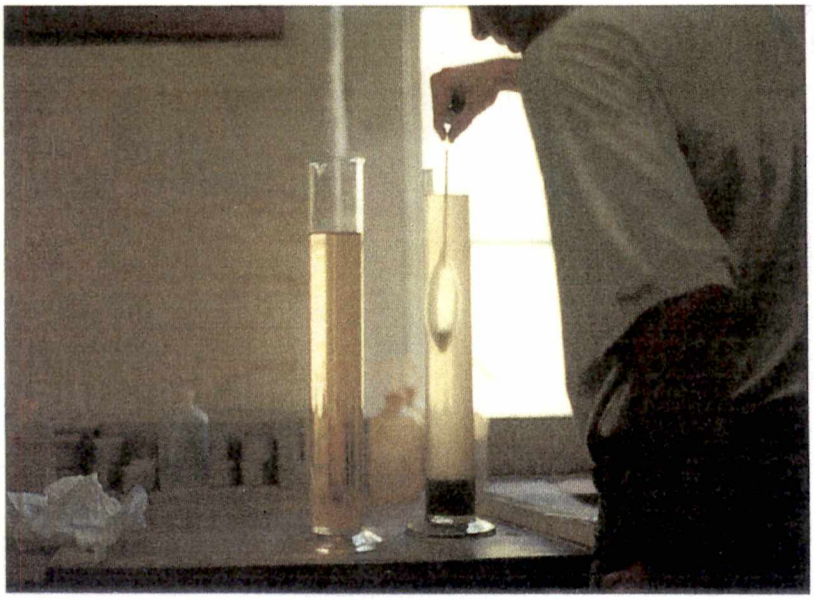


Figure 10. Hydrometer Test

presented in Figure 11. For comparison purposes, the results of the Coulter LS Particle Size Analysis, conducted on a sample of the Lonestar Type I Portland cement, are also presented in Figure 11. As shown in the Figure, the hydrometer test conducted with Delvo resulted in a more realistic particle size distribution (closer to the results provided by the Coulter LS Particle Size Analysis) than the test conducted without Delvo. Based on this observation, it was decided to proceed with the hydrometer test with an appropriate dosage of Delvo to evaluate relative differences in Portland cement particle sizes.

A series of hydrometer tests were run to determine the optimum Delvo dosage to be used in the test. Based on this preliminary study, a dosage rate of approximately 5%

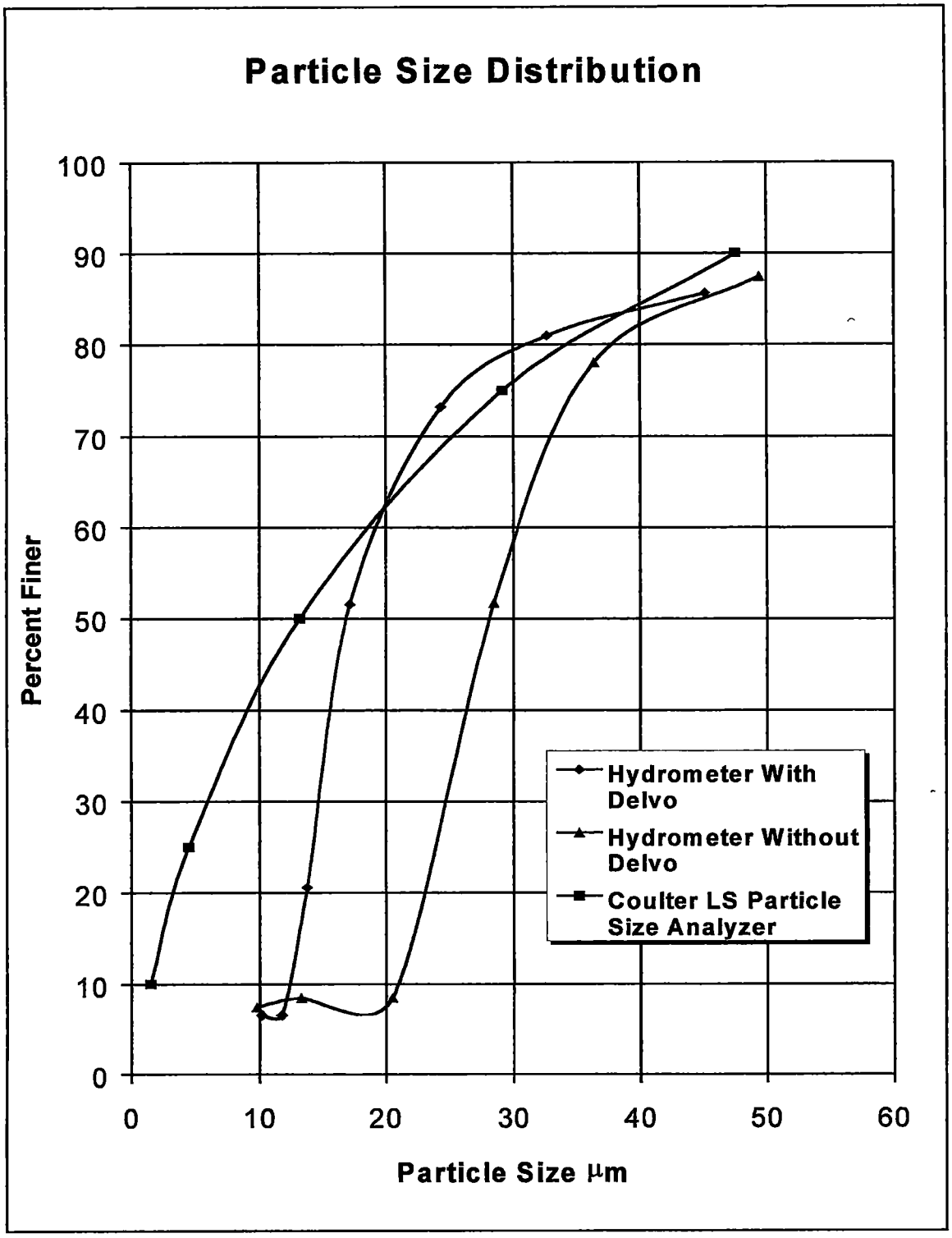


Figure 11. Typical Particle Size Analysis Test Results, Type I Portland Cement.

by weight of cement was identified as a reasonable dosage for the purposes of this test program.

Next, slurries containing Lehigh Microcem B and Type I Portland cement were tested by the hydrometer procedure and compared with the particle size analysis results provided by the Coulter LS Particle Size Analyzer. The results of these tests are presented in Figures 12 and 13. As presented in Figures 12 and 13, the hydrometer analysis was found to consistently result in particle size distributions coarser than those resulting from the Coulter LS Particle Size Analyzer. The results of the Coulter LS Particle Size Analyzer for samples of the Microcem B and Type I Portland cement are presented in Figure 14. As can be seen in Figure 14, the Coulter LS Particle Size Analyzer clearly differentiates between the Microfine cement and the Type I Portland cement. As shown in Figure 15, the hydrometer results also distinguish the relative differences in particle sizes of the Microfine cement from the Type I Portland cement. It should be noted that the relative magnitude of the difference in particle size between the two slurries is similar for the respective test methods. Based on this observation, the hydrometer test method was deemed suitable for a preliminary evaluation of relative changes in particle size of cement after processing through the colloidal mill. It is important to emphasize that the particle size distribution obtained for a given cement slurry by the hydrometer method is known to be coarser than the actual particle size distribution, as determined by the Coulter LS Particle Size Analyzer or other more sophisticated test equipment.

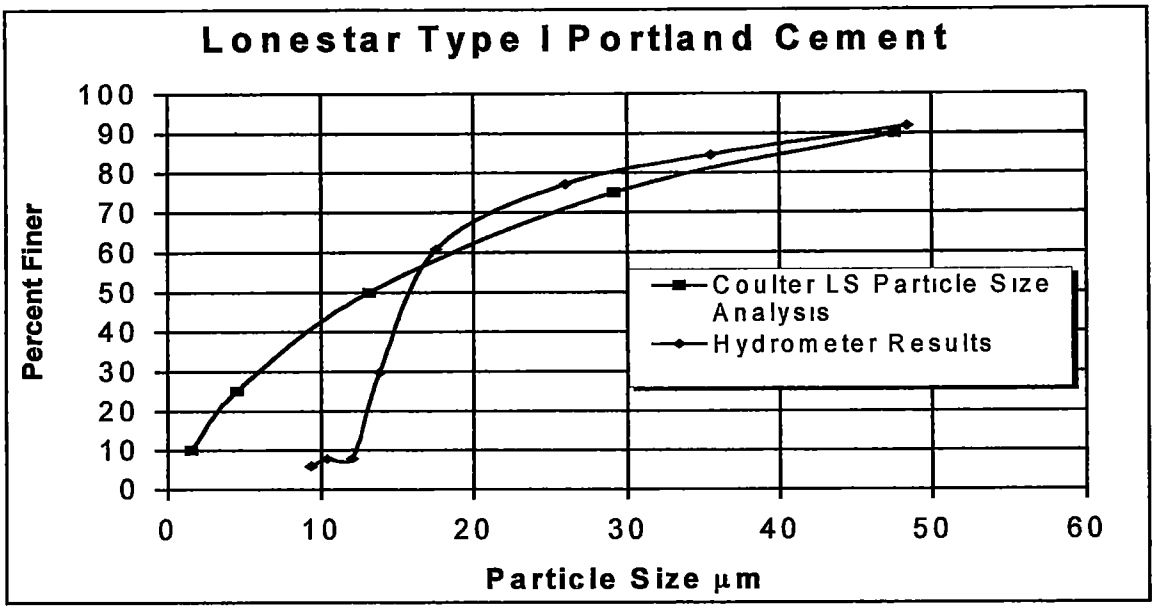


Figure 12. Comparison of Hydrometer Test Results with Coulter LS Particle Size Analysis for Lonestar Type I Portland Cement.

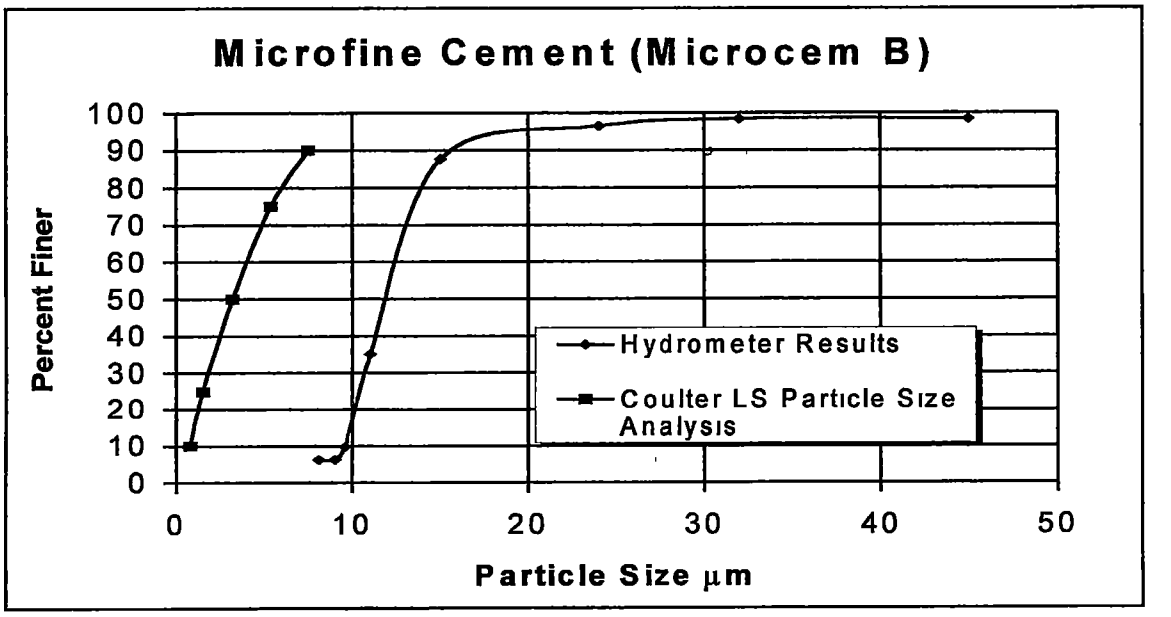


Figure 13. Comparison of Hydrometer Test Results with Coulter LS Particle Size Analysis for Microcem B Microfine Cement.

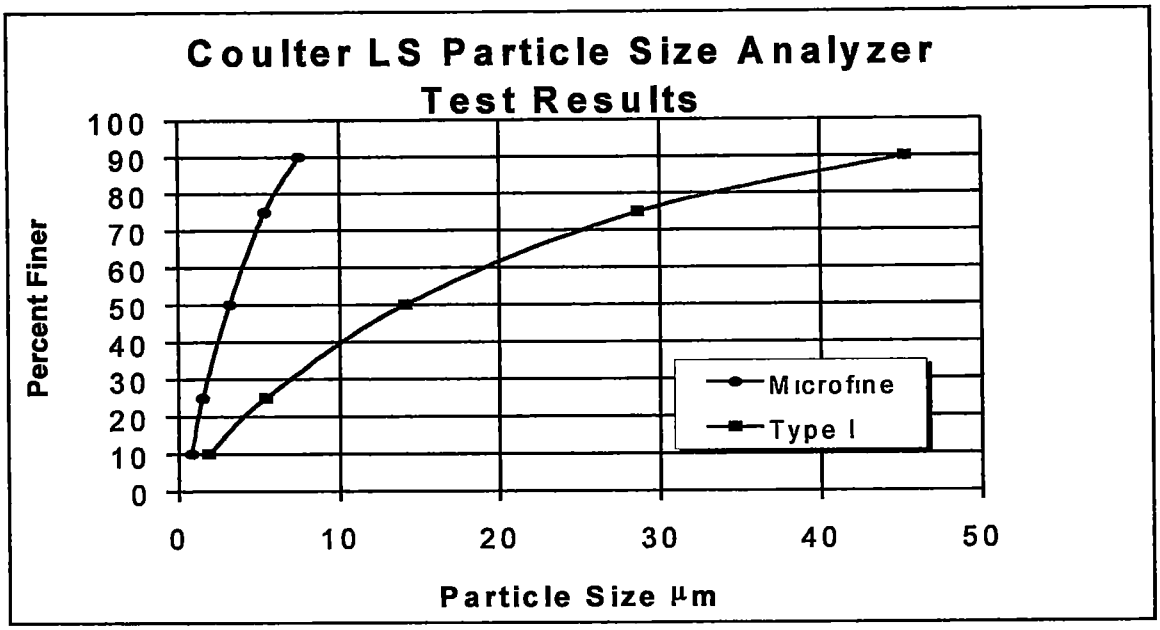


Figure 14. Coulter LS Particle Size Analyzer Results for Lonestar Type I Portland Cement and Microcem B Microfine Cement.

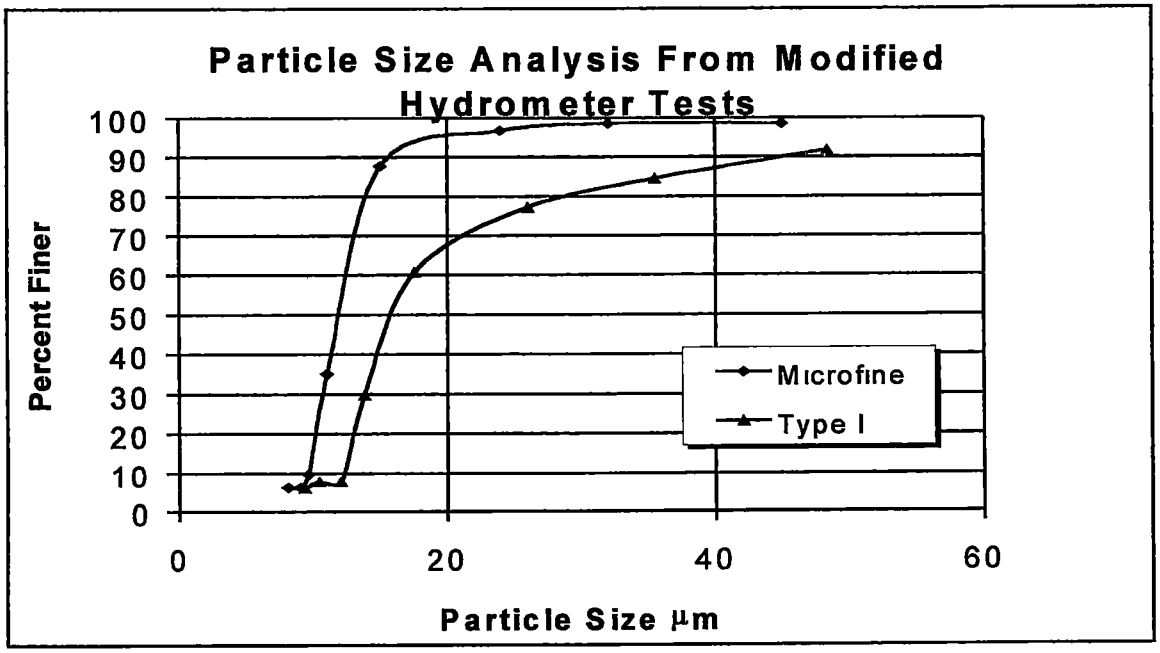


Figure 15. Modified Hydrometer Test Results for Microfine Cement Versus Type I Portland Cement.

3.4 Laboratory Trial Runs

Several trial batches of Type I Portland cement slurry were processed through the Quinetics colloidal mill. The different batches were processed with water-to-cement ratios ranging from 0.5 to 2.0. It should be noted that the upper end of this range in water-to-cement ratio is in excess of what would be typical for Portland cement concrete mixes, but is not uncommon for permeation grouting applications.

3.5 Preliminary Test Results

Preliminary results are presented in Figure 16. Based on the preliminary results illustrated in Figure 16, it appears that the particle size of the Type I Portland cement is reduced as a result of passing the slurry through the Quinetics colloidal mill.

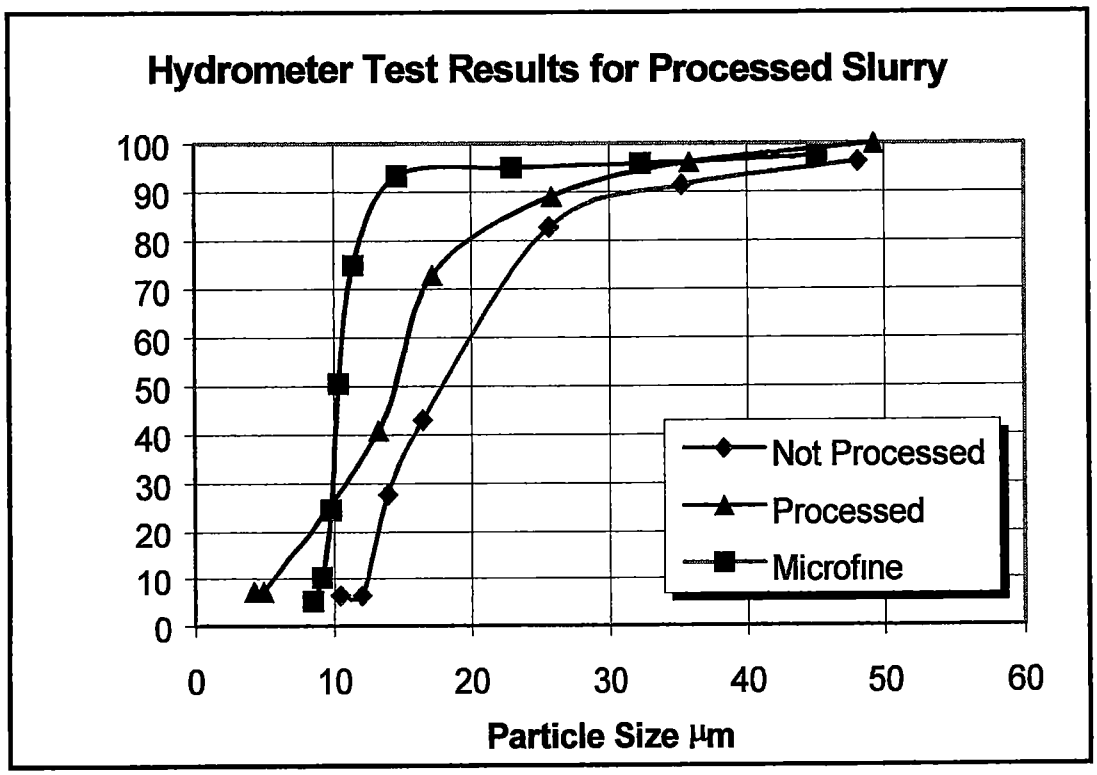


Figure 16. Typical Hydrometer Test Results for Processed Versus Non-Processed Type I Portland Cement and Microfine Cement.

The particle size distribution of the processed slurry was found to be finer than that of the parent cement based on the hydrometer method. This observation suggested that the Quinetics colloidal mill did slightly reduce the mean particle size of Type I cement. Additional testing with more sophisticated measuring equipment was deemed necessary to verify the test results obtained in this preliminary test program.

CHAPTER 4.0

ADVANCED TESTING OF COLLOIDAL MILL – PHASE II

4.1 Introduction

The advanced testing program for this study consisted of approved ASTM methods that are believed to be applicable and helpful in determining the effectiveness of the colloidal mill in reducing the particle size of Type I Portland cement. The following is the outline of the laboratory work performed:

1. Five colloidal mill trial batch runs were conducted by mixing slurry with water cement ratio of 0.5 and 1.0 (See Appendix A for colloidal mill operating steps and graph). A photograph of the colloidal mill mixing and processing equipment is presented in Figure 17.
2. Particle size distribution analyses were performed on the slurry products using the Coulter Multisizer II.
3. The Marsh Flow Cone test was also performed in accordance with ASTM method C 939 "Standard Test Method for Flow of Grout for Preplaced-Aggregate Concrete, Flow Cone Method". (See Appendix B for Marsh Flow Cone Testing Protocol)
4. Mortar cubes were prepared in accordance with ASTM C-109 and compressive strength testing was conducted in accordance with ASTM C-130. Mortar Cubes were prepared and tested for Type I Portland cement slurry before and after processing through the colloidal mill.



Figure 17. Slurry Mixing Operation, and Colloidal Mill in Use

4.2 Advanced Base-Line Testing – Phase II

As described in the preliminary testing phase of this study, it was necessary to know the particle size distribution of slurry products before and after processing through the colloidal mill. The particle size distributions for all materials evaluated in this advanced testing phase were determined by the Coulter Multisizer II equipment housed at the University of Tennessee. A base line testing program was performed to verify the accuracy of this equipment. This program consisted of determining the particle size distribution for unprocessed Type I Portland cement, unprocessed Type III Portland cement, and commercially available Microfine cement. The results were compared with published particle size distributions for these same cement products.

Particle size distributions were analyzed for dry powder and slurry. Each series consisted of at least three runs. The final results of each series were determined from the

average of the different runs. As previously noted, retarding agent Delvo was introduced to the slurry mix series for the purpose of preventing cement hydration for a specific and desirable length of time. (See Appendix C-1 for Base-Line Test Data)

4.2.1 Sample Preparation

Test sample preparation for dry powder cement consisted of adding 1 gram of dry cement to 100 ml of Isoton solution. For the slurry mix series, the water-to-cement ratio was 1:1, which translated to 10 grams of water added to 10 grams of cement with the Delvo added to the mix at a dosage of 5% by weight of cement. One drop of this slurry was introduced to 100 ml of Isoton solution for analysis in the Coulter Multisizer II.

4.2.2. Results Of Particle Size Distribution Analysis on Type I Portland Cement.

The following discussion summarizes the data obtained by using the Coulter Multisizer II equipment used in particle size analysis for the three different cement products evaluated in this study. Based on the data presented here, it is clear that the particle size of Microfine cement is the smallest among the three different cement products tested. Also by comparing results gathered by the Coulter machine to the published gradation curves provided in Figure 3 of Chapter 2, it is concluded that the Coulter Multisizer II is suitable for obtaining the actual or "true" particle size distribution for the different cement products in this study. The Coulter Multisizer II test data output for each test is provided in Appendix C.

The results for the Type I Portland cement in dry powder and slurry forms are summarized in Tables 5 and 6. By comparing these results, it is evident that the dry powder form results in a slightly smaller particle size distribution than the slurry form. In

Table 5. Type I Portland Cement in Dry Powder Form

Cement Particle Size (μm)					
Run#	**% Passing**				
	10%	**25%**	**50%**	**75%**	**90%**
1	6.788	11.43	18.68	29.85	40.10
2	7.578	12.32	20.03	30.93	41.97
3	7.646	12.97	21.7	33.52	45.63
Average (μm)	7.337	12.24	20.137	31.433	42.63

Table 6. Type I Portland Cement in Slurry Form.

Cement Particle Size (μm)					
Run#	**% Passing**				
	10%	**25%**	**50%**	**75%**	**90%**
1	8.727	14.43	22.97	32.95	43.16
2	9.035	14.89	23.68	33.67	43.57
3	10.39	16.74	25.25	35.1	47.47
Average (μm)	9.38	15.35	24.00	33.91	44.743

addition, these results were compared with the particle size distribution from published data (Figure. 3) and this comparison is presented in Figure 18. As can be seen from this figure, the particle size at 50 percent passing for the three different gradations shown is very close. The particle size of 50 percent obtained for dry powder was about 20 μm, for slurry was about 24 μm, and the particle size of 50 percent of Type I Portland cement presented in the typical gradation curve was 22 μm.

It was also observed that the particle size for the slurry mix results are very close to the dry powder results; thus the Coulter Multisizer II is a better test than the preliminary hydrometer approach.

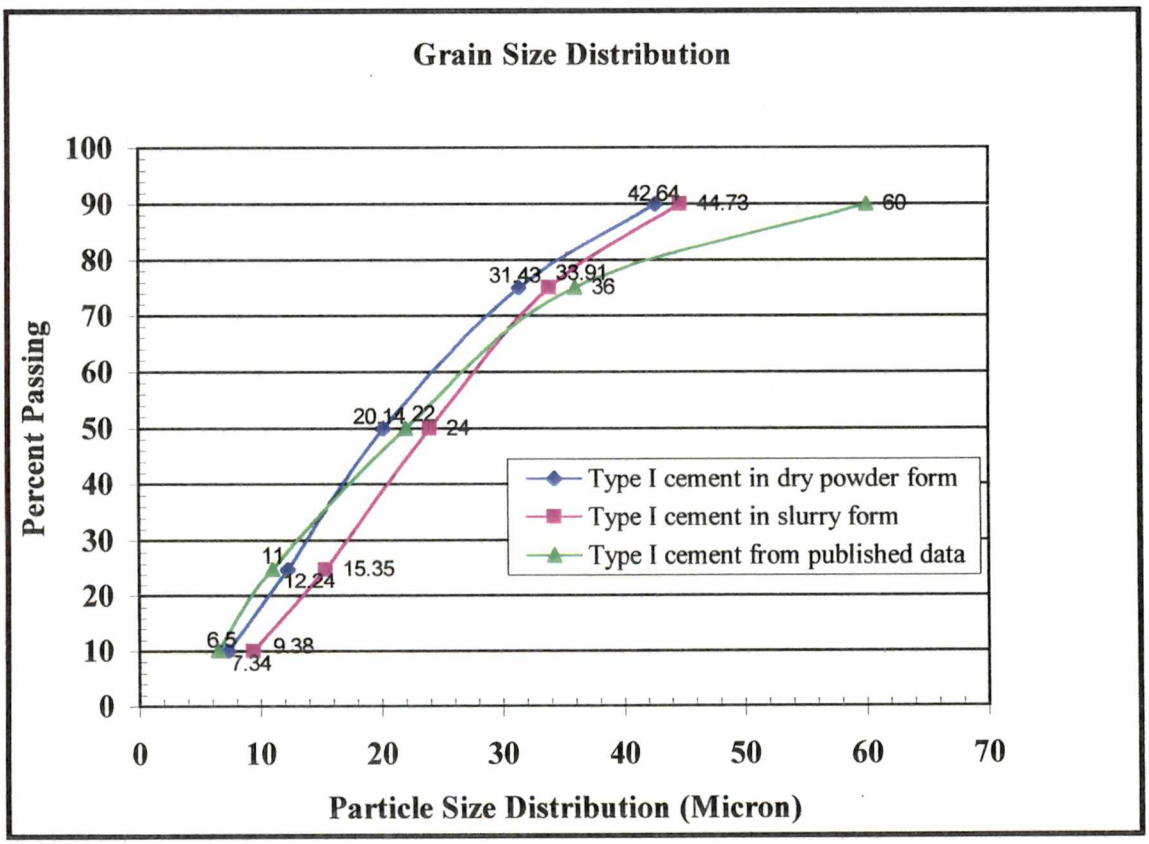


Figure 18. Type I Portland Cement Grain Size Distribution

4.2.3. Results Of Particle Size Distribution Analysis on Microfine Cement.

The particle size of 50 percent results for the Microfine cement in dry powder and slurry forms are summarized in Tables 7 and 8. In addition, the particle size of 50 percent of Microfine cement in dry powder and slurry form was compared with that of the typical gradation curve (Figure 3), and all were plotted and presented in Figure 19. It was noted that the particle size obtained for dry powder sample was about 6 μm, for slurry was about 9 μm, and the particle size of 50 percent of Microfine cement presented in the typical gradation curve was 4 μm.

4.2.4 Results Of Particle Size Distribution Analysis on Type III Portland Cement.

The particle size of 50 percent results for the Type III Portland cement in dry powder and slurry forms are summarized in Tables 9 and 10. In addition, the particle size of 50 percent of Type III, Portland cement in dry powder and slurry form was compared with that of the typical gradation curve (Figure 3), and all were plotted and presented in Figure 20. It was noted that the particle size obtained for dry powder and slurry mix was about 14 μm, and the particle size of 50 percent of this referenced cement type presented in the typical gradation curve was 16 μm.

4.3 Testing of Processed Slurry

4.3.1 Introduction

At this stage of the research the properties of the processed slurry product from the colloidal mill were compared with properties of the slurry before the mill process.

Table 7. Microfine Cement in Dry Powder Form

Cement Particle Size (μm)					
Run#	**% Passing**				
	10%	**25%**	**50%**	**75%**	**90%**
1	2.757	4.379	6.345	8.275	10.38
2	2.858	4.698	6.911	8.7	10.22
3	2.147	3.291	5.378	7.478	9.738
Average (μm)	2.591	4.11	6.21	8.15	10.11

Table 8. Microfine Cement in Slurry Form

Cement Particle Size (μm)					
Run#	**% Passing**				
	10%	**25%**	**50%**	**75%**	**90%**
1	4.454	6.301	8.647	12.04	16.29
2	4.822	6.788	9.696	14.25	19.20
3	4.413	6.303	8.94	13.78	18.38
Average (μm)	4.564	6.464	9.09	13.30	17.96

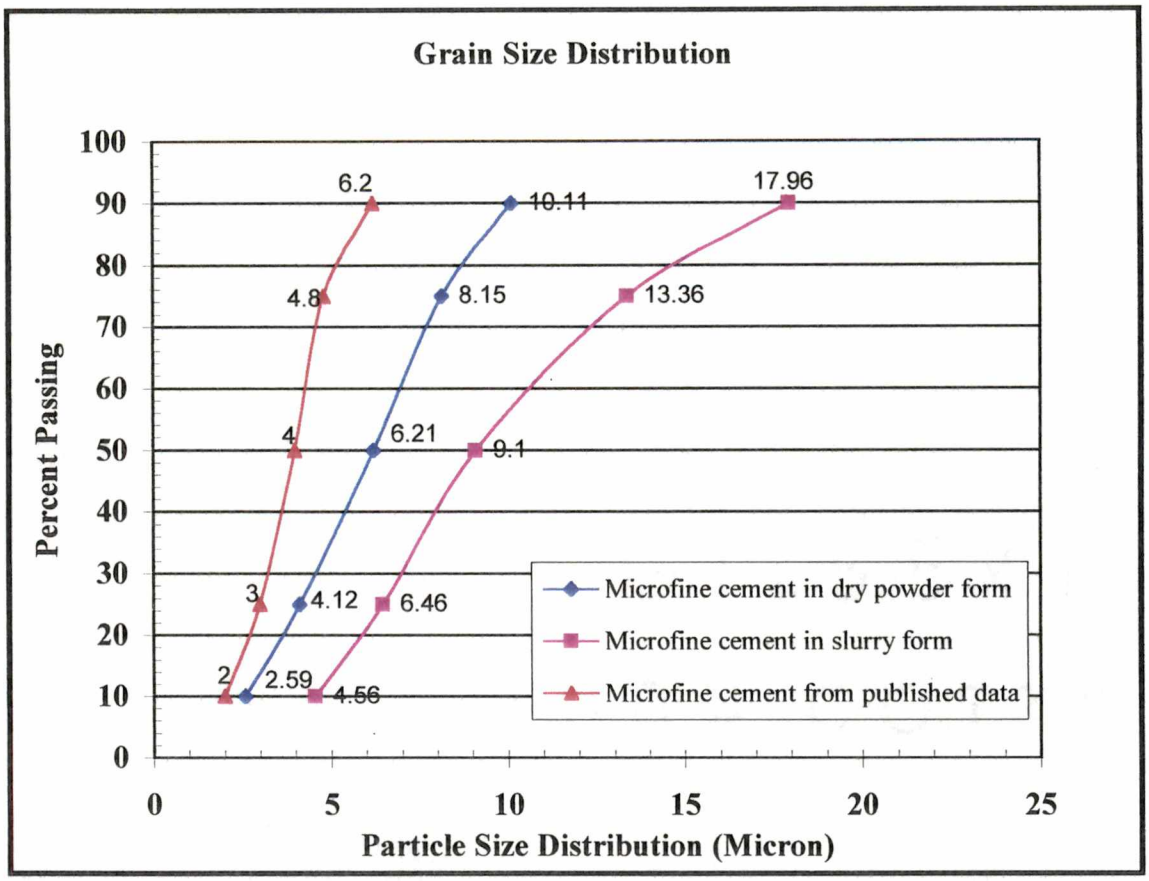


Figure 19. Microfine Cement Grain Size Distribution

Table 9. Type III Portland Cement in Dry Powder Form

Cement Particle Size (μm)					
Run#	**% Passing**				
	10%	**25%**	**50%**	**75%**	**90%**
1	5.739	9.067	14.13	20.43	27.97
2	5.527	9.028	14.35	21.56	29.84
Average (μm)	5.633	9.048	14.240	21.00	28.905

Table 10. Type III Portland Cement in Slurry Form.

Cement Particle Size (μm)					
Run#	% Passing				
	10%	25%	50%	75%	90%
1	6.73	9.83	14.22	19.34	25.19
2	6.62	9.77	14.25	20.18	27.16
3	6.79	10.37	15.43	21.60	28.67
Average (μm)	6.71	9.99	14.63	20.37	27.01

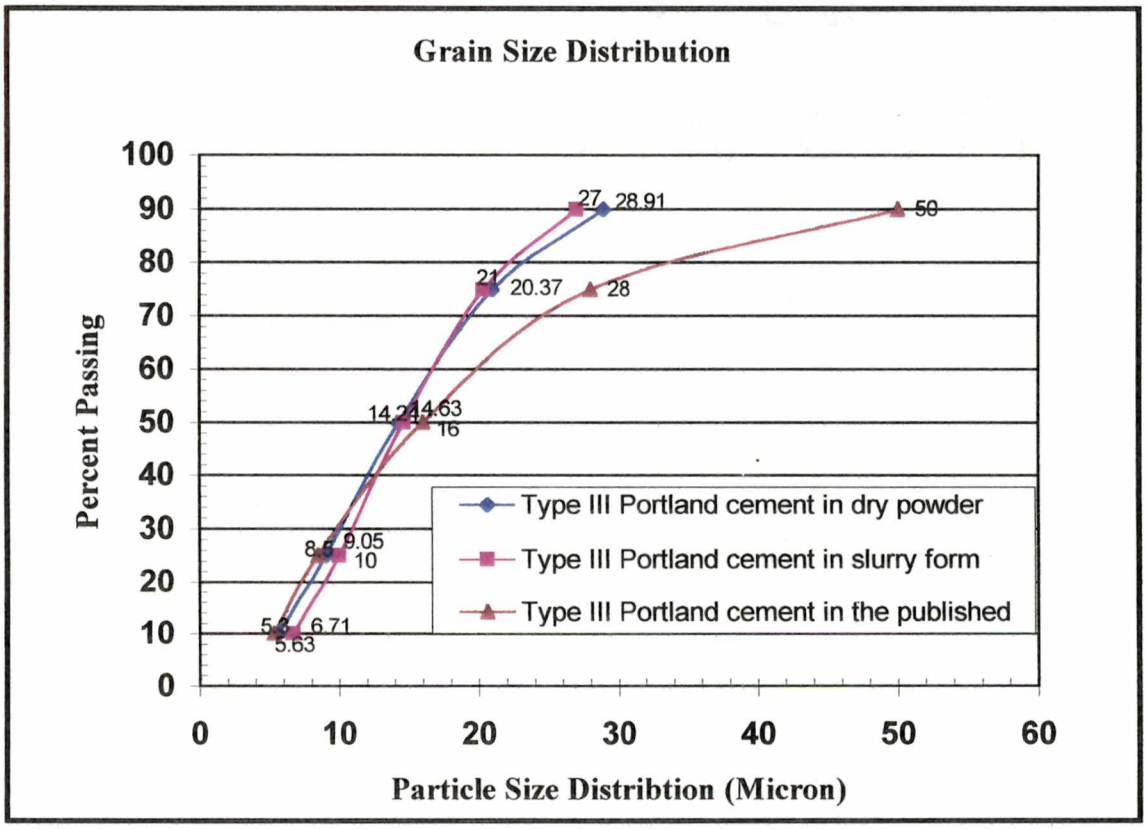


Figure 20. Type III Portland Cement Grain Size Distribution

The following paragraphs describe the test program and the sampling and testing methods used in this evaluation.

4.3.2 Trial Batch Mix

Four of the colloidal mill trial runs were conducted at a water-to-cement ratio = 1.0, with batch weights of 176 lbs. of Type I Portland cement and 176 lbs. of water. One trial run was conducted at a water-to-cement ratio = 0.5, with batch weights of 176 lbs. of type I Portland cement and 88 lbs. of water. All trial mixes were thoroughly mixed with high-speed mixers. Representative slurry samples were obtained before and after processing through the mill. (See Appendix B for Slurry Preparation Protocol)

4.3.3 Sampling

All samples were obtained by passing a glass receptacle (See Figure 21) with volume capacity of 25 cubic inch through the slurry discharge stream. All samples were dosed with 5% of Delvo by cement weight. A photograph of the slurry sampling process is presented in Figure21. (See Appendix B for Slurry Sampling Protocol)

4.3.4 Particle Size Analysis

The Coulter Multisizer II equipment was used for the evaluation of the particle size of the slurry produced by the colloidal mill. The main reason for electing to use the Coulter Multisizer II was that results obtained during base line testing were found to be very reliable. The other factor that is equally important is the speed in obtaining test results as compared to other available methods.



Figure 21. Slurry sampling from the mill

4.3.5 Compressive Strength

Compressive strength can also be related to the size of the cement particles if the water to cement ratio is held constant. In fact, one of the advantages of Microfine cement is the higher early strength achieved relative to Type I Portland cement. With this in mind, compressive strength tests were performed on grout cubes. These grout cubes were molded, stored, cured, and tested in accordance with ASTM C 109. The different grouts were prepared with standard silica sand provided by Rembco Engineering Corporation. Mortar cubes were tested for compressive strength at 1,3,7, and 28 days. Results of the compressive strength testing of grout cubes are provided in Appendix D.

4.3.6 Flow Characteristics

The Marsh Flow Cone test was also performed on samples of slurry to evaluate relative changes in fluid viscosity. The flow of a slurry provides an indication of relative viscosity of the slurry. The viscosity of the slurry can be indirectly related to the size and distribution of the solid particles within the slurry. The standard flow cone test is described by ASTM C 939 "Standard Test Method for Flow of Grout for Preplaced-Aggregate Concrete [Flow Cone Method]". In this study, the Marsh Flow Cone was used instead of the standard flow cone. The sample of grout used with the Marsh Flow Cone was 32 ounces (946mL), instead of the standard amount of 1725mL. The diameter of the discharge tube for the Marsh funnel was 0.250 inches instead of the standard 0.500 inches. A photograph of the Marsh Flow Cone test apparatus is presented in Figure 22.



Figure 22. The Marsh Flow Cone test apparatus

CHAPTER 5.0

LABORATORY TEST RESULTS AND ANALYSIS

5.1 Coulter Multisizer II Test Results (Particle Size Analysis)

The grain size distributions determined by the Coulter Multisizer II for Type I Portland cement for all runs for the non-processed and processed mixes are presented below in Tables 11 and 12, respectively. These results are presented for water-cement ratios of 0.5 and 1.0. Also for the processed mixes, the results are presented for one and two passes through the colloidal mill. At the end of each table, the average passing, the average mean, and the average standard deviation of all the runs are presented. The test data is contained in Appendix C-2 and is summarized below.

Table 11. Results of all Type I Portland Cement Before Mill Process Runs.

				Cement Particle Size (μm)				
Run #	**Batch #**	**Date Tested**	**W/C Ratio**	**% Passing**				
				10%	**25%**	**50%**	**75%**	**90%**
1	1	5/20/99	0.5	6.063	9.346	14.94	21.47	28.39
				5.883	9.039	14.21	20.97	27.56
2	1	5/13/99	1.0	5.446	8.752	14.45	21.8	28.71
3	1	2/17/99	1.0	6.053	9.124	14.02	20.73	28.32
				5.742	9.66	15.64	23.05	31.37
4	1	1/08/99	0.5	6.25	10	15.71	22.62	29.39
Average (μm)				**5.91**	**9.32**	**14.83**	**21.77**	**29.00**
Mean Particle Size (μm)				**16.225**				
Standard Deviation (μm)				**8.76**				

Table 12. Results of all Type I Portland Cement After Mill Process Runs.

Run #	Batch #	No. Of Passes	Date Tested	W/C Ratio	Cement Particle Size (μm)				
					% Passing				
					10%	25%	50%	75%	90%
1	1	1	5/20/99	0.5	6.157	9.523	14.9	22.18	28.93
	1	1			6.166	9.454	15.06	21.97	32.07
2	1	1	5/13/99	1.0	5.458	8.48	13.77	20.56	27.59
	1	1			5.793	8.989	14.61	21.93	28.75
	1	2			6.072	9.406	15.03	22.09	29.36
	1	2			5.761	9.35	15.43	22.98	29.55
3	1	1	2/17/99	1.0	6.995	10.67	16.17	23.12	29.98
	1	1			5.318	9.579	16.06	24.19	32.71
4	1	1	1/08/99	0.5	6.429	10.09	15.83	22.47	29.76
	2	1			6.672	10.59	16.40	23.11	29.83
	2	1			4.661	9.494	15.71	23.50	30.70
	2	1			6.539	10.11	15.42	22.11	28.64
	2	2			6.489	10.21	15.93	22.55	29.41
5	1	1	8/26/98	1.0	5.997	9.041	13.77	19.63	25.61
					6.100	9.606	15.01	21.65	27.49
					5.653	9.543	15.74	23.29	30.61
Average (μm)					**6.02**	**9.63**	**15.3**	**22.33**	**29.44**
Mean Particle Size (μm)					**16.59**				
Standard Deviation (μm)					**8.85**				

The results of non-processed and processed slurries for trial run number 1 using w/c ratio of 0.5 are plotted in Figure 23. As can be seen from Figure 23 there was no reduction in particle size resulting from the processing through the Quintics collodial mill.

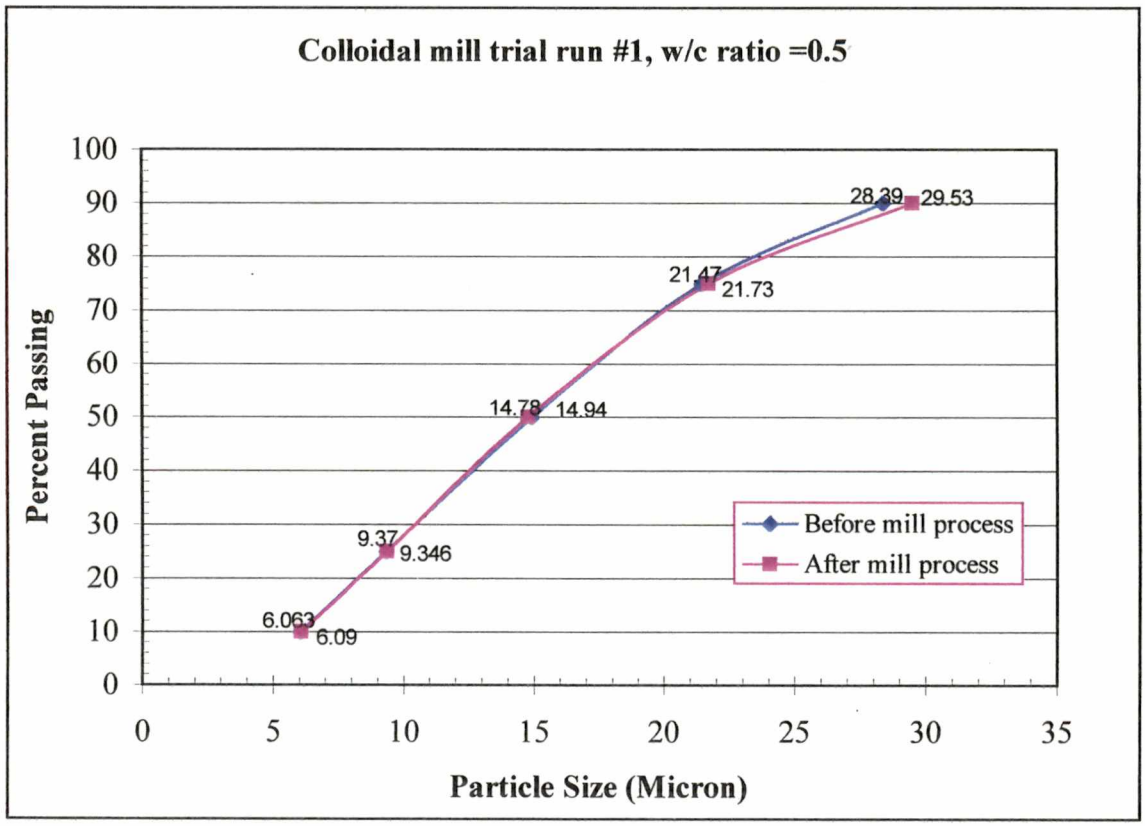


Figure 23. The Results of Non-Processed and Processed Slurries for Trial Run Number 1.

The results of non-processed, and processed slurries that contains one and two passes through the colloidal mill for trial run number 2 using water to cement ratio of 1.0 were presented and plotted in Figure 24. As can be seen from Figure 24 there was no reduction in particle size resulting from the processing through the Quintics collodial

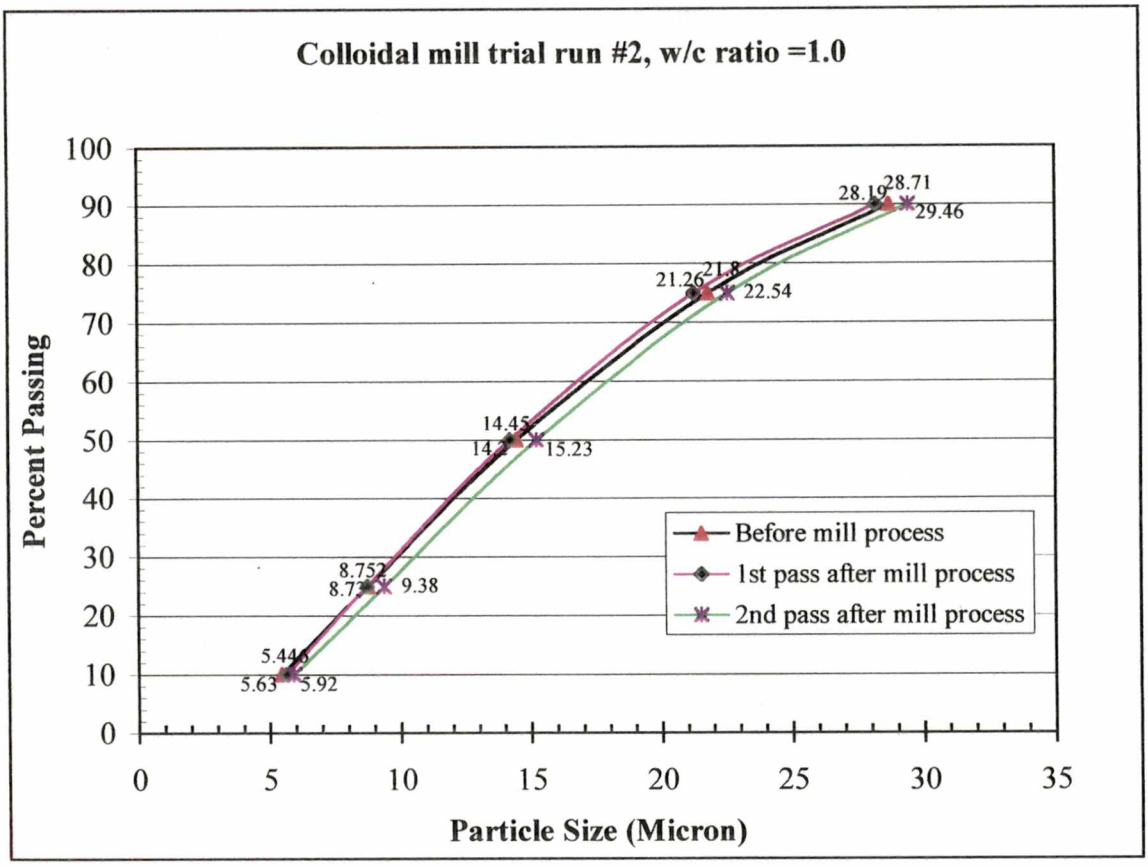


Figure 24. The Results of Non-Processed and Processed Slurries for Trial Run Number 2.

mill. It was also observed that passing the same slurry mix two times through the colloidal mill had no success in reducing the particle size.

The results of non-processed and processed slurries that represented one and two passes through the colloidal mill for trial run number 3 using water to cement ratio of 1.0 are presented and plotted in Figure 25. As can be seen from Figure 25 there was no reduction in particle size resulting from the processing through the Quintics collodial mill.

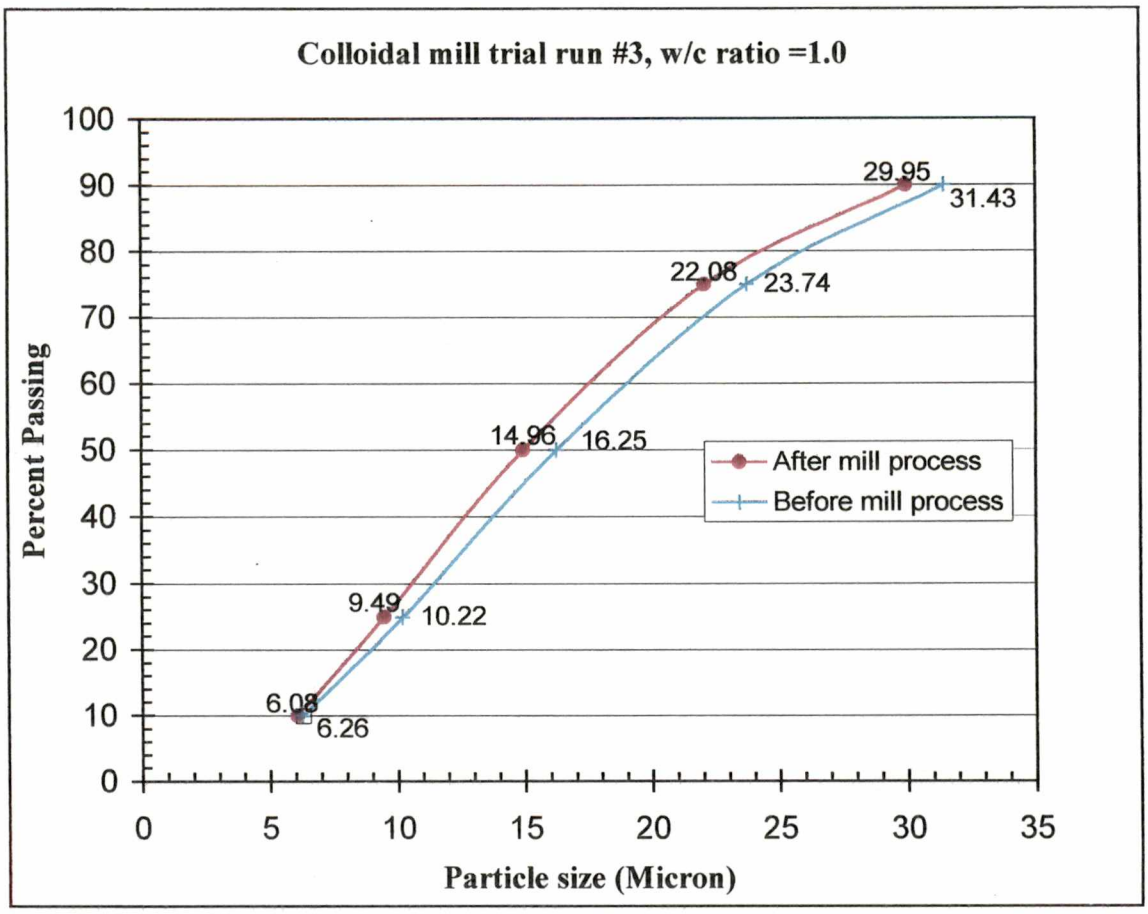


Figure 25. The Results of Non-Processed and Processed Slurries for Trial Run Number 3.

Trial run number 4 had two batches using water to cement ratio of 0.5. The first batch results contain non-processed, and one pass through the Quintics colloidal mill. The second batch results contain non-processed, and one and two passes through the Quintics colloidal mill. All results of this trial run are plotted and presented in Figure 26. As can be seen from Figure 26 there was no reduction in particle size resulting from the processing through the Quintics collodial mill. It was also observed in the second batch that passing the same slurry mix two times through the colloidal mill had no effect in reducing the particle size.

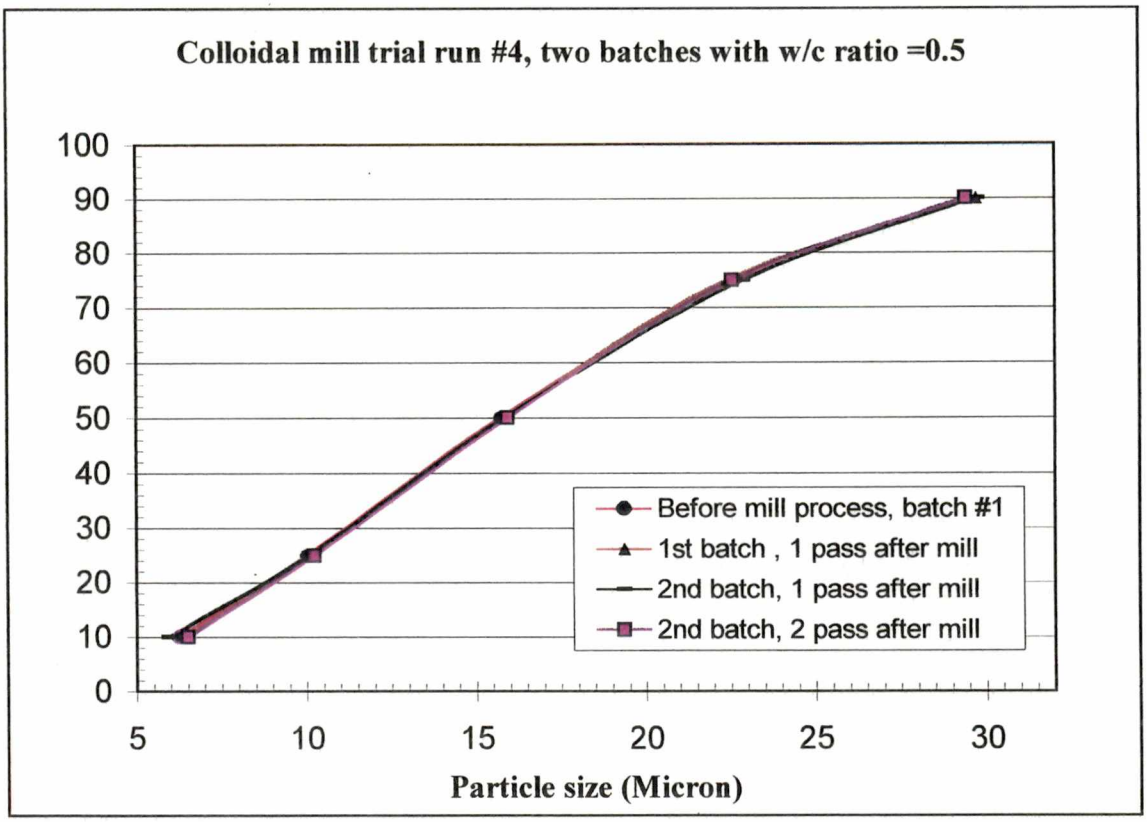


Figure 26. The Results of Non-Processed and Processed Slurries for Trial Run Number 4.

The results of trial run number 5 using water to cement ratio of 1.0 are presented in Table 12 only which is for the processed slurry because there was no testing for the non-process slurry. This trial run was mainly made to test the equipment used to perform these trial runs.

The average results of Tables 11 and 12 were plotted and presented in Figure 27. Clearly, and as can be seen from Figure 27, there was no reduction in particle size resulting from the processing through the Quintics collodial mill.

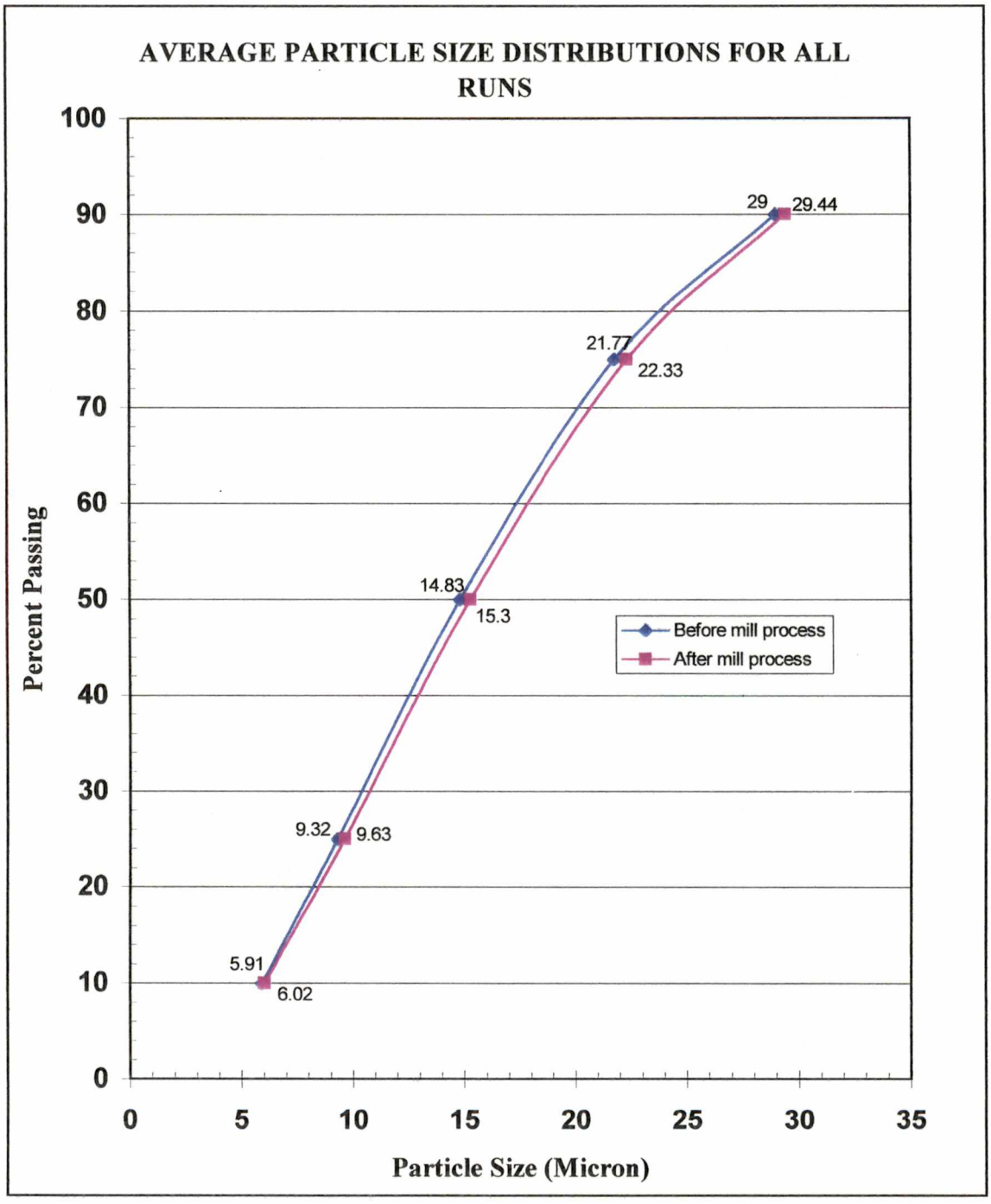


Figure 27. The Results of Non-Processed and Processed Slurries for All Trial Runs.

5.2 Flow and Viscosity Test Results

The results from the Marsh flow cone for process runs 1 (water-cement ratio =0.5) and 2 (water cement ratio =1.0) are also presented in figures 28 and 29. The results show that in the case of run number 1, there was a significant reduction in viscosity after one pass. The difference in viscosity between runs 1 and 2 reflects the difference in water-to-cement ratio between the two slurries (0.5 versus 1.0, respectively). The results of these runs indicate that run number 1 has a relatively more dispersed structure.

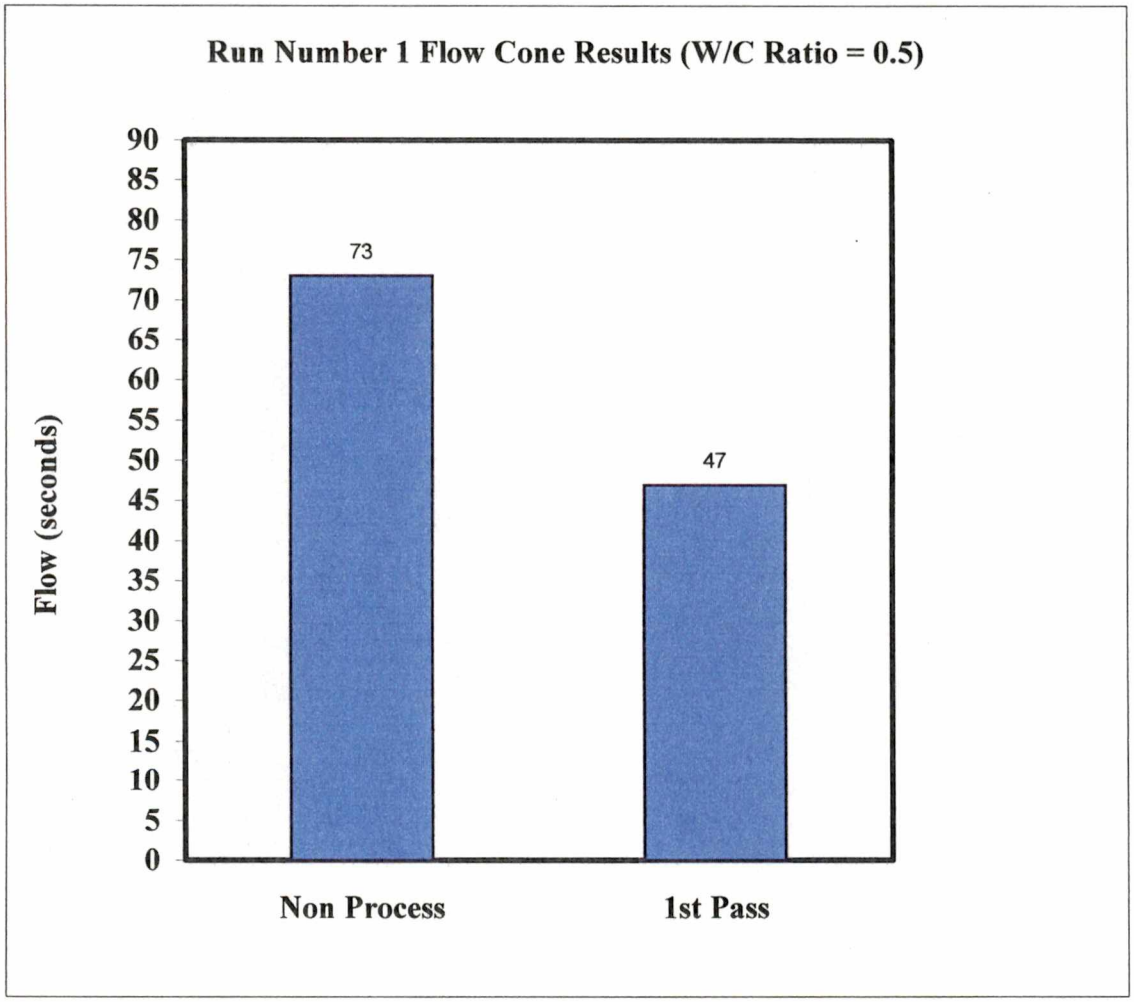


Figure 28. Flow Cone Results for Run Number 1

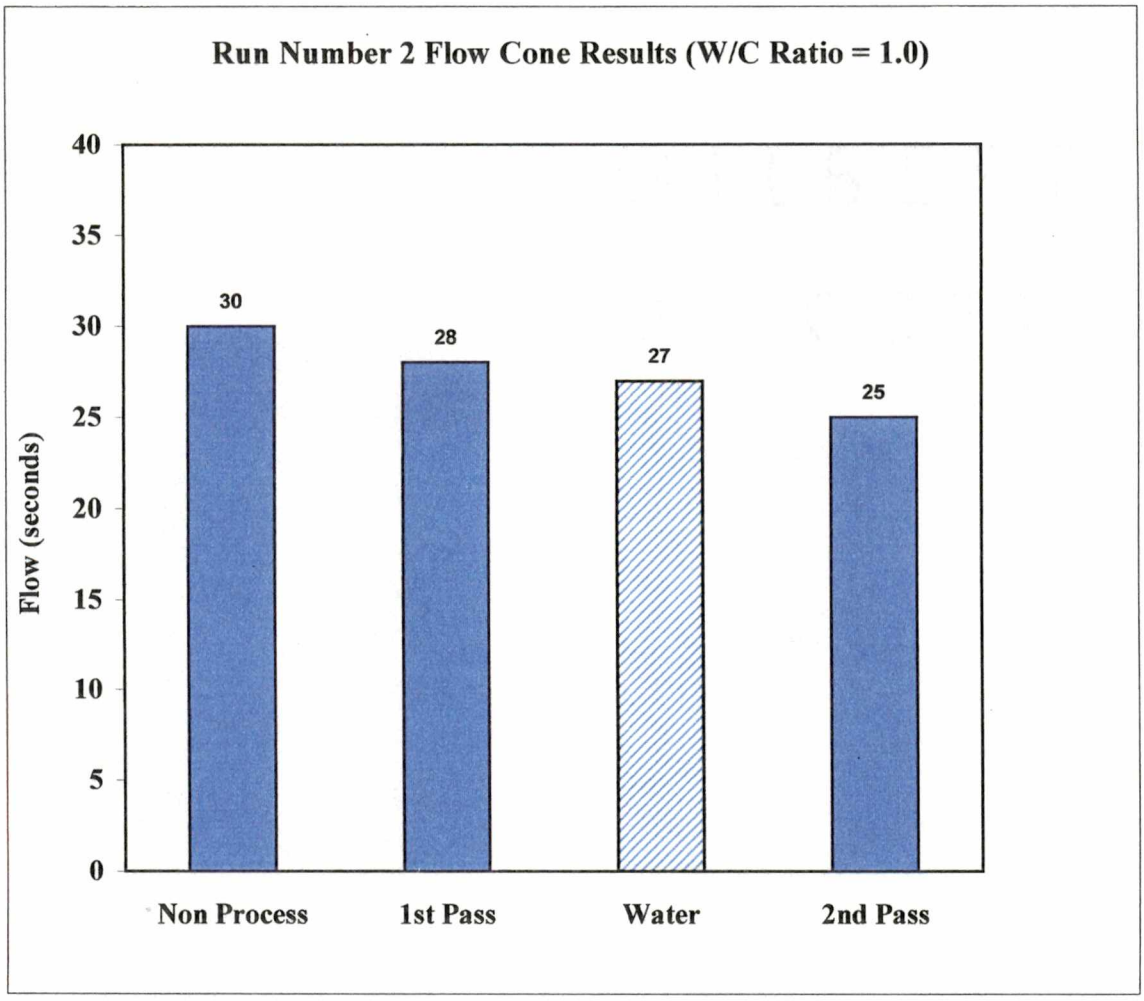


Figure 29. Flow Cone Results for Run Number 2

The results for run number 2 show that there was no dramatic decrease in viscosity with increased number of passes through the mill. This is believed to be a strong indication that particle size reduction has not occurred. The slight decrease in viscosity is attributed to dispersion. This is in agreement with the published literature as was discussed in chapter 2, which suggest that dispersion rather than particle size reduction was the main factor in obtaining such results.

5.3 Compressive Strength Test Results

Compressive strength test results are presented in Appendix D. The test results are summarized in Figures 30 and 31. The results presented in Figure 30 illustrate that there was no observed increase in compressive strength for the Type I Portland cement after passing through the Quinetics Colloidal Mill. In fact, these results indicate that the compressive strength cubes fabricated from the processed Type I Portland cement slurry remained the same or decreased relative to the cubes fabricated from the non-processed slurry. The results presented in Figure 31 also illustrate that there was no observed increase in the compressive strength for the Type I Portland cement after passing through the Quinetics Colloidal Mill, but to the contrary, a decrease in the compressive strength was observed.

It was expected that there would be a relative increase in the compressive strength in the processed Type I cement as a result of particle size reduction. The non-improvement in the compressive strength is a significant observation in that it suggests that no particle size reduction has occurred to the processed Type I slurry. Thus, it is suspected that the preliminary observations from the hydrometer test analysis resulted from improved particle dispersion rather than particle size reduction.

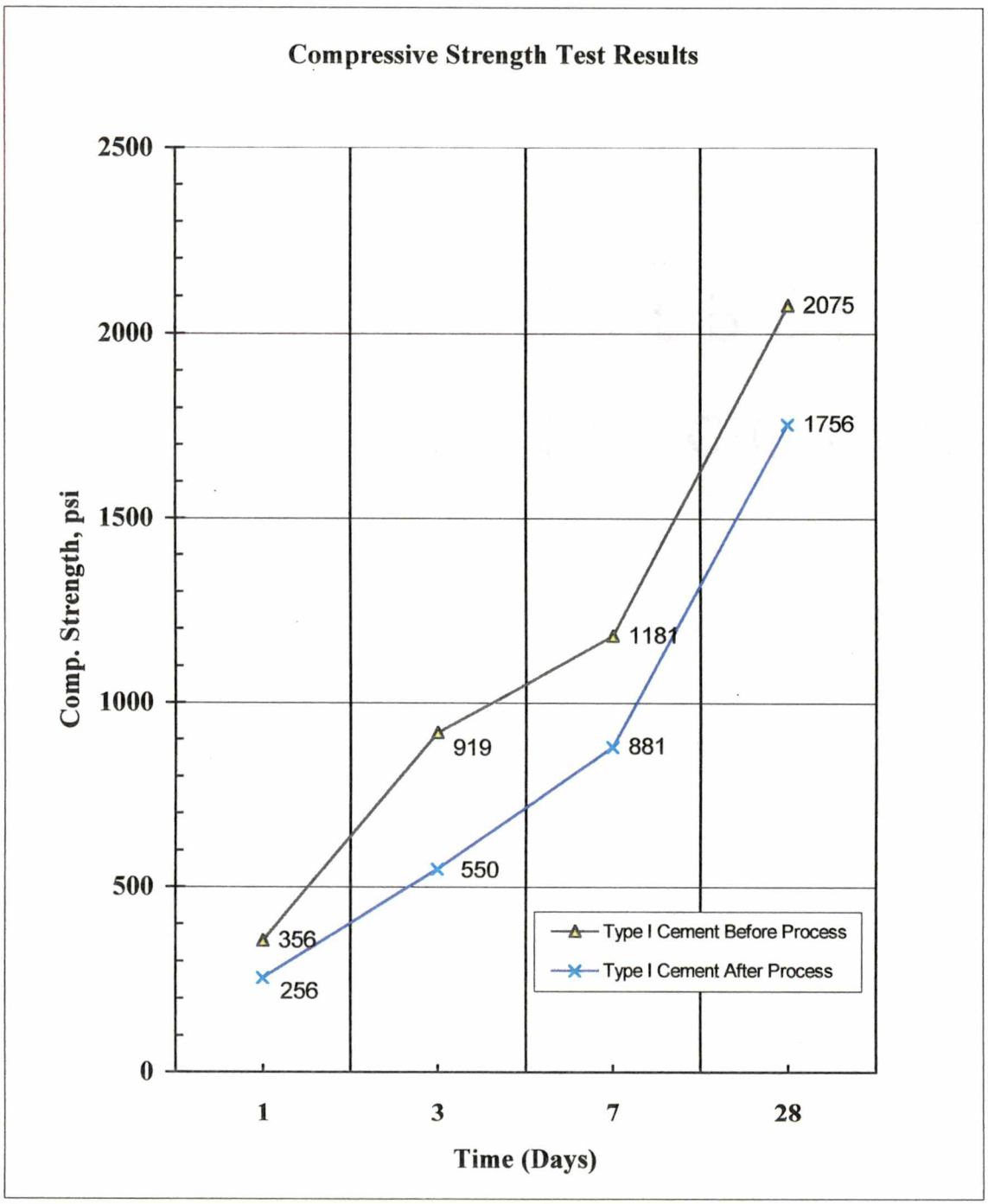


Figure 30. Compressive Strength Test Results For Trial Run Number 3 (W/C =1.0)

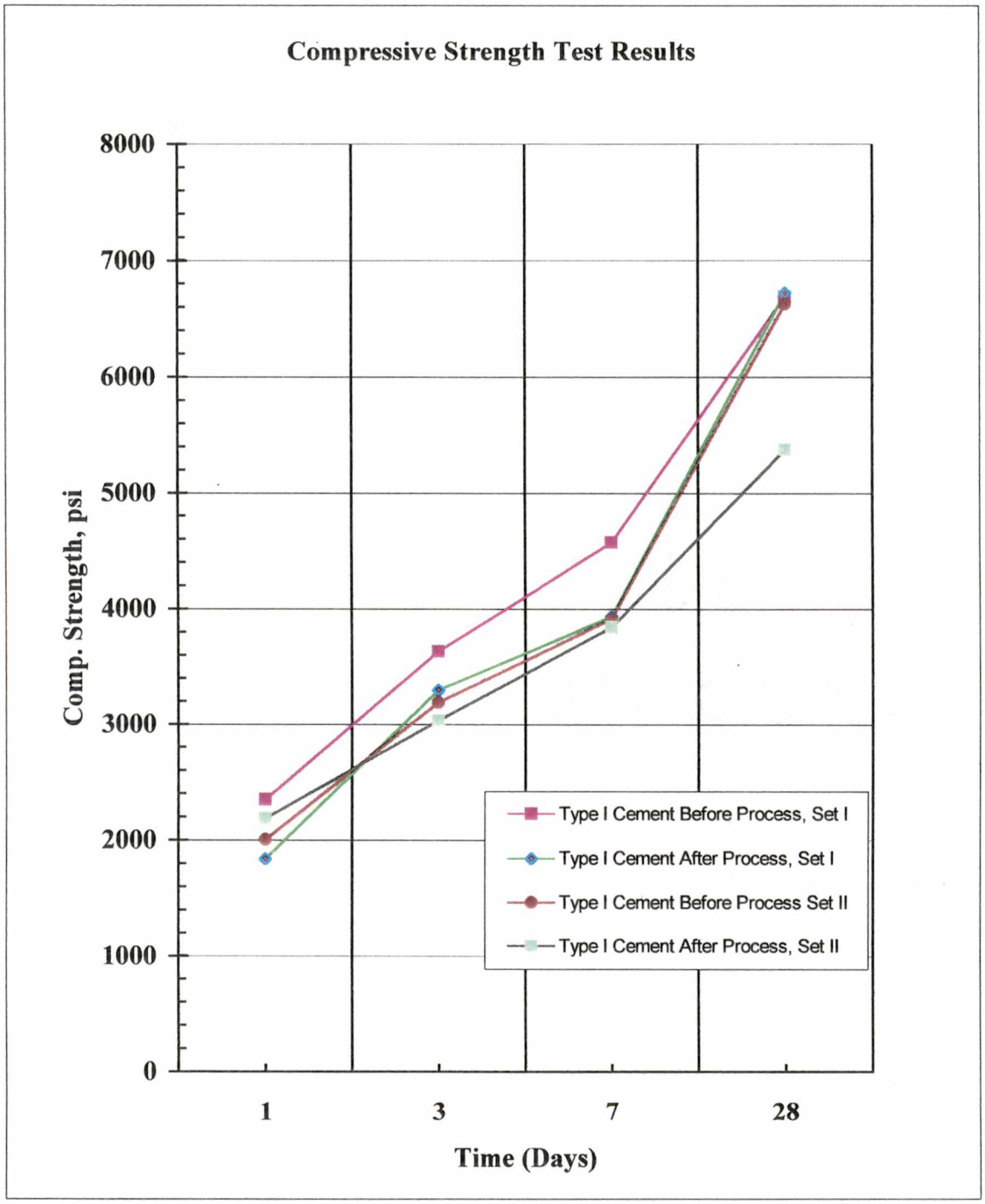


Figure 31. Compressive Strength Test Results For Trial Run Number 4 (W/C =0.5)

CHAPTER 6.0

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

Microfine cement is used to promote high early strength and improved pumping characteristics in grouting and soil stabilization applications. A proprietary colloidal mill device, developed by Quinetics of San Antonio, Texas, was proposed for the manufacture of Microfine cement from typical Type I Portland cement.

Type I Portland cement slurry was processed through the Quinetics colloidal mill. Samples of the processed slurry were tested to evaluate relative changes in cement particle size. The tests performed on the slurry samples include the particle size distribution analysis by hydrometer and Coulter Multisizer II, the Marsh flow cone test, and mortar cube compressive strength testing.

The results of the preliminary hydrometer tests indicated that some reduction in particle size was occurring, as presented in Figure 16 of Chapter 3. However, upon further testing with more advanced techniques, it was found that the apparent reduction in particle size observed with the hydrometer test was the result of dispersion characteristics.

The results of the flow cone tests exhibit a slight reduction in viscosity in one run and relatively more reduction in viscosity in another run. This is indicative of improved dispersion of the cement particles in slurry solution.

The results of mortar cube compressive strength test results exhibit that there was no observed increase in strength for the slurry that was processed through the mill versus

that which was not. This also supports the premise that the preliminary observations are more likely the result of dispersion rather than particle size reduction.

It is concluded from this study that the colloidal mill was unsuccessful in producing a cement with similar characteristics to Microfine cement.

6.2 Recommendations

The following items are recommended for further evaluation:

- Practical limit of the Quinetics colloidal mill on fine particle size.
- An overall evaluation of limits and benefits of using the Quinetics colloidal mill should be conducted.
- Evaluation of benefits on pumpability.
- Evaluation of the effectiveness of this process on different materials other than cement slurry.

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REFERENCES

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APPENDICES

APPENDIX A

EQUIPMENT OPERATION PROTOCOL

Coulter Multisizer II Operating Steps

I. Computer

1- Type win at the prompt to activate windows.

2- Multi (double click)

3- Multi.exe (double click)

4- O.K.

5- Preferences

6- file preferences (make your own)

7- Acquire

A- New Sample

A- Group ID?

B- Sample ID?

C- Operator?

D- Comments?

E- Electro? Iston (or whatever solution used)

F- Dispersment? Type IB

G- Electro Volume? (ml)

H- Sample Volume?

I- Sample Mass? (ml)

Hit O.K.

- Acquire

- Collect from multi-seizer

II. Coulter Multisizer II

A- Power

B- Setup Menu

1- Date could be changed by using menu arrows

2- Orifice Dia? 70 mm (depend on what is used.)

3- Orifice length? depending on orifice diameter.

4- Setup? Auto Manual

5- Analysis? Sample Blank (First time use blank)

6- Calibration? Recall (all the time)

- Push Cal

1- Kd? Automatically will change after pushing Cal button.

2- Size? Automatically will change after pushing Cal button.

3- Size units? m

- Setup button

1- Current and gain? Manual (leave at all time)

2- Aperture Current? 1600 ma

A- Gain? 2

B- Polarity? +

3- Instrument

A- Control: Time

Time: 130

Channel: 5000

Total: 500+-2

- Setup button again (don't change anything)

- Setup

Communication setup (don't change anything)

- Setup button

* Note: once finished 1st blank for background, change analysis to sample.

- Then hit full button setup

Full

Narrow

III. Sampling Stand II.

- Isoton in 100 ml glass and pot (fill to the mark)

- We have two pots at the reservoir, one marked with yellow mark (waste pot), should be empty before run test, and the 2nd pot (reservoir should be filled up to mark 250 ml).

Check List Before Beginning Test

1- Check 250 ml of clean electr. on the clean reservoir

Check 250 ml of clean electr. on the beaker stand.

empty on the waste reservoir bottle.

2- Check the starter control to OFF

Reset/Count to COUNT

Fill/Close to CLOSE

3- Press the power switch ON

Begin Testing

* Note: All buttons must be turned clockwise

1- Time mode

1- Focus on the tube.

2- Set the 'Manometer select' switch to the out position.

3- Set 'Reset/count' control to reset position.

4- Set fill/ close to fill.

5- Adjust stirrer controller around 2.5

6- Power

7- Get two drops or more in the waste pot and close Fill/ Close to close

8- Adjust valve to control Mercury to be below starting bulb. Allow 1 min. for the mercury to set.

9- Push start button on the Coulter Multisizer it takes time set at 'Analysis setup-1'

10- Push Print Button

11- For sample test go to Acquire again.

12- Collect from multi-seizer.

13- Reset (coulter multi II)

14- Setup

15- Change Analysis to: Sample

16- Hit full for screen.

17- But sample in glass

18- Hit start on Coulter Multi-seizer II.

19- Hit print on Coulter Multi-seizer II.

20- Go to Analyze on the computer screen

A- Subtract background.

B- Choose Blank file.

C- Hit open

21- View

- Listing

- Print

22- Save File after subtracting.

Shutdown Procedure

1- Set "Reset/ Count" to "count"

"Fill/ Close" to "close"

2- Reset & Turn the sample stand II off

3- Release the vacuum from the waste bottle by lifting

the vacuum from the waste bottle cap.

4- Rotate Vacuum controller anticlockwise all the way.

5- Set reset/ count to reset

6- Set fill/ closed to fill

7- Turn on again

8- Turn it off again

9- Take beaker and aperture tube out.

10- Release the vacuum by lifting vacuum release valve on the waste bottle cap

11- Lower the beaker platform and remove the beaker.

Moyno Pump and Colloidal Mill Operation Protocol

(See Figure A-1)

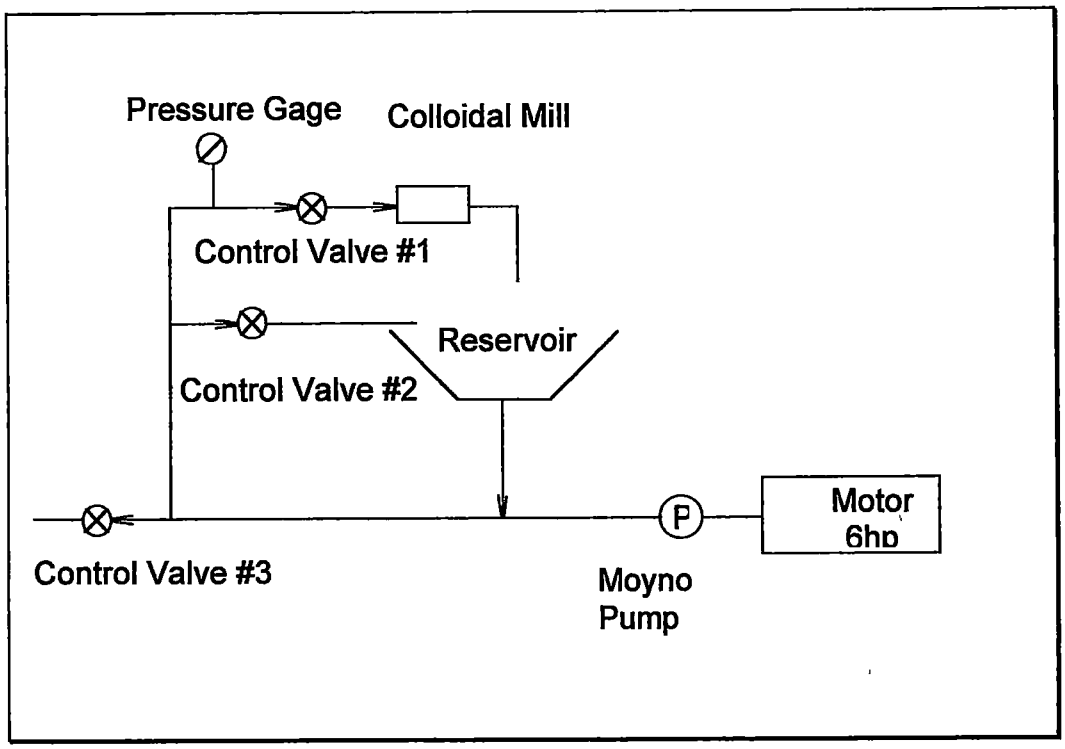


Figure A-1. Schematic of Slurry Circulation Process.

1) Open valve 1.

2) Close valve 2.

3) Close valve 3.

4) Disengage pump clutch.

5) Start pump motor (note: use only in a well ventilated area).

6) Adjust the throttle.

7) Prepare test slurry (see slurry preparation protocol).

8) Place slurry in pump hopper.

9) Engage pump clutch.

10) Allow slurry to circulate for 1 minute.

11) Open valve 2.

12) Close valve 1 partially (to provide 40-70 psi to the mill).

13) Allow slurry to pass through colloidal mill for prescribed number of passes or time period.

14) Sample slurry from discharge stream from colloidal mill for designated testing (see sampling and testing protocols).

15) Disengage pump clutch.

APPENDIX B

TESTING METHODS PROTOCOLS

Modified Hydrometer Testing Protocol

1. Obtain the following equipment: two 1000 ml sedimentation cylinders (one serves as a control cylinder and the other serves as the testing cylinder); one hydrometer bulb (provide graph for converting hydrometer reading to length in cm); one thermometer (Celsius preferred); and one stopwatch.
2. Prepare the control cylinder by mixing 50 ml of Delvo, which serves as the dispersing agent, with distilled water to the calibration mark. Mix this solution and place the hydrometer bulb in the control cylinder to obtain a dispersing agent correction. Assume a meniscus correction of 0.5.
3. Obtain enough slurry sample to ensure approximately 50g of cement is tested. This is based on the water-cement ratio of 0.50. Place the quantity of slurry and 50 ml of Delvo in the test cylinder with enough distilled water to reach the calibration mark. Mix this solution by hand for 1 minute.
4. Slowly insert the hydrometer bulb into the test cylinder. Obtain hydrometer readings at intervals of 30 seconds, 1, 2, 5, 10, 15, 20, 25 minutes, etc., until the difference between readings becomes imperceptible.

Marsh Flow Cone Testing Protocol

1. Obtain slurry sample using 32 oz (946 mL) sampling container.
2. Pour sample contents into the Marsh Funnel.
3. Measure time in which it takes slurry to completely flow out of Marsh Funnel.
4. Record time on data sheet.

Slurry Preparation Protocol (for 0.50 water-cement ratio)

1) Place 30.6 pounds of Type I Portland Cement in a dry 5-gallon container.

2) Combine 15.3 pounds of water and 660mL of Delvo (which constitutes a 5% Delvo content by weight of cement) in a separate container.

3) Slowly add the cement to the water/Delvo mixture while blending with a high-speed mixer.

4) Blend slurry with the high-speed mixer for 1 minute, or until no dry cement remains in the slurry.

5) Place slurry in the pump hopper (see schematic on Pump and Colloidal Mill Protocol, Figure A-1).

6) Allow slurry to re-circulate in the reservoir for 1 minute prior to sampling or diverting to colloidal mill.

Slurry Sampling Protocol

1) Pass sampling container horizontally through the discharge stream from the colloidal mill.
2) Obtain approximately 75-grams of slurry for hydrometer testing.
3) Obtain 32oz (946mL) for flow cone testing.
4) Obtain a volume capacity of 25 cubic inch for coulter Multisizer II testing.
5) Obtain approximately 393-mL (per set of three cubes) for grout cube mold.

Note: All samples must be obtained from the discharge stream from the colloidal mill. Continuous mixing of test samples must be provided until testing is initiated.

APPENDIX C

COULTER MULTISIZER II TEST RESULTS

APPENDIX C-1

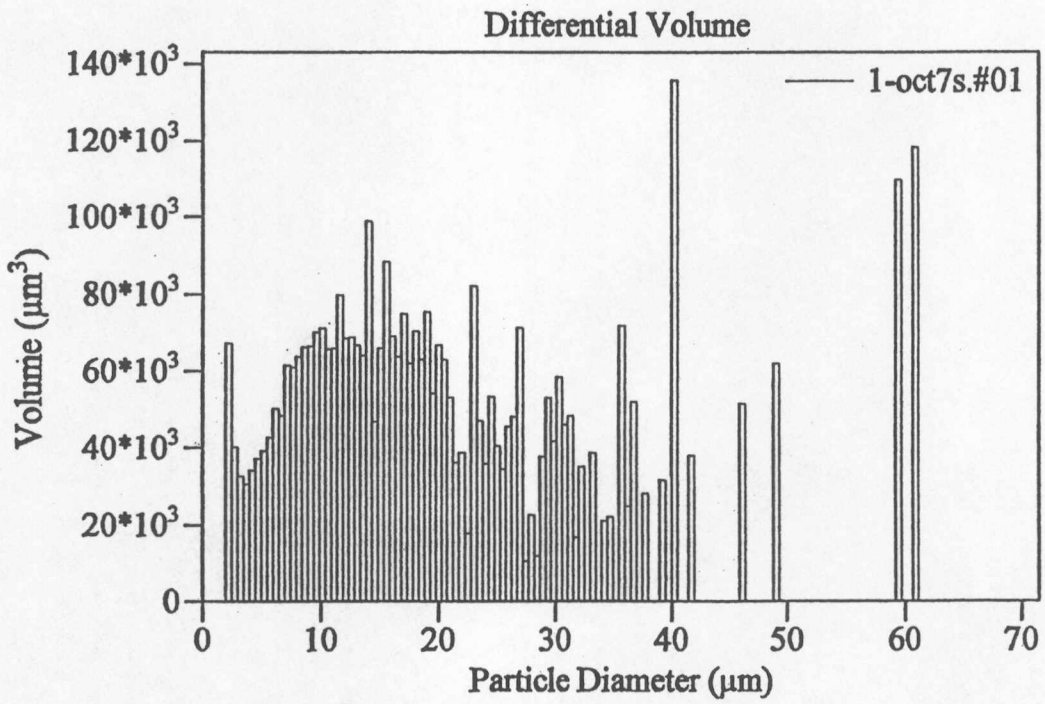
BASE-LINE TEST DATA

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	1-oct7s.#01	Sample Number:	1
Group ID:	blank		
Sample ID:	oct7b-#01		
Comment:	Type I		
Operator:	nha		
Raw Count:	70,533	Coinc. Corr. Count:	79,895
Acquired:	20:09 10 Jun 1997		



Volume Statistics (Arithmetic) 1-oct7s.#01

Calculations from 1.972 µm to 63.11 µm

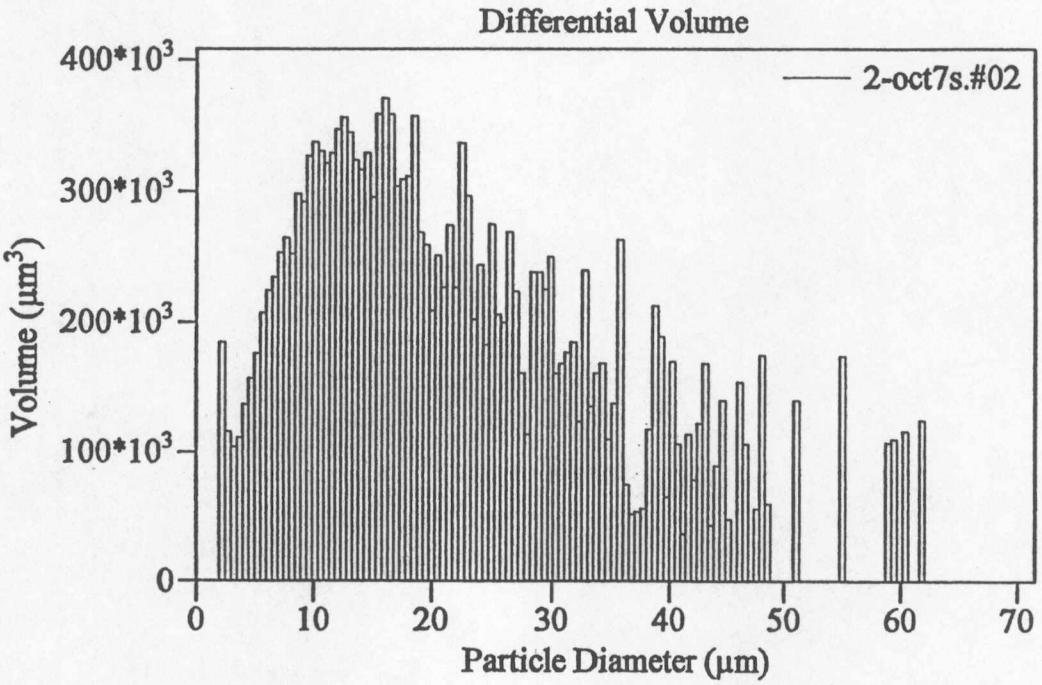
Volume:	4.113e6 µm³		
Mean:	22.06 µm	95% Conf. Limits:	21.88-22.24 µm
Median:	18.68 µm	S.D.:	14.32 µm
Mean/Median Ratio:	1.181		

% <	10	25	50	75	90
µm	6.788	11.43	18.68	29.85	40.31

COULTER AccuComp® 2.01

20 Sep 1999

File name:	2-oct7s.#02	Sample Number:	1
Group ID:	S-B		
Sample ID:	oct7-#02		
Comment:	TYPE I		
Operator:	NHA		
Raw Count:	164,619	Coinc. Corr. Count:	1
Acquired:	20:40 10 Jun 1997		



Volume Statistics (Arithmetic) 2-oct7s.#02

Calculations from 1.972 µm to 63.11 µm

Volume:	20.01e6 µm³		
Mean:	22.70 µm	95% Conf. Limits:	22.60-22.79 µm
Median:	20.03 µm	S.D.:	13.31 µm
Mean/Median Ratio:	1.133		

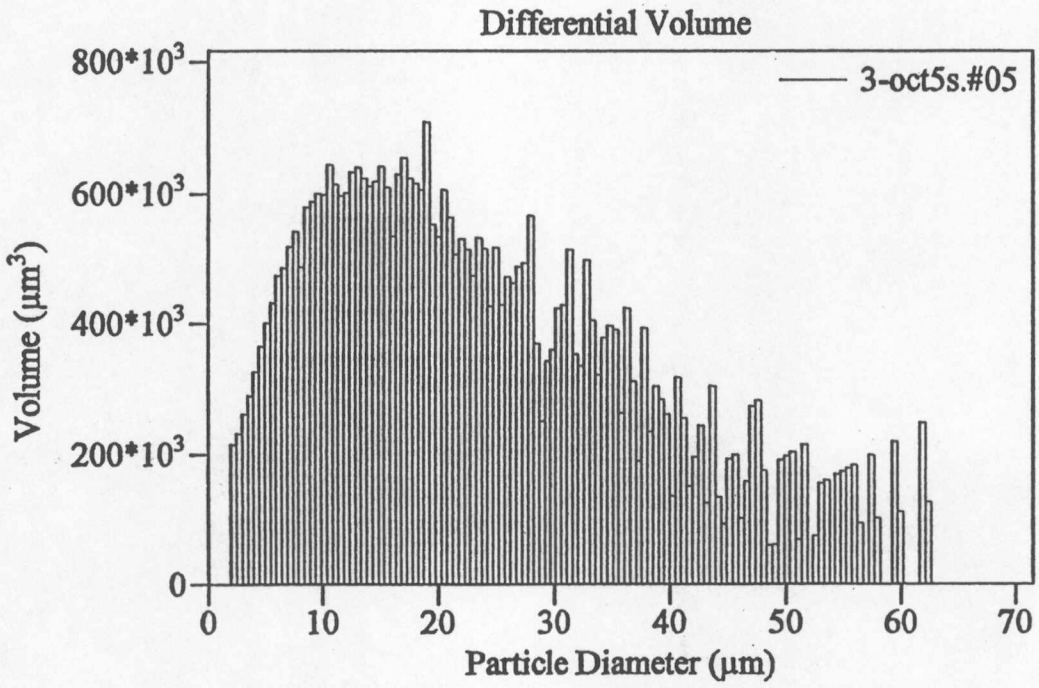
% <	10	25	50	75	90
µm	7.578	12.32	20.03	30.93	41.97

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	3-oct5s.#05	Sample Number:	1
Group ID:	Blank		
Sample ID:	oct5b-#5		
Comment:	Type-I		
Operator:	NHA		
Raw Count:	178,267	Coinc. Corr. Count:	1
Acquired:	18:56 10 Jun 1997		



Volume Statistics (Arithmetic) 3-oct5s.#05

Calculations from 1.972 µm to 63.11 µm

Volume:	42.78e6 µm³		
Mean:	24.32 µm	95% Conf. Limits:	24.25-24.40 µm
Median:	21.70 µm	S.D.:	14.22 µm
Mean/Median Ratio:	1.121		

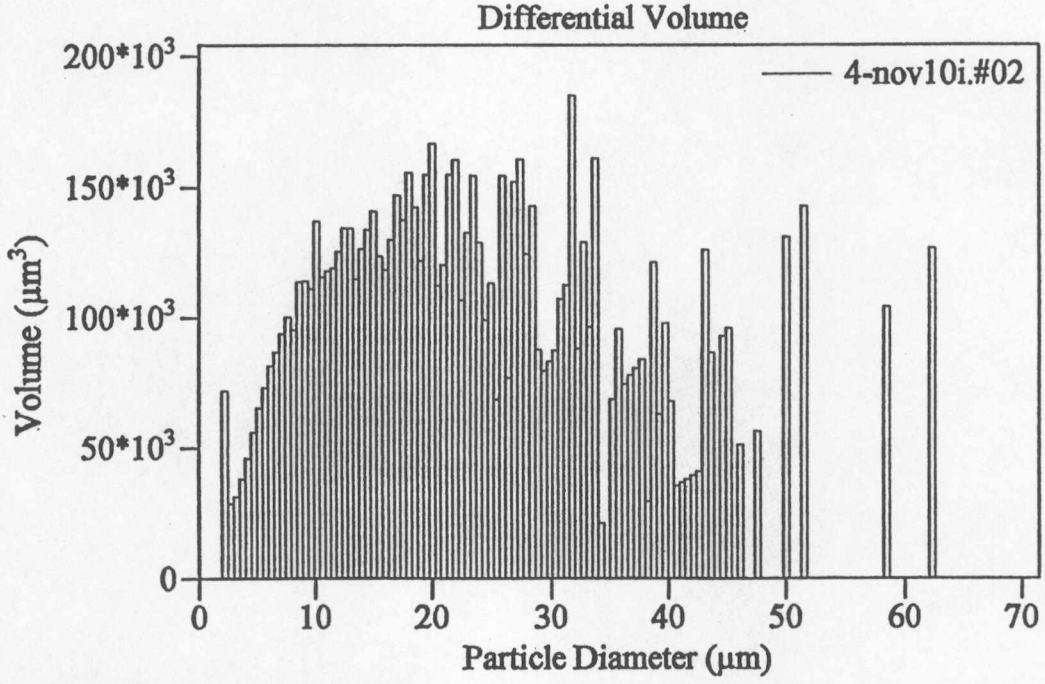
% <	10	25	50	75	90
µm	7.646	12.97	21.70	33.52	45.63

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	4-nov10i.#02	Sample Number:	1
Group ID:	Type I slurry		
Sample ID:	nov10.#02		
Comment:	with delvo		
Operator:	NHA		
Raw Count:	99,240	Coinc. Corr. Count:	1
Acquired:	21:13 11 Jun 1997		



Volume Statistics (Arithmetic) 4-nov10i.#02

Calculations from 1.972 µm to 63.11 µm

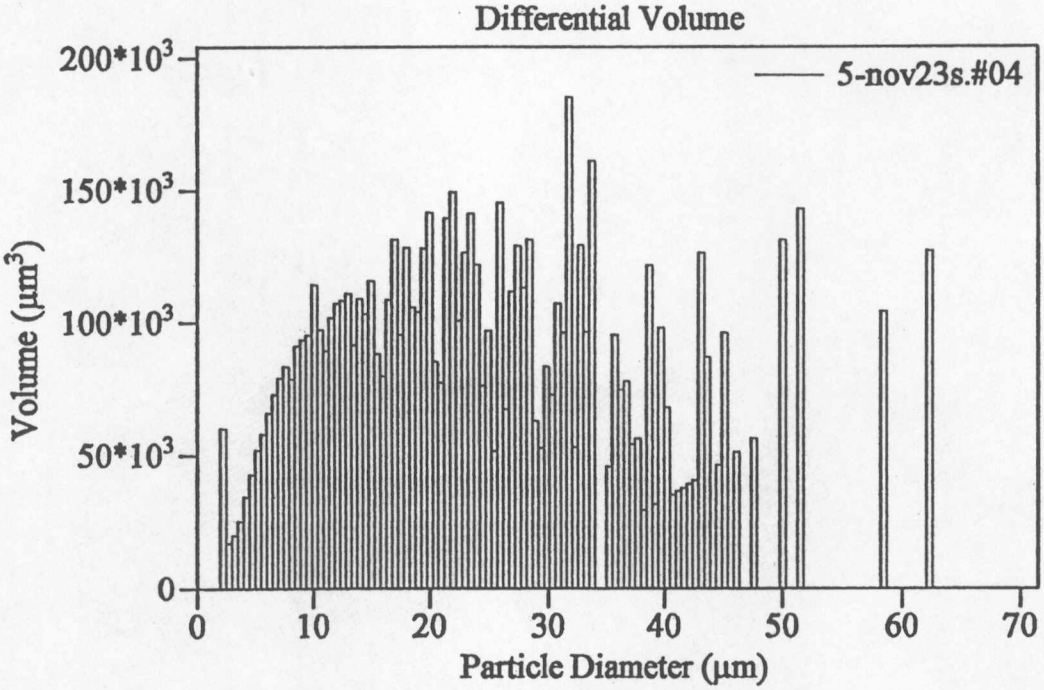
Volume:	9.477e6 µm^3		
Mean:	24.68 µm	95% Conf. Limits:	24.53-24.84 µm
Median:	22.97 µm	S.D.:	13.17 µm
Mean/Median Ratio:	1.074		

% <	10	25	50	75	90
µm	8.727	14.43	22.97	32.95	43.16

COULTER AccuComp® 2.01

20 Sep 1999

File name:	5-nov23s.#04	Sample Number:	1
Group ID:	Type I slurry		
Sample ID:	nov23.#04		
Comment:	with delvo		
Operator:	NHA		
Raw Count:	99,240	Coinc. Corr. Count:	1
Acquired:	21:13 11 Jun 1997		
Background Run:	NOV23ISB.#04		



Volume Statistics (Arithmetic) 5-nov23s.#04

Calculations from 1.972 µm to 63.11 µm

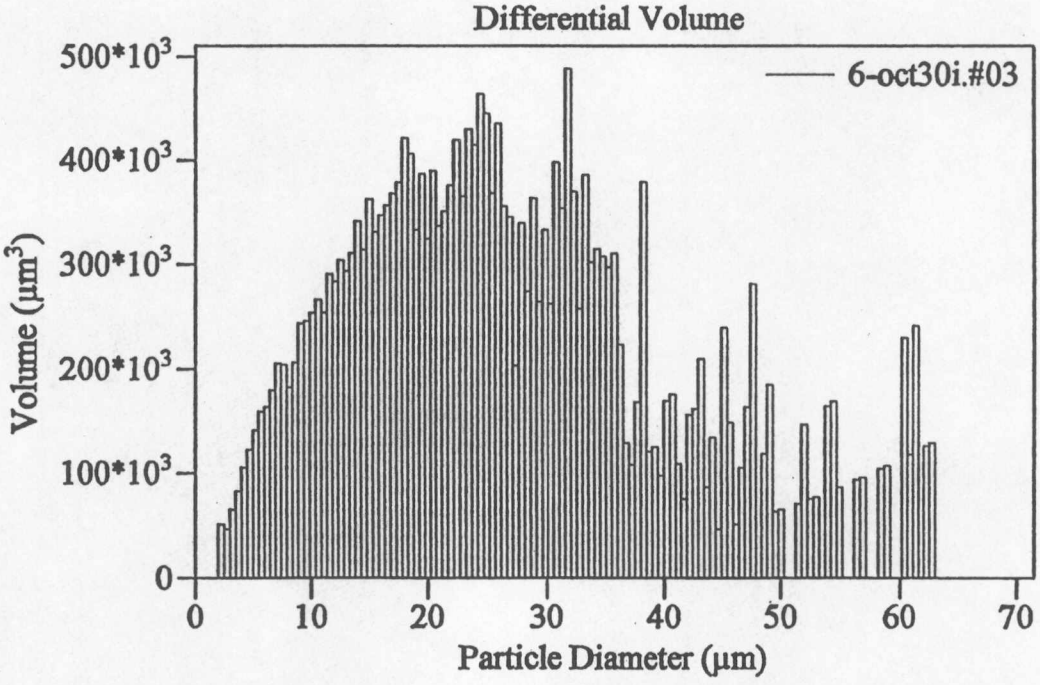
Volume:	8.106e6 µm³		
Mean:	25.46 µm	95% Conf. Limits:	25.30-25.62 µm
Median:	23.68 µm	S.D.:	13.42 µm
Mean/Median Ratio:	1.075		

% <	10	25	50	75	90
µm	9.035	14.89	23.68	33.67	43.57

COULTER AccuComp® 2.01

20 Sep 1999

File name:	6-oct30i.#03	Sample Number:	1
Group ID:	Type I slurry		
Sample ID:	oct30-#03		
Comment:	delvo added in glass		
Operator:	nha		
Raw Count:	91,448	Coinc. Corr. Count:	108,110
Acquired:	9:13 11 Jun 1997		



Volume Statistics (Arithmetic) 6-oct30i.#03

Calculations from 1.972 µm to 63.11 µm

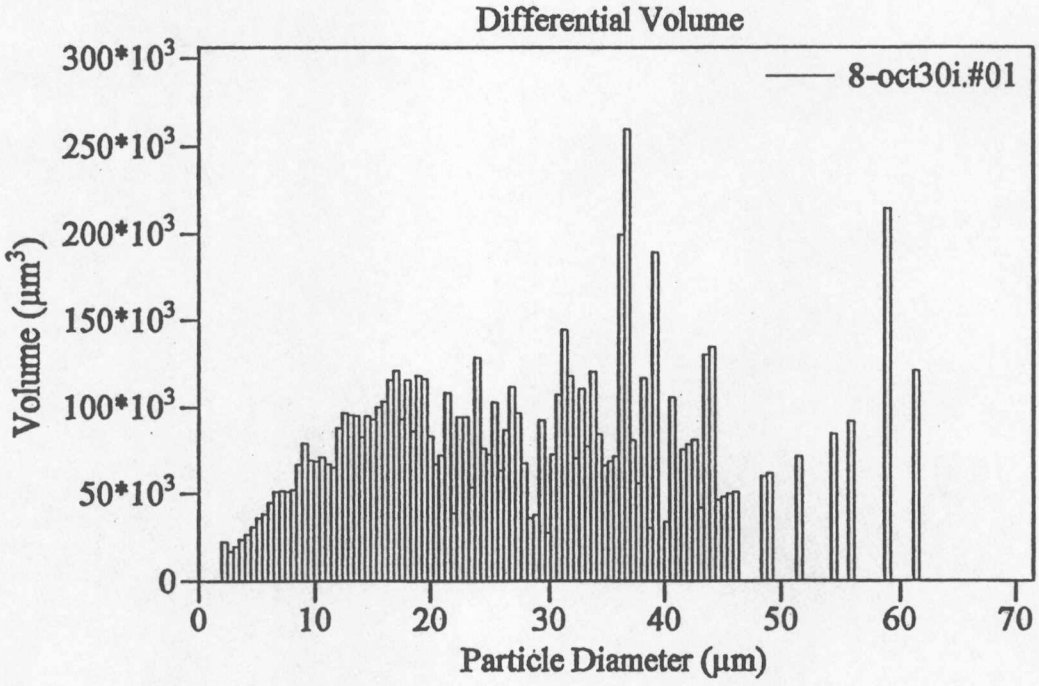
Volume:	26.85e6 µm³		
Mean:	27.22 µm	95% Conf. Limits:	27.09-27.36 µm
Median:	25.25 µm	S.D.:	13.95 µm
Mean/Median Ratio:	1.078		

% <	10	25	50	75	90
µm	10.39	16.74	25.25	35.10	47.47

COULTER AccuComp® 2.01

20 Sep 1999

File name:	8-oct30i.#01	Sample Number:	1
Group ID:	type i slurry		
Sample ID:	oct30-#01		
Comment:	slurry with delovo after mix in glass		
Operator:	nha		
Raw Count:	35,896	Coinc. Corr. Count:	38,120
Acquired:	8:22 11 Jun 1997		



Volume Statistics (Arithmetic) 8-oct30i.#01

Calculations from 1.972 µm to 63.11 µm

Volume:	7.814e6 µm³		
Mean:	28.41 µm	95% Conf. Limits:	28.17-28.65 µm
Median:	27.53 µm	S.D.:	14.05 µm
Mean/Median Ratio:	1.032		

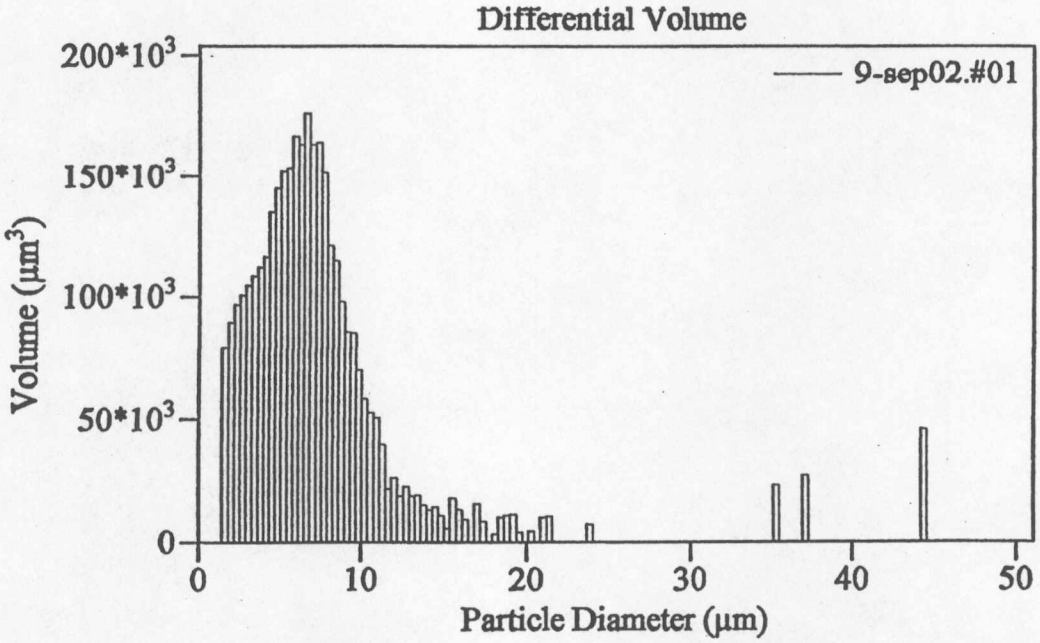
% <	10	25	50	75	90
µm	10.57	16.90	27.53	37.67	45.61

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	9-sep02.#01	Sample Number:	1
Group ID:	sep02		
Sample ID:	sample1		
Comment:	Microfine dry cement		
Operator:	nabeel		
Acquired:	0:42 9 Jun 1997		



Volume Statistics (Arithmetic) 9-sep02.#01

Calculations from 1.436 µm to 15.43 µm

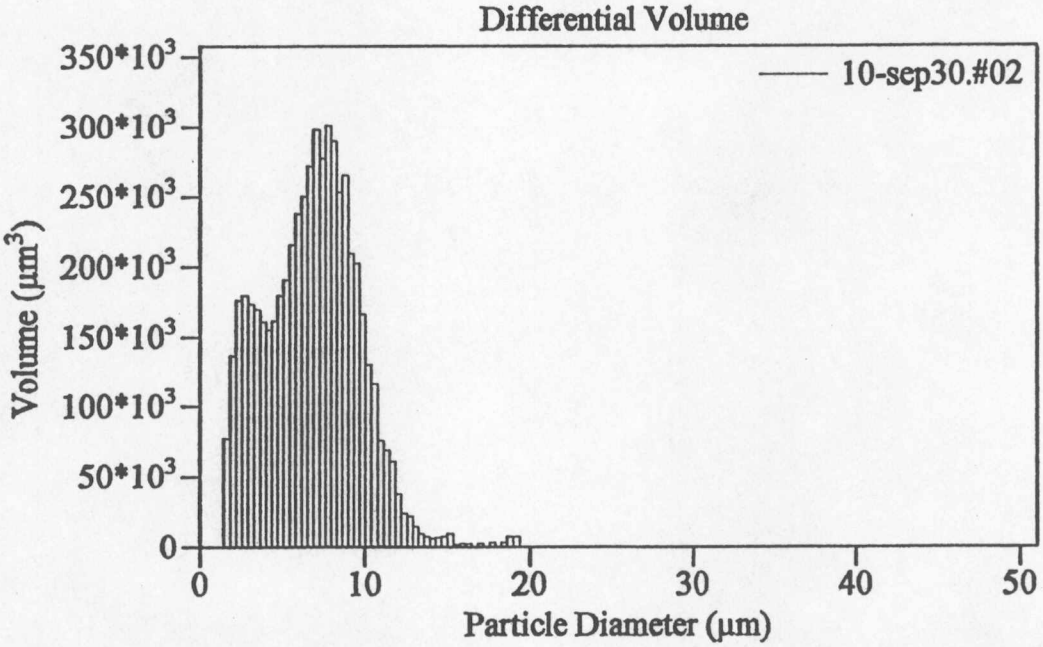
Volume:	3.342e6 µm³		
Mean:	6.509 µm	95% Conf. Limits:	6.492-6.526 µm
Median:	6.345 µm	S.D.:	2.886 µm
Mean/Median Ratio:	1.026		

% <	10	25	50	75	90
µm	2.757	4.379	6.345	8.275	10.38

COULTER AccuComp® 2.01

20 Sep 1999

File name:	10-sep30.#02	Sample Number:	1
Group ID:	blank		
Sample ID:	sep30-02		
Comment:	microfine dry cement		
Operator:	nha		
Raw Count:	216,838	Coinc. Corr. Count:	245,666
Acquired:	13:59 10 Jun 1997		



Volume Statistics (Arithmetic) 10-sep30.#02

Calculations from 1.436 µm to 34.10 µm

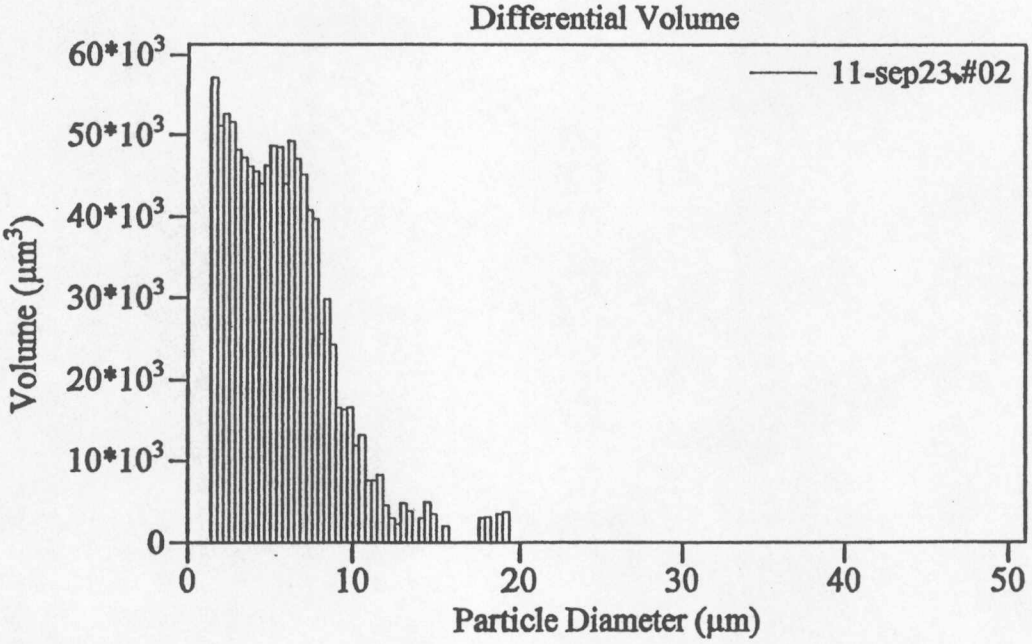
Volume:	5.621e6 µm³		
Mean:	6.799 µm	95% Conf. Limits:	6.785-6.812 µm
Median:	6.911 µm	S.D.:	2.828 µm
Mean/Median Ratio:	0.984		

% <	10	25	50	75	90
µm	2.856	4.698	6.911	8.698	10.22

COULTER AccuComp® 2.01

20 Sep 1999

File name:	11-sep23.#02	Sample Number:	1
Group ID:	sep23		
Sample ID:	sample2		
Comment:	microfine		
Operator:	nha		
Raw Count:	134,706	Coinc. Corr. Count:	145,108
Acquired:	2:59 10 Jun 1997		



Volume Statistics (Arithmetic) 11-sep23.#02

Calculations from 0 µm to 45.94 µm

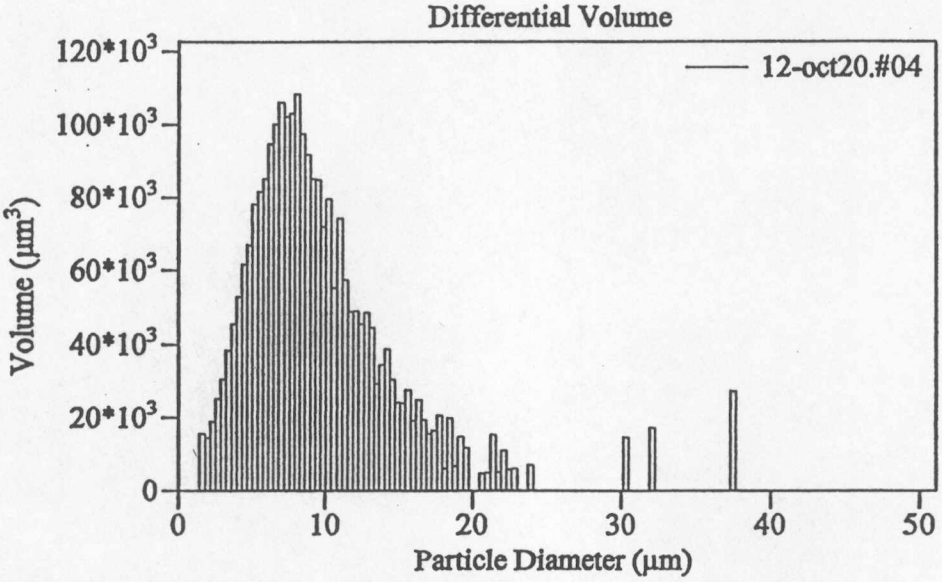
Volume:	1.075e6 µm³		
Mean:	5.780 µm	95% Conf. Limits:	5.755-5.805 µm
Median:	5.378 µm	S.D.:	3.186 µm
Mean/Median Ratio:	1.075		

% <	10	25	50	75	90
µm	2.147	3.291	5.378	7.478	9.738

COULTER AccuComp® 2.01

20 Sep 1999

File name:	12-oct20.#04	Sample Number:	1
Group ID:	Microfine Slurry		
Sample ID:	Oct20-#04		
Comment:	Microfine slurry with 5% Delvo		
Operator:	NHA		
Raw Count:	68,160	Coinc. Corr. Count:	70,691
Acquired:	6:25 11 Jun 1997		



Volume Statistics (Arithmetic) 12-oct20.#04

Calculations from 1.436 µm to 45.94 µm

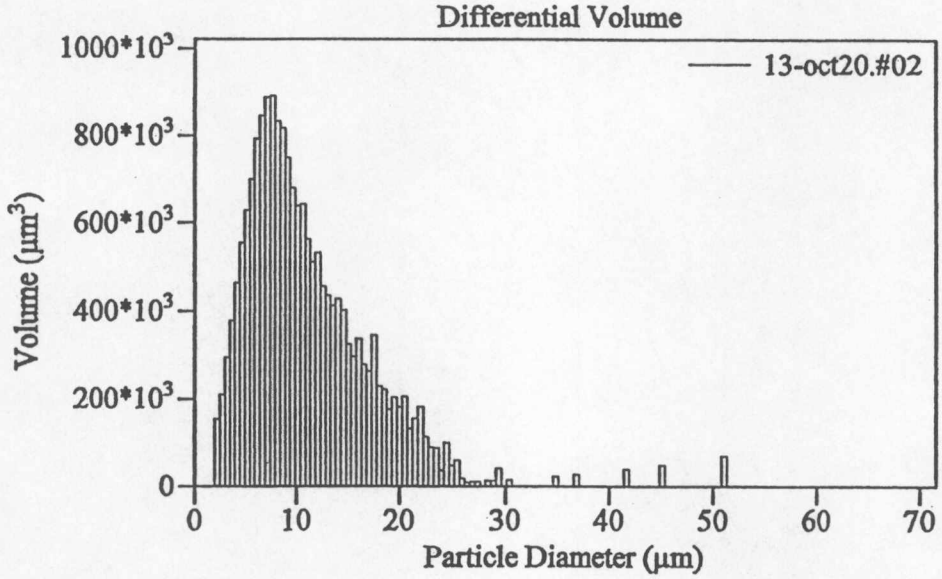
Volume:	2.672e6 µm³		
Mean:	9.878 µm	95% Conf. Limits:	9.817-9.939 µm
Median:	8.647 µm	S.D.:	5.655 µm
Mean/Median Ratio:	1.142		

% <	10	25	50	75	90
µm	4.454	6.301	8.647	12.04	16.29

COULTER AccuComp® 2.01

20 Sep 1999

File name:	13-oct20.#02	Sample Number:	1
Group ID:	Microfine Slurry		
Sample ID:	oct20-#02		
Comment:	with 5% Delvo		
Operator:	NHA		
Raw Count:	204,088	Coinc. Corr. Count:	1
Acquired:	5:40 11 Jun 1997		



Volume Statistics (Arithmetic) 13-oct20.#02

Calculations from 1.972 µm to 63.11 µm

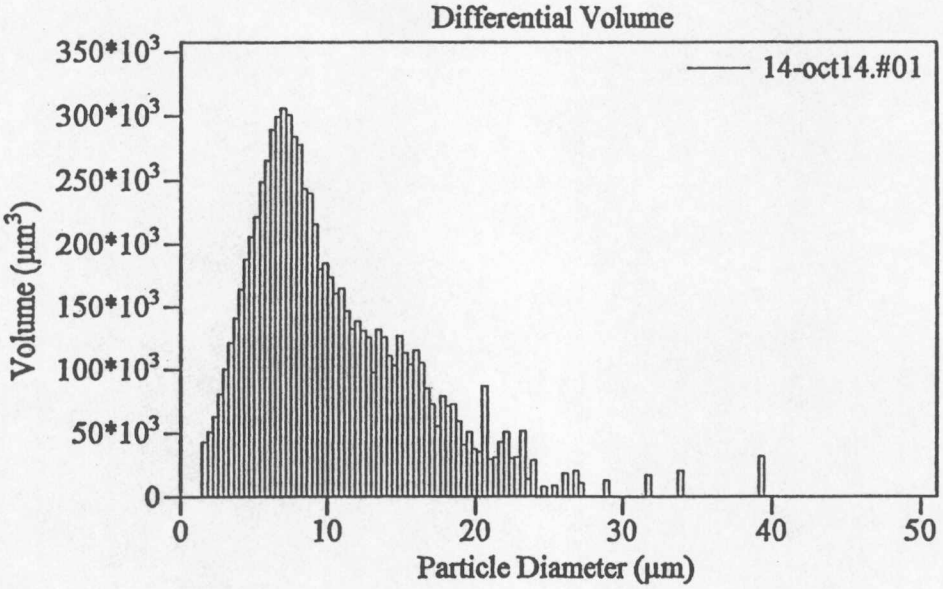
Volume:	19.31e6 µm³		
Mean:	11.16 µm	95% Conf. Limits:	11.13-11.19 µm
Median:	9.693 µm	S.D.:	6.364 µm
Mean/Median Ratio:	1.151		

% <	10	25	50	75	90
µm	4.822	6.788	9.693	14.27	19.21

COULTER AccuComp® 2.01

20 Sep 1999

File name:	14-oct14.#01	Sample Number:	1
Group ID:	Slurry		
Sample ID:	oct14-01		
Comment:	Slurry microfine with 5% Delvo		
Operator:	NHA		
Raw Count:	133,592	Coinc. Corr. Count:	143,813
Acquired:	0:28 11 Jun 1997		



Volume Statistics (Arithmetic) 14-oct14.#01

Calculations from 1.436 µm to 45.94 µm

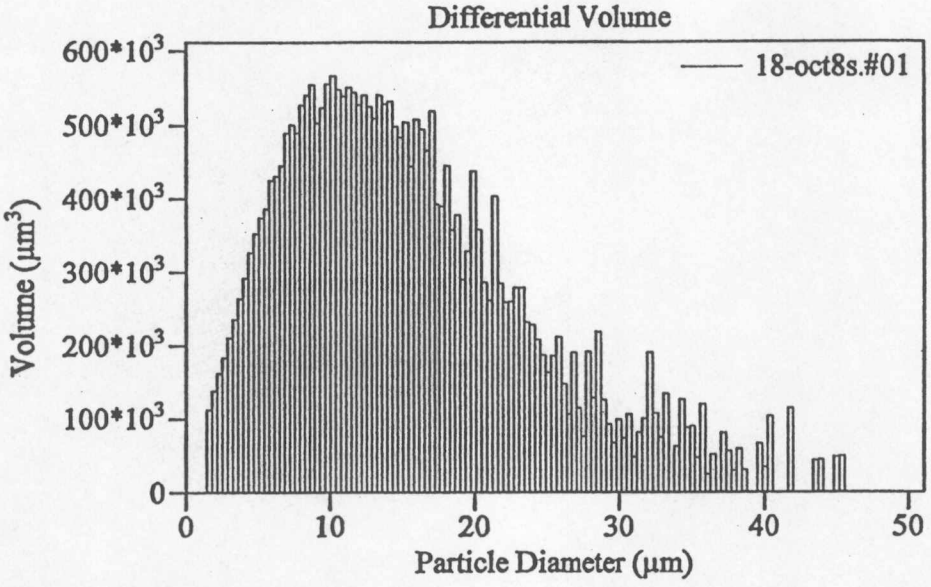
Volume:	8.223e6 µm³		
Mean:	10.45 µm	95% Conf. Limits:	10.42-10.49 µm
Median:	8.940 µm	S.D.:	5.789 µm
Mean/Median Ratio:	1.169		

% <	10	25	50	75	90
µm	4.418	6.303	8.940	13.78	18.38

COULTER AccuComp® 2.01

20 Sep 1999

File name:	18-oct8s.#01	Sample Number:	1
Group ID:	s-b		
Sample ID:	oct8sb3-01		
Comment:	type3 cement		
Operator:	nha		
Raw Count:	287,191	Coinc. Corr. Count:	341,032
Acquired:	22:52 10 Jun 1997		



Volume Statistics (Arithmetic) 18-oct8s.#01

Calculations from 1.436 µm to 45.94 µm

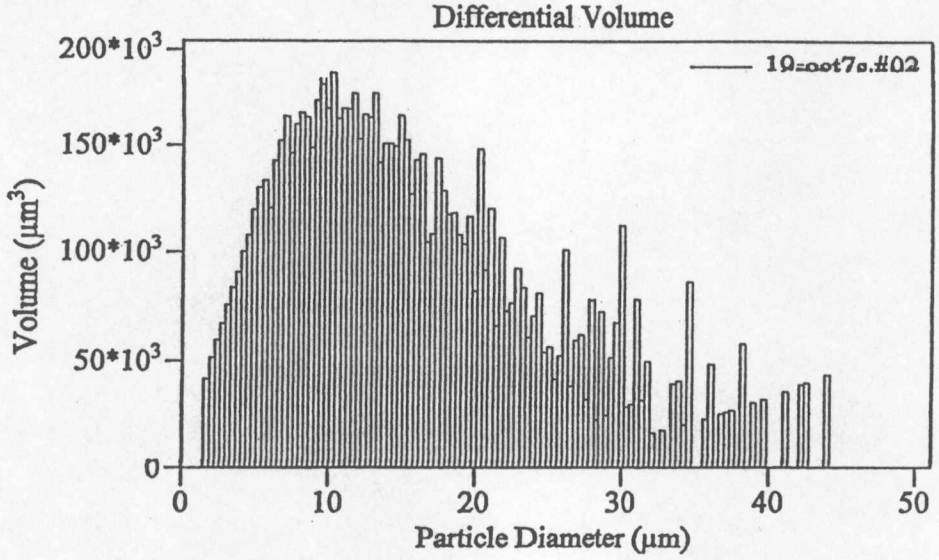
Volume:	30.34e6 µm³		
Mean:	15.62 µm	95% Conf. Limits:	15.59-15.66 µm
Median:	14.15 µm	S.D.:	8.681 µm
Mean/Median Ratio:	1.104		

% <	10	25	50	75	90
µm	5.741	9.076	14.15	20.44	27.95

COULTER AccuComp® 2.01

20 Sep 1999

File name:	19-oct7s.#02	Sample Number:	1
Group ID:	s-b		
Sample ID:	oct7sb3-02		
Comment:	type3		
Operator:	nha		
Raw Count:	99,450	Coinc. Corr. Count:	104,967
Acquired:	22:14 10 Jun 1997		



Volume Statistics (Arithmetic) 19-oct7s.#02

Calculations from 1.436 µm to 45.94 µm

Volume:	9.825e6 µm³		
Mean:	16.13 µm	95% Conf. Limits:	16.07-16.20 µm
Median:	14.36 µm	S.D.:	9.290 µm
Mean/Median Ratio:	1.123		

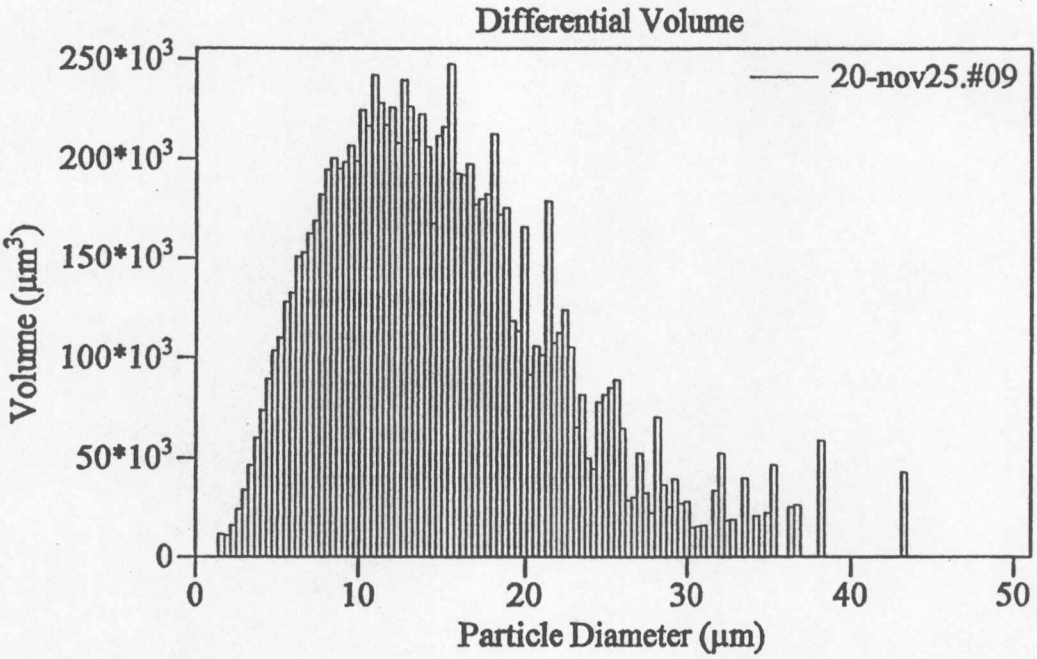
% <	10	25	50	75	90
µm	5.522	9.030	14.36	21.57	29.83

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	20-nov25.#09	Sample Number:	1
Group ID:	Slurry Type3		
Sample ID:	nov25.#09		
Comment:	with delvo		
Operator:	NHA		
Raw Count:	77,232	Coinc. Corr. Count:	80,502
Acquired:	14:27 12 Jun 1997		



Volume Statistics (Arithmetic) 20-nov25.#09

Calculations from 1.436 µm to 45.22 µm

Volume:	10.84e6 µm³		
Mean:	15.23 µm	95% Conf. Limits:	15.16-15.30 µm
Median:	14.19 µm	S.D.:	7.351 µm
Mean/Median Ratio:	1.073		

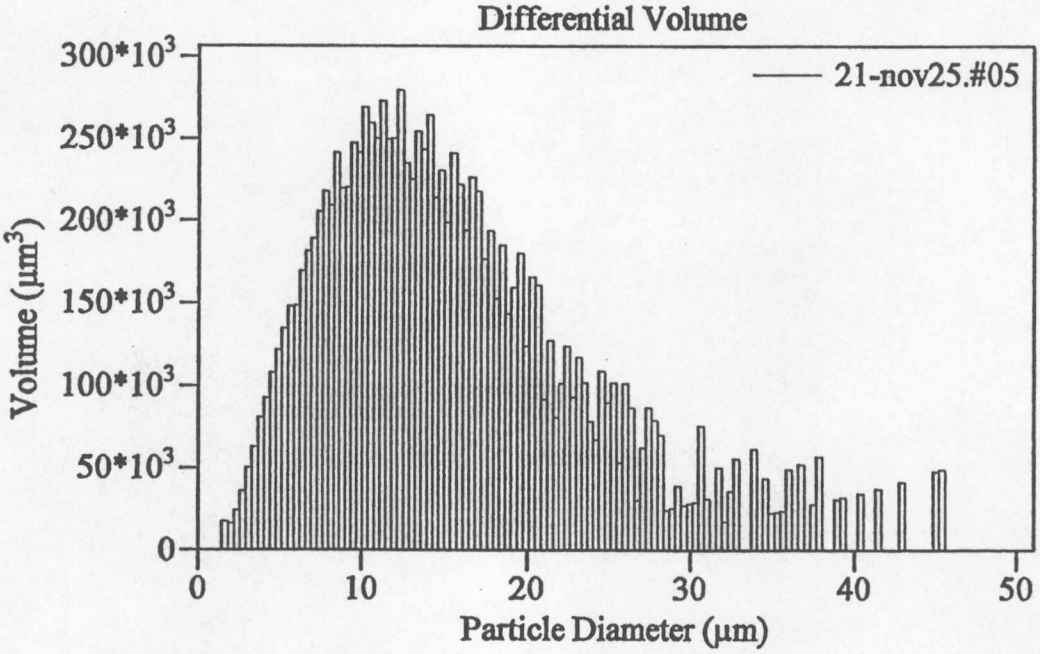
% <	10	25	50	75	90
µm	6.682	9.798	14.19	19.31	25.11

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	21-nov25.#05	Sample Number:	1
Group ID:	Slurry Type3		
Sample ID:	nov25.#05		
Comment:	with delvo		
Operator:	NHA		
Raw Count:	111,482	Coinc. Corr. Count:	118,478
Acquired:	13:20 12 Jun 1997		



Volume Statistics (Arithmetic) 21-nov25.#05

Calculations from 1.436 µm to 45.22 µm

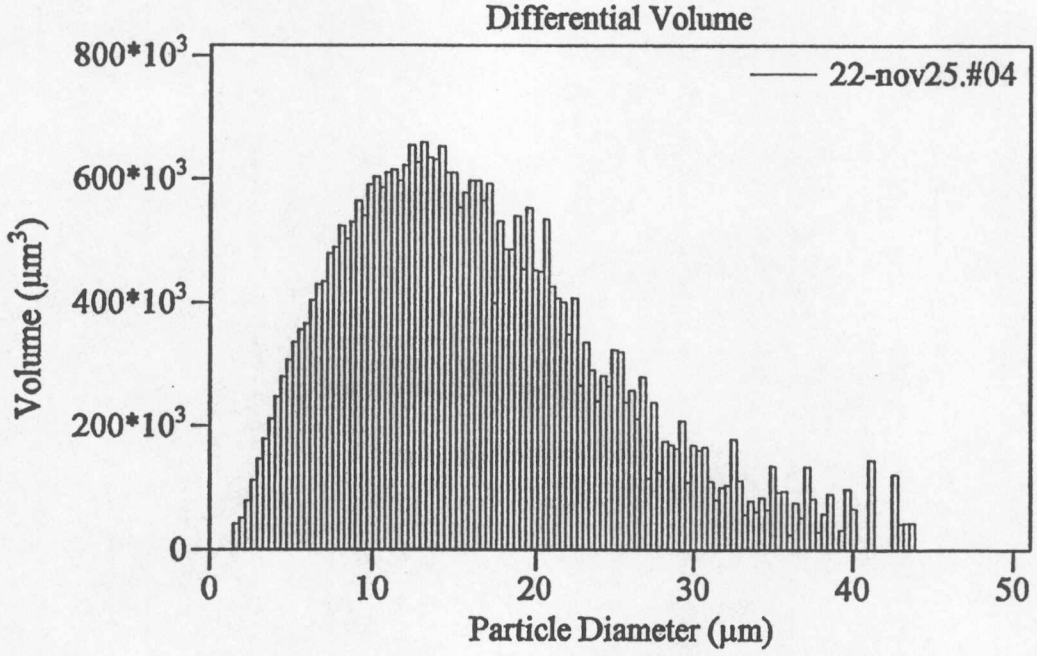
Volume:	12.70e6 µm³		
Mean:	15.79 µm	95% Conf. Limits:	15.72-15.86 µm
Median:	14.24 µm	S.D.:	8.295 µm
Mean/Median Ratio:	1.109		

% <	10	25	50	75	90
µm	6.576	9.737	14.24	20.16	27.21

COULTER AccuComp® 2.01

20 Sep 1999

File name:	22-nov25.#04	Sample Number:	1
Group ID:	Slurry Type3		
Sample ID:	nov25.#04		
Comment:	with delvo		
Operator:	NHA		
Raw Count:	229,735	Coinc. Corr. Count:	262,457
Acquired:	13:01 12 Jun 1997		



Volume Statistics (Arithmetic) 22-nov25.#04

Calculations from 1.436 µm to 45.22 µm

Volume:	34.91e6 µm^3		
Mean:	16.72 µm	95% Conf. Limits:	16.68-16.77 µm
Median:	15.42 µm	S.D.:	8.503 µm
Mean/Median Ratio:	1.085		

% <	10	25	50	75	90
µm	6.766	10.36	15.42	21.58	28.68

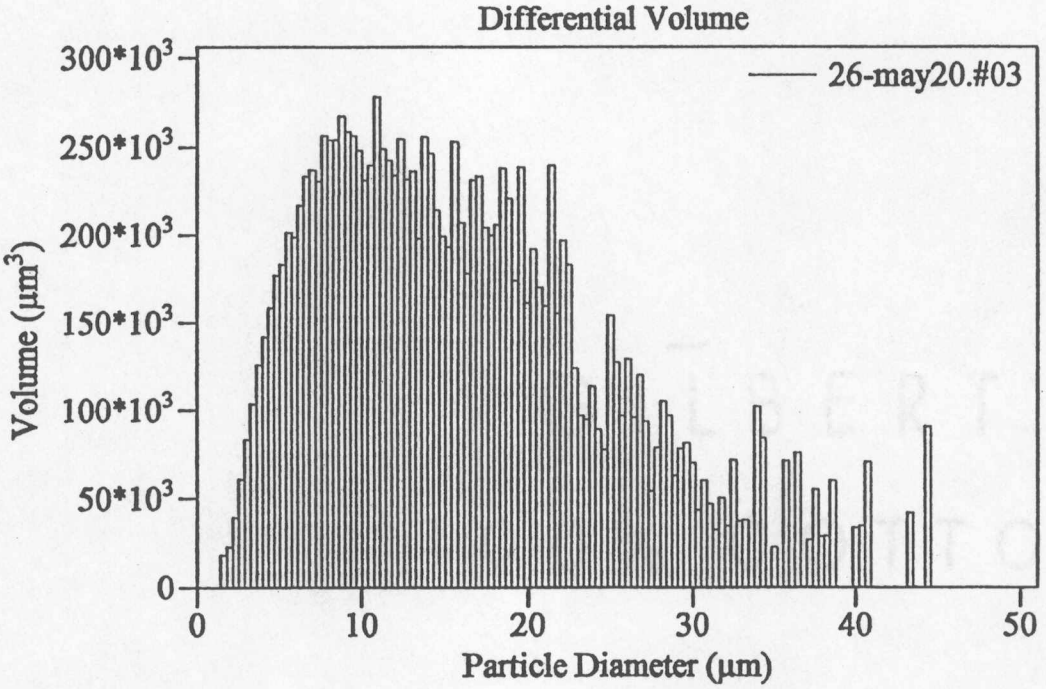
APPENDIX C-2

QUINETICS COLLOIDAL MILL TRIAL RUNS DATA

COULTER AccuComp® 2.01

20 Sep 1999

File name:	26-may20.#03	Sample Number:	3
Group ID:	May20y99.#02		
Sample ID:	Type I Slurry before mill proccess		
Comment:	Type I cement before mill w/ delvo w/c=0.5		
Operator:	NHA		
Raw Count:	135,762	Coinc. Corr. Count:	1
Acquired:	5:58 16 Jun 1997		
Background Run:	MAY20Y99.#03		



Volume Statistics (Arithmetic) 26-may20.#03

Calculations from 1.436 µm to 41.28 µm

Volume:	14.94e6 µm³		
Mean:	16.19 µm	95% Conf. Limits:	16.13-16.26 µm
Median:	14.94 µm	S.D.:	8.587 µm
Mean/Median Ratio:	1.084		

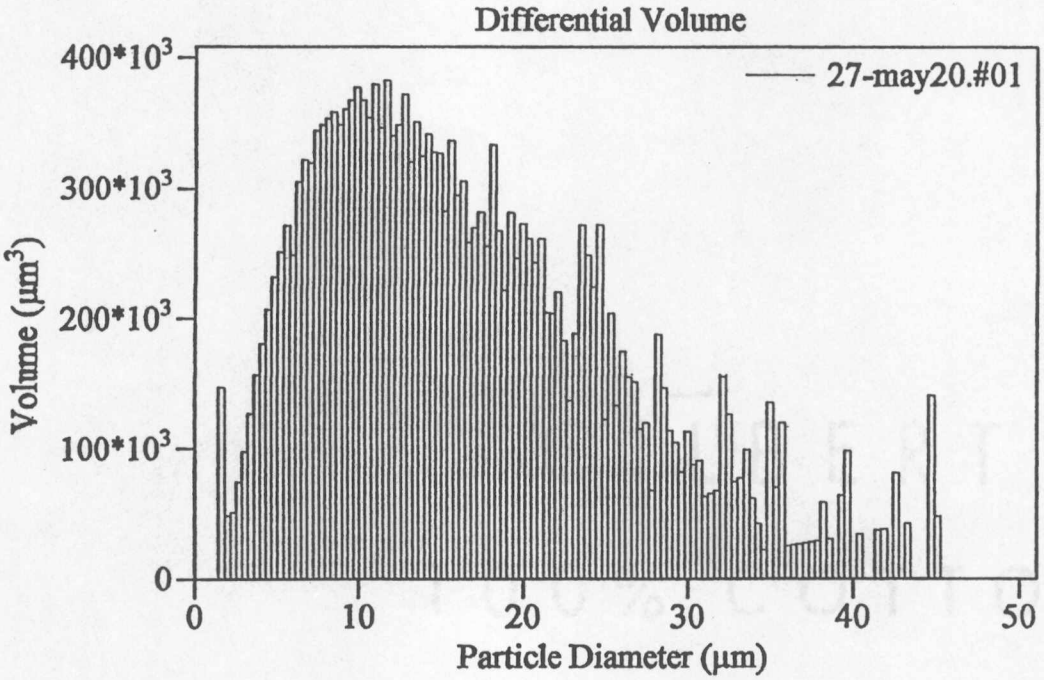
% <	10	25	50	75	90
µm	6.063	9.346	14.94	21.47	28.39

COULTER

AccuComp® 2.01

20 Sep 1999

File name:	27-may20.#01	Sample Number:	3
Group ID:	May20y99.#01		
Sample ID:	Type I Slurry after mill proccess		
Comment:	Type I cement after mill w/ delvo w/c=0.5		
Operator:	NHA		
Raw Count:	293,855	Coinc. Corr. Count:	350,577
Acquired:	5:31 16 Jun 1997		
Background Run:	MAY20Y99.#01		



Volume Statistics (Arithmetic) 27-may20.#01

Calculations from 1.436 µm to 41.28 µm

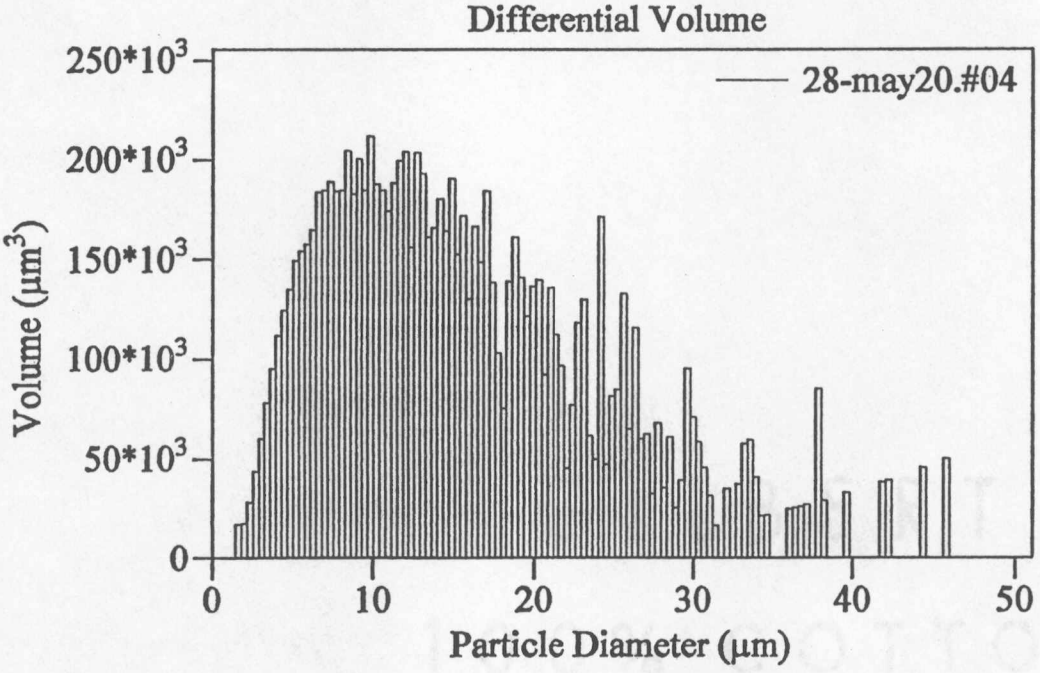
Volume:	21.40e6 µm³		
Mean:	16.40 µm	95% Conf. Limits:	16.36-16.44 µm
Median:	14.98 µm	S.D.:	8.683 µm
Mean/Median Ratio:	1.095		

% <	10	25	50	75	90
µm	6.157	9.523	14.98	22.18	28.93

COULTER AccuComp® 2.01

22 Sep 1999

File name:	28-may20.#04	Sample Number:	3
Group ID:	May20y99.#04		
Sample ID:	Type I Slurry before mill proccess		
Comment:	Type I cement before mill w/ delvo w/c=0.5		
Operator:	NHA		
Raw Count:	117,607	Coinc. Corr. Count:	1
Acquired:	6:14 16 Jun 1997		
Background Run:	MAY20Y99.#04		



Volume Statistics (Arithmetic) 28-may20.#04

Calculations from 1.436 µm to 41.28 µm

Volume:	10.75e6 µm^3		
Mean:	15.64 µm	95% Conf. Limits:	15.57-15.71 µm
Median:	14.21 µm	S.D.:	8.334 µm
Mean/Median Ratio:	1.101		

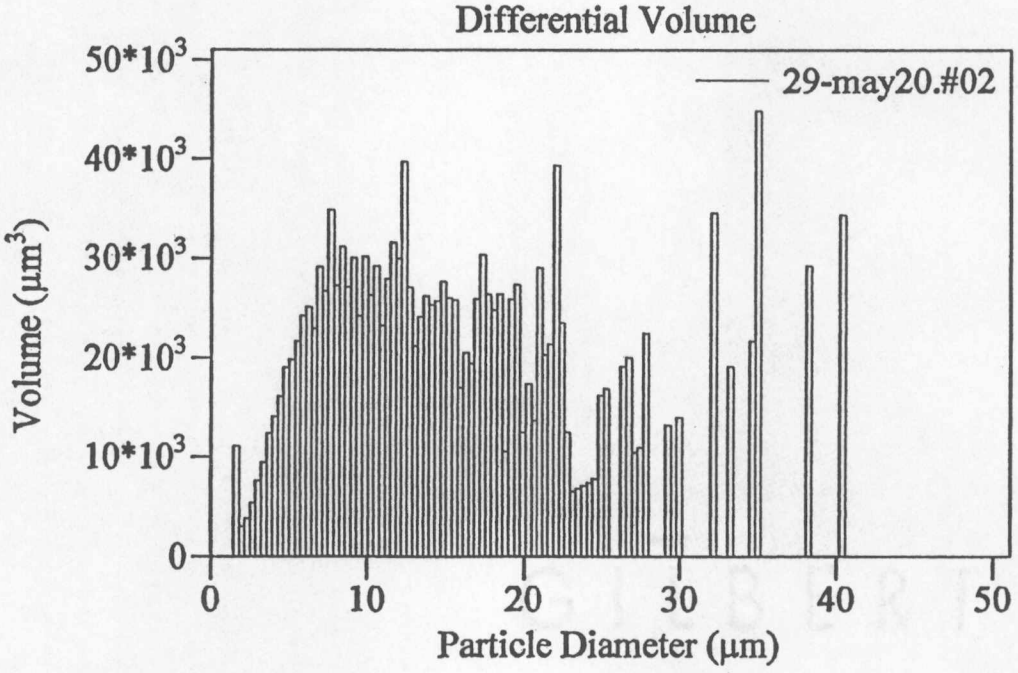
% <	10	25	50	75	90
µm	5.883	9.039	14.21	20.97	27.56

COULTER

AccuComp® 2.01

22 Sep 1999

File name:	29-may20.#02	Sample Number:	3
Group ID:	May20y99.#02		
Sample ID:	Type I Slurry after mill proccess		
Comment:	Type I cement after mill w/ delvo w/c=0.5		
Operator:	NHA		
Raw Count:	62,397	Coinc. Corr. Count:	69,570
Acquired:	5:43 16 Jun 1997		



Volume Statistics (Arithmetic) 29-may20.#02

Calculations from 1.436 µm to 41.28 µm

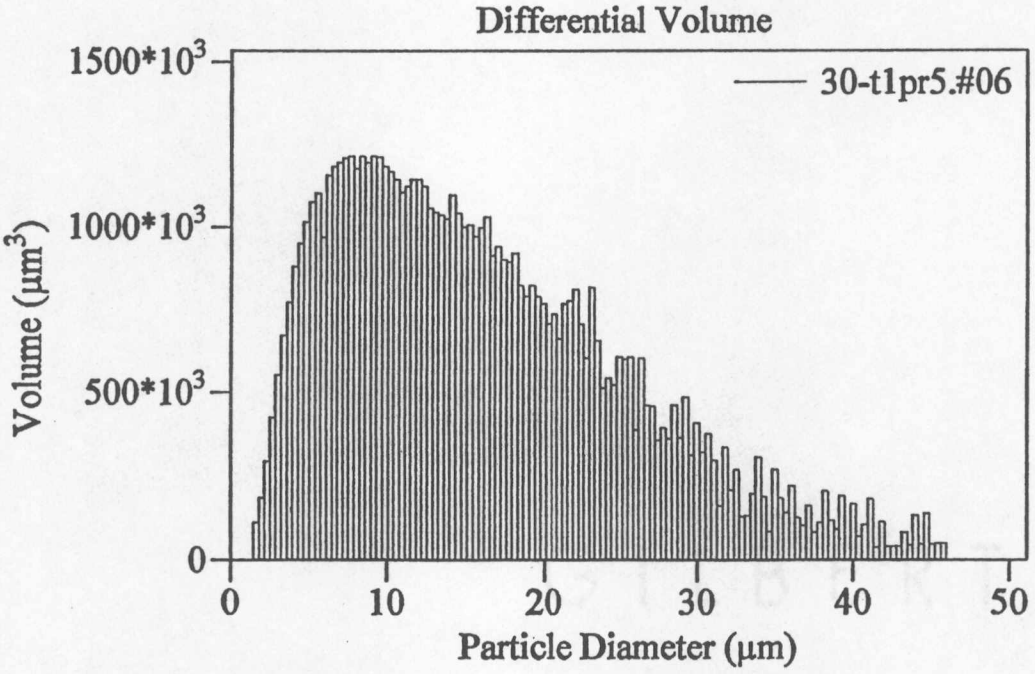
Volume:	1.724e6 µm³		
Mean:	16.73 µm	95% Conf. Limits:	16.56-16.90 µm
Median:	15.06 µm	S.D.:	9.254 µm
Mean/Median Ratio:	1.111		

% <	10	25	50	75	90
µm	6.166	9.454	15.06	21.97	32.07

COULTER AccuComp® 2.01

22 Sep 1999

File name:	30-t1pr5.#06	Sample Number:	3
Group ID:	t1pr0513.04m		
Sample ID:	type I slurry before mill process		
Comment:	1:1 ratio w/delvo 2		
Operator:	nha		
Raw Count:	625,717	Coinc. Corr. Count:	1
Acquired:	21:41 15 Jun 1997		
Background Run:	T1PR0513.#06		



Volume Statistics (Arithmetic) 30-t1pr5.#06

Calculations from 1.436 µm to 41.28 µm

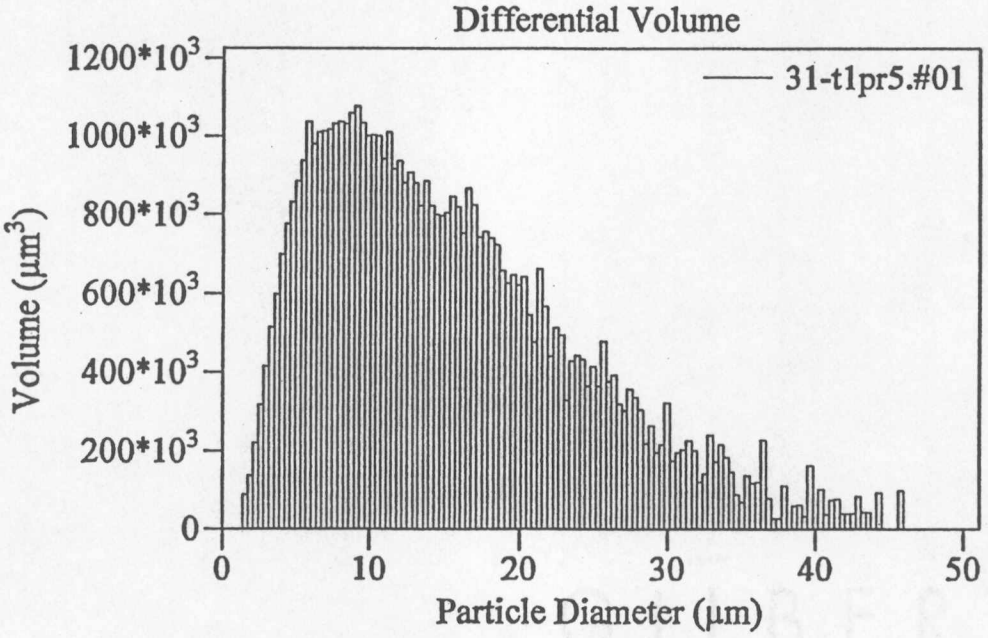
Volume:	71.33e6 µm^3		
Mean:	15.90 µm	95% Conf. Limits:	15.88-15.93 µm
Median:	14.45 µm	S.D.:	8.852 µm
Mean/Median Ratio:	1.101		

% <	10	25	50	75	90
µm	5.446	8.752	14.45	21.80	28.71

COULTER AccuComp® 2.01

22 Sep 1999

File name:	31-t1pr5.#01	Sample Number:	3
Group ID:	t1pr0513.01		
Sample ID:	type I slurry after mill process		
Comment:	1:1 ratio with delvo		
Operator:	nha		
Raw Count:	509,482	Coinc. Corr. Count:	1
Acquired:	19:28 15 Jun 1997		



Volume Statistics (Arithmetic) 31-t1pr5.#01

Calculations from 1.436 µm to 41.28 µm

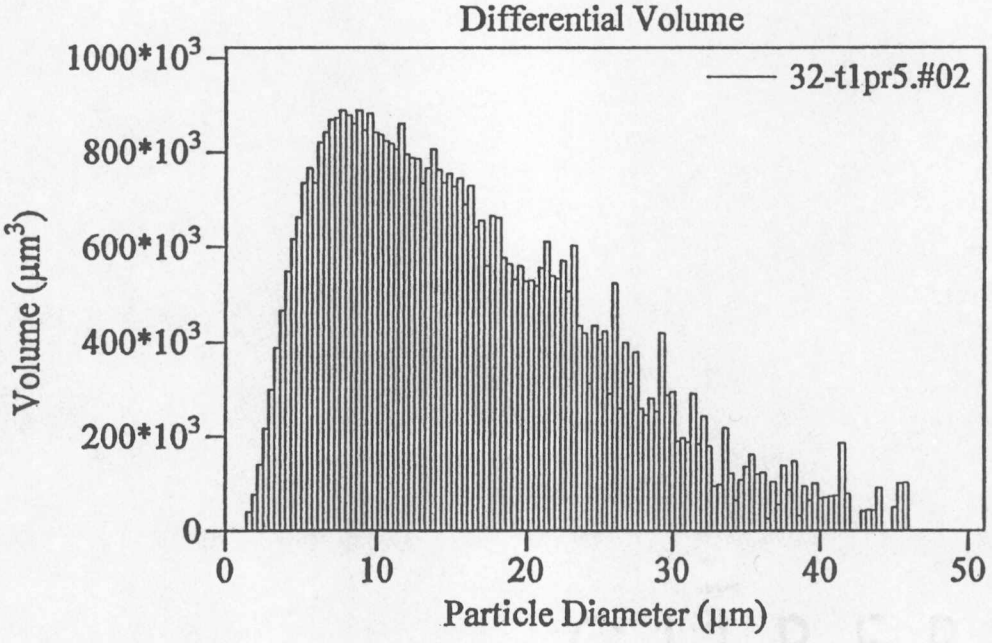
Volume:	56.79e6 µm^3		
Mean:	15.27 µm	95% Conf. Limits:	15.24-15.30 µm
Median:	13.77 µm	S.D.:	8.497 µm
Mean/Median Ratio:	1.108		

% <	10	25	50	75	90
µm	5.458	8.480	13.77	20.56	27.59

COULTER AccuComp® 2.01

22 Sep 1999

File name:	32-t1pr5.#02	Sample Number:	3
Group ID:	t1pr0513.02		
Sample ID:	type I slurry after mill process		
Comment:	1:1 ratio with delvo		
Operator:	nha		
Raw Count:	379,040	Coinc. Corr. Count:	1
Acquired:	19:44 15 Jun 1997		
Background Run:	T1PR0513.#02		



Volume Statistics (Arithmetic) 32-t1pr5.#02

Calculations from 1.436 µm to 41.28 µm

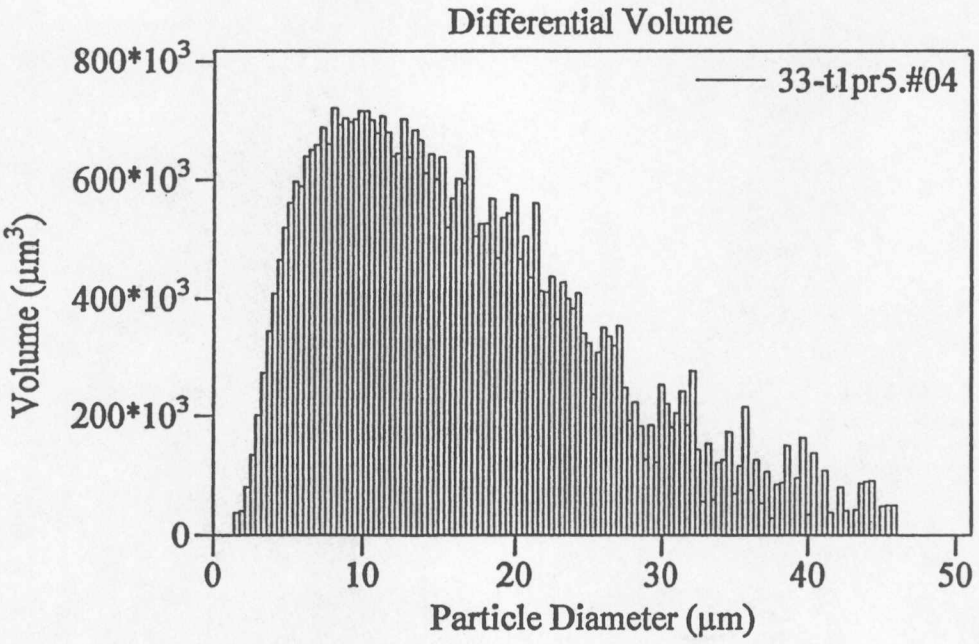
Volume:	50.56e6 µm^3		
Mean:	16.06 µm	95% Conf. Limits:	16.03-16.10 µm
Median:	14.61 µm	S.D.:	8.711 µm
Mean/Median Ratio:	1.099		

% <	10	25	50	75	90
µm	5.793	8.989	14.61	21.93	28.75

COULTER AccuComp® 2.01

22 Sep 1999

File name:	33-t1pr5.#04	Sample Number:	3
Group ID:	t1pr0513.04		
Sample ID:	type I slurry after mill process		
Comment:	1:1 ratio w/delvo 2 passes through the mill		
Operator:	nha		
Raw Count:	325,457	Coinc. Corr. Count:	1
Acquired:	20:51 15 Jun 1997		
Background Run:	T1PR0513.#04		



Volume Statistics (Arithmetic) 33-t1pr5.#04

Calculations from 1.436 µm to 41.28 µm

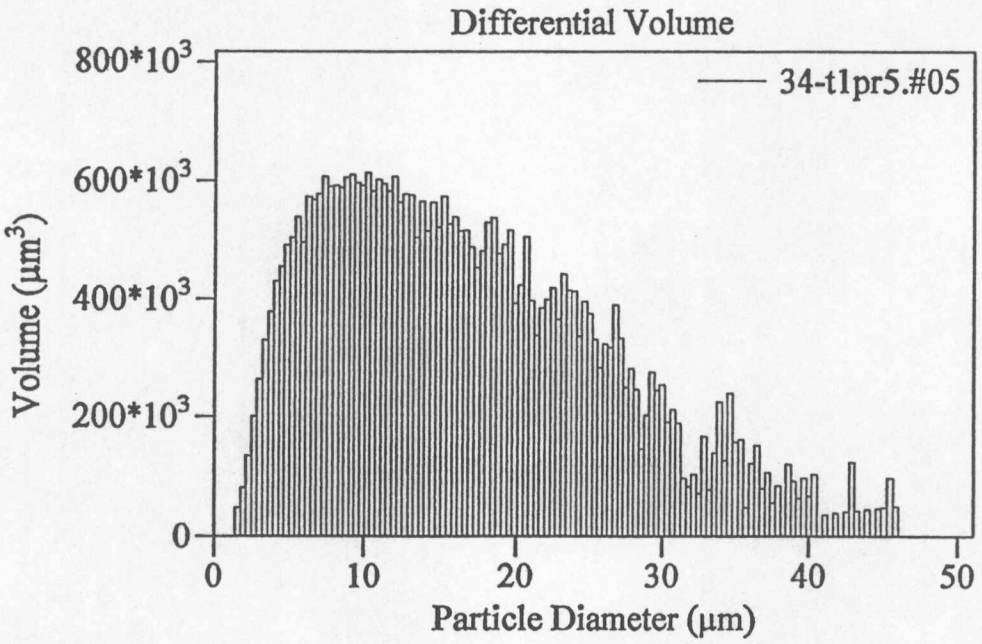
Volume:	42.06e6 µm³		
Mean:	16.46 µm	95% Conf. Limits:	16.42-16.50 µm
Median:	15.03 µm	S.D.:	8.802 µm
Mean/Median Ratio:	1.095		

% <	10	25	50	75	90
µm	6.072	9.406	15.03	22.09	29.36

COULTER AccuComp® 2.01

22 Sep 1999

File name:	34-t1pr5.#05	Sample Number:	3
Group ID:	t1pr0513.04m		
Sample ID:	type I slurry after mill process		
Comment:	1:1 ratio w/delvo 2 passes through the mill		
Operator:	nha		
Raw Count:	331,847	Coinc. Corr. Count:	1
Acquired:	21:23 15 Jun 1997		
Background Run:	T1PR0513.#05		



Volume Statistics (Arithmetic) 34-t1pr5.#05

Calculations from 1.436 µm to 41.28 µm

Volume:	38.86e6 µm³		
Mean:	16.69 µm	95% Conf. Limits:	16.65-16.73 µm
Median:	15.43 µm	S.D.:	9.004 µm
Mean/Median Ratio:	1.081		

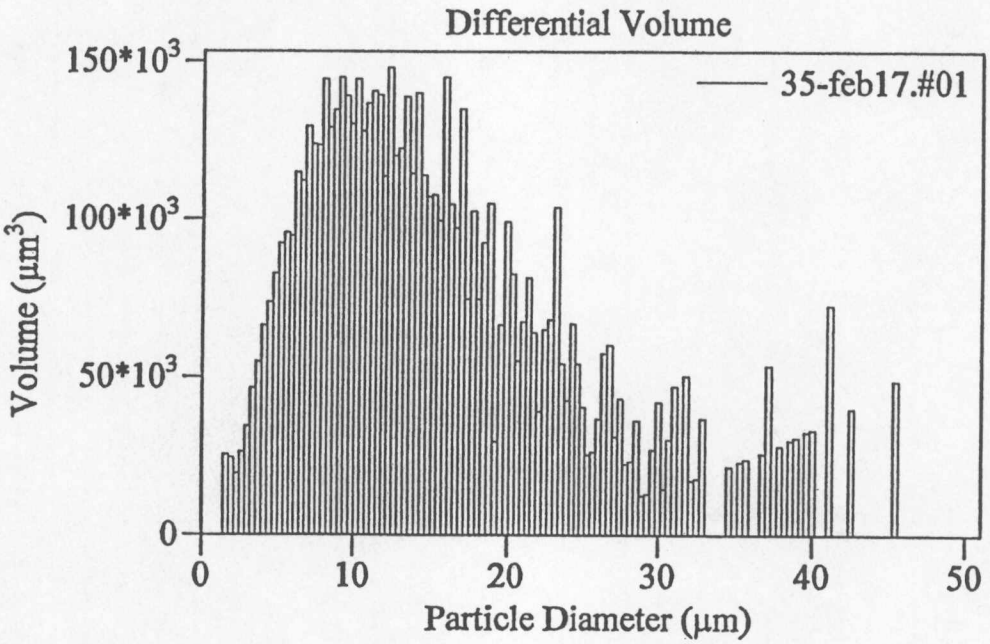
% <	10	25	50	75	90
µm	5.761	9.352	15.43	22.98	29.55

COULTER

AccuComp® 2.01

22 Sep 1999

File name:	35-feb17.#01	Sample Number:	3
Group ID:	Feb17-#01		
Sample ID:	Before the mill process		
Comment:	Type I Cement Slurry with Delvo		
Operator:	NHA		
Raw Count:	109,629	Coinc. Corr. Count:	1
Acquired:	15:38 15 Jun 1997		



Volume Statistics (Arithmetic) 35-feb17.#01

Calculations from 1.436 µm to 41.28 µm

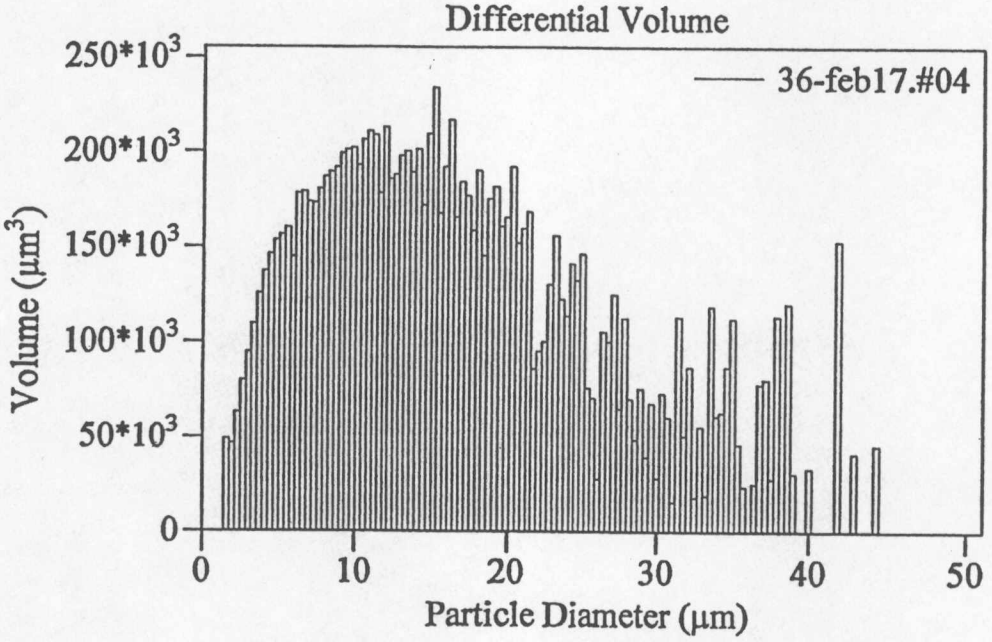
Volume:	7.252e6 µm^3		
Mean:	15.79 µm	95% Conf. Limits:	15.70-15.87 µm
Median:	14.02 µm	S.D.:	8.813 µm
Mean/Median Ratio:	1.126		

% <	10	25	50	75	90
µm	6.053	9.124	14.02	20.73	28.32

COULTER AccuComp® 2.01

22 Sep 1999

File name:	36-feb17.#04	Sample Number:	3
Group ID:	Feb17-#04		
Sample ID:	Before the mill proccess		
Comment:	Type I Cement Slurry with Delvo		
Operator:	NHA		
Raw Count:	179,407	Coinc. Corr. Count:	1
Acquired:	16:30 15 Jun 1997		



Volume Statistics (Arithmetic) 36-feb17.#04

Calculations from 1.436 µm to 41.28 µm

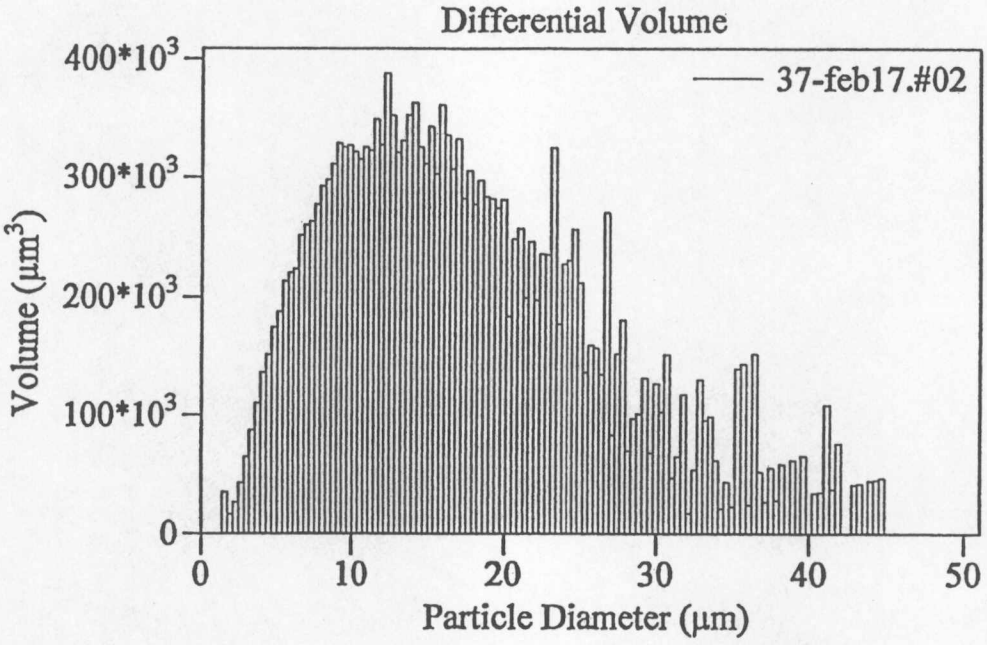
Volume:	13.22e6 µm³		
Mean:	16.98 µm	95% Conf. Limits:	16.92-17.04 µm
Median:	15.64 µm	S.D.:	9.249 µm
Mean/Median Ratio:	1.086		

% <	10	25	50	75	90
µm	5.742	9.660	15.64	23.05	31.37

COULTER AccuComp® 2.01

22 Sep 1999

File name:	37-feb17.#02	Sample Number:	3
Group ID:	Feb17-#01		
Sample ID:	After the mill proccess		
Comment:	Type I Cement Slurry with Delvo		
Operator:	NHA		
Raw Count:	169,063	Coinc. Corr. Count:	1
Acquired:	15:55 15 Jun 1997		



Volume Statistics (Arithmetic) 37-feb17.#02

Calculations from 1.436 µm to 41.28 µm

Volume:	20.71e6 µm^3		
Mean:	17.43 µm	95% Conf. Limits:	17.37-17.49 µm
Median:	16.17 µm	S.D.:	8.685 µm
Mean/Median Ratio:	1.078		

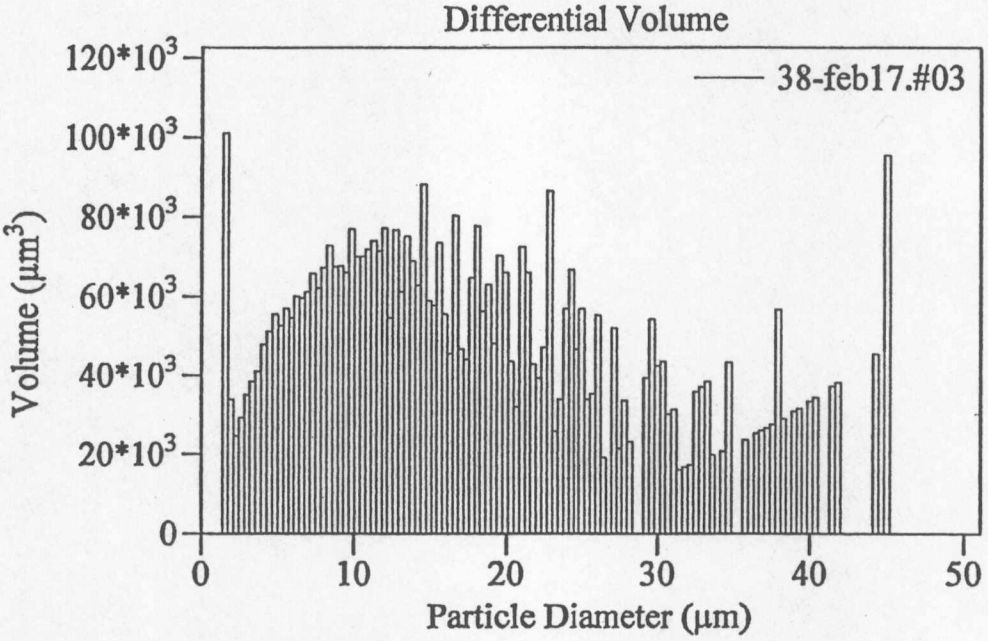
% <	10	25	50	75	90
µm	6.995	10.67	16.17	23.12	29.98

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AccuComp® 2.01

22 Sep 1999

File name:	38-feb17.#03	Sample Number:	3
Group ID:	Feb17-#03		
Sample ID:	After the mill proccess		
Comment:	Type I Cement Slurry with Delvo		
Operator:	NHA		
Raw Count:	206,607	Coinc. Corr. Count:	1
Acquired:	16:14 15 Jun 1997		



Volume Statistics (Arithmetic) 38-feb17.#03

Calculations from 1.436 µm to 41.28 µm

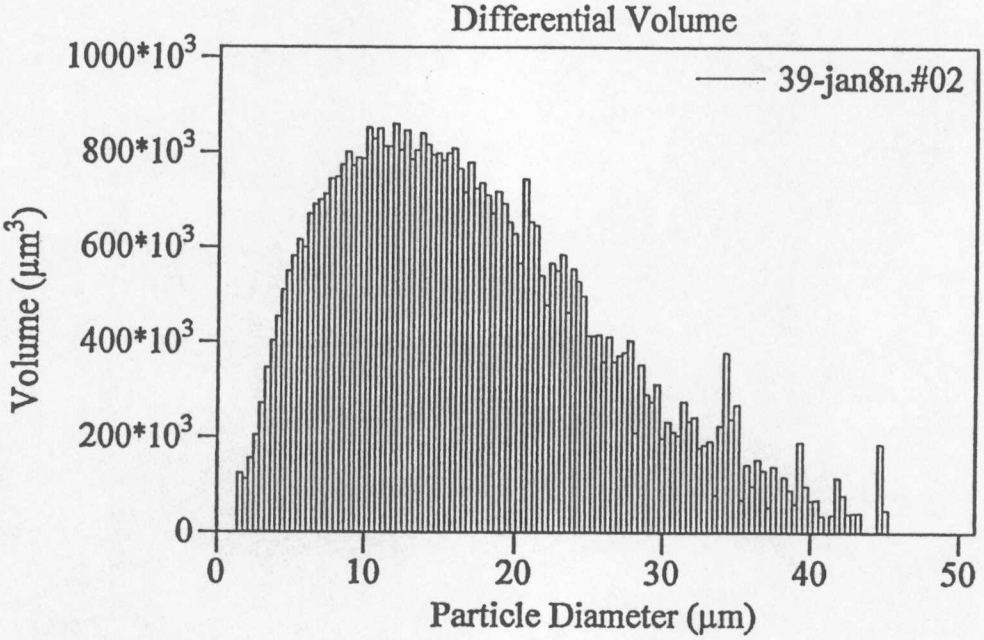
Volume:	5.013e6 µm³		
Mean:	17.53 µm	95% Conf. Limits:	17.46-17.60 µm
Median:	16.06 µm	S.D.:	10.00 µm
Mean/Median Ratio:	1.092		

% <	10	25	50	75	90
µm	5.318	9.579	16.06	24.19	32.71

COULTER AccuComp® 2.01

22 Sep 1999

File name:	39-jan8n.#02	Sample Number:	3
Group ID:	Jan8N1.#02		
Sample ID:	1/08/99		
Comment:	1St run, non process sample with 5% delvo		
Operator:	NHA		
Raw Count:	472,484	Coinc. Corr. Count:	1
Acquired:	11:34 15 Jun 1997		



Volume Statistics (Arithmetic) 39-jan8n.#02

Calculations from 1.436 µm to 41.28 µm

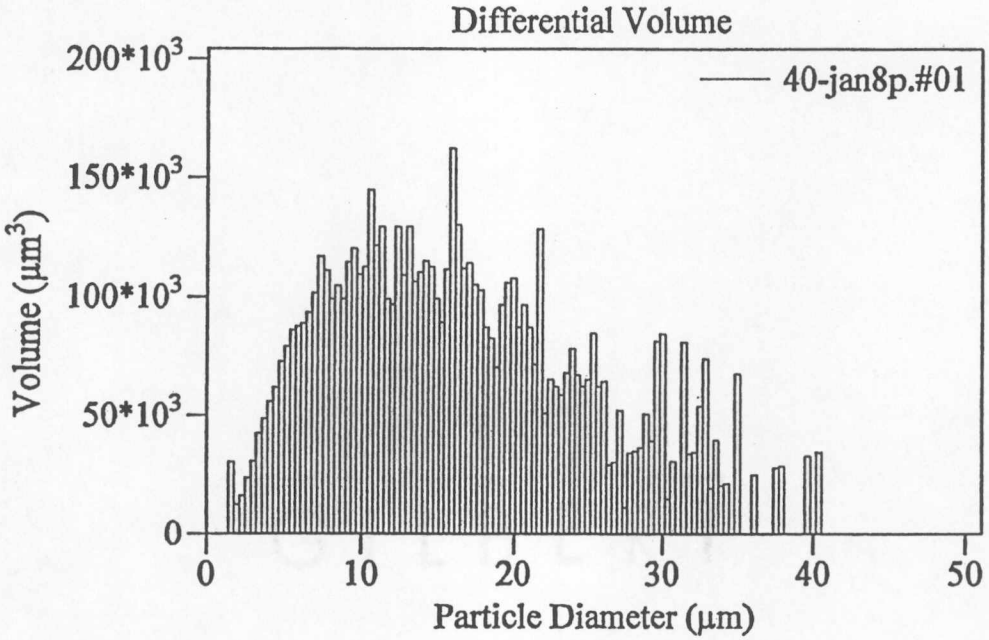
Volume:	52.07e6 µm^3		
Mean:	16.85 µm	95% Conf. Limits:	16.82-16.88 µm
Median:	15.71 µm	S.D.:	8.698 µm
Mean/Median Ratio:	1.073		

% <	10	25	50	75	90
µm	6.253	10.00	15.71	22.62	29.39

COULTER AccuComp® 2.01

22 Sep 1999

File name:	40-jan8p.#01	Sample Number:	3
Group ID:	jan8p1.#01		
Sample ID:	1/08/99		
Comment:	1St run, 1 pass processed sample with 5% delvo		
Operator:	NHA		
Raw Count:	111,003	Coinc. Corr. Count:	1
Acquired:	11:54 15 Jun 1997		



Volume Statistics (Arithmetic) 40-jan8p.#01

Calculations from 1.436 µm to 41.28 µm

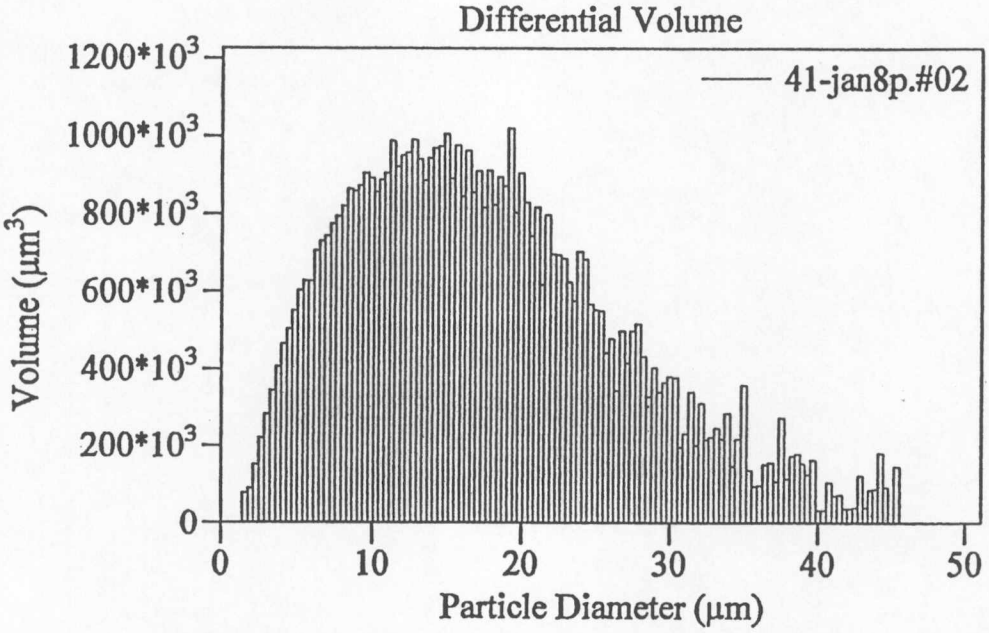
Volume:	7.280e6 µm^3		
Mean:	16.86 µm	95% Conf. Limits:	16.77-16.94 µm
Median:	15.83 µm	S.D.:	8.609 µm
Mean/Median Ratio:	1.065		

% <	10	25	50	75	90
µm	6.429	10.09	15.83	22.47	29.76

COULTER AccuComp® 2.01

22 Sep 1999

File name:	41-jan8p.#02	Sample Number:	3
Group ID:	jan8p1b1.#02		
Sample ID:	1/08/99		
Comment:	2nd. run,1 pass processed sample with 5% delvo		
Operator:	NHA		
Raw Count:	429,064	Coinc. Corr. Count:	1
Acquired:	13:03 15 Jun 1997		



Volume Statistics (Arithmetic) 41-jan8p.#02

Calculations from 1.436 µm to 41.28 µm

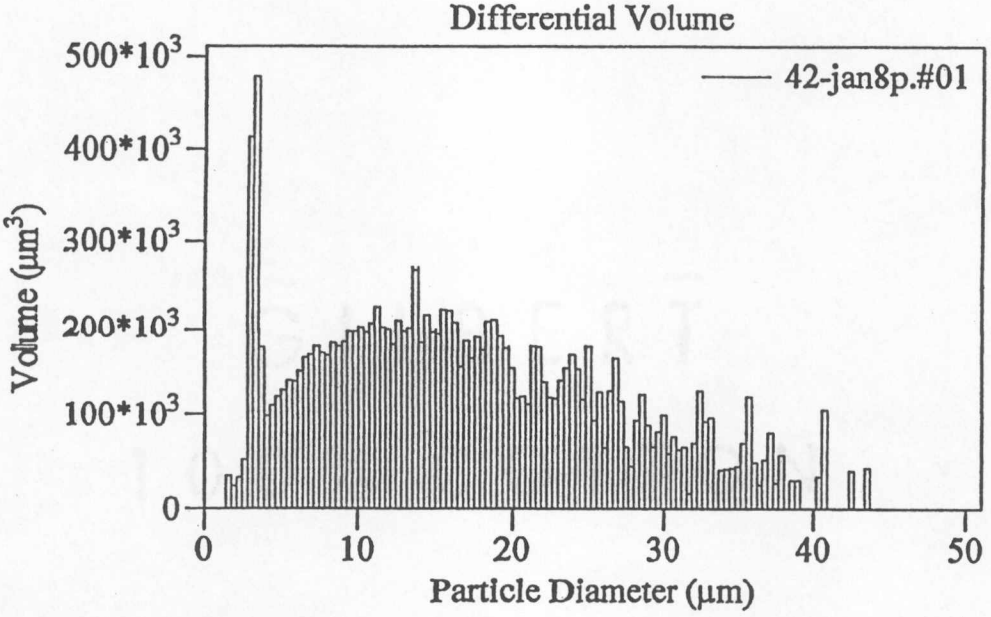
Volume:	61.01e6 µm³		
Mean:	17.41 µm	95% Conf. Limits:	17.37-17.44 µm
Median:	16.40 µm	S.D.:	8.714 µm
Mean/Median Ratio:	1.062		

% <	10	25	50	75	90
µm	6.672	10.59	16.40	23.11	29.83

COULTER AccuComp® 2.01

22 Sep 1999

File name:	42-jan8p.#01	Sample Number:	3
Group ID:	jan8p1b.#02		
Sample ID:	1/08/99		
Comment:	2nd. run,1 pass processed sample with 5% delvo		
Operator:	NHA		
Raw Count:	210,330	Coinc. Corr. Count:	1
Acquired:	12:42 15 Jun 1997		



Volume Statistics (Arithmetic) 42-jan8p.#01

Calculations from 1.436 µm to 41.28 µm

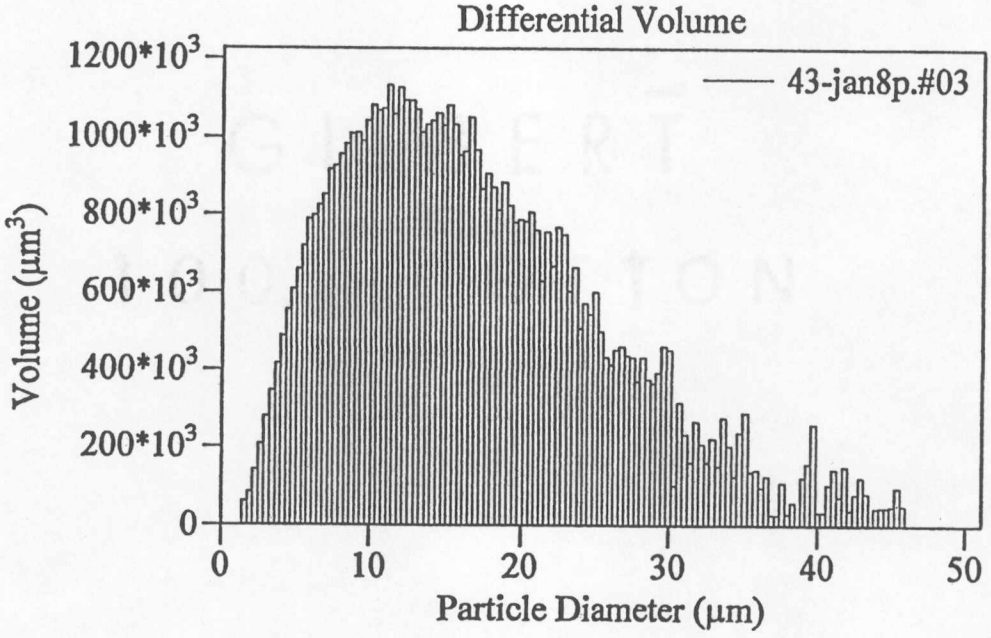
Volume:	14.17e6 µm^3		
Mean:	16.90 µm	95% Conf. Limits:	16.85-16.96 µm
Median:	15.71 µm	S.D.:	9.442 µm
Mean/Median Ratio:	1.076		

% <	10	25	50	75	90
µm	4.661	9.494	15.71	23.50	30.70

COULTER AccuComp® 2.01

22 Sep 1999

File name:	43-jan8p.#03	Sample Number:	3
Group ID:	jan8p1b2.#03		
Sample ID:	1/08/99		
Comment:	2nd. run,1 pass processed sample with 5% delvo		
Operator:	NHA		
Raw Count:	388,884	Coinc. Corr. Count:	1
Acquired:	13:20 15 Jun 1997		



Volume Statistics (Arithmetic) 43-jan8p.#03

Calculations from 1.436 µm to 41.28 µm

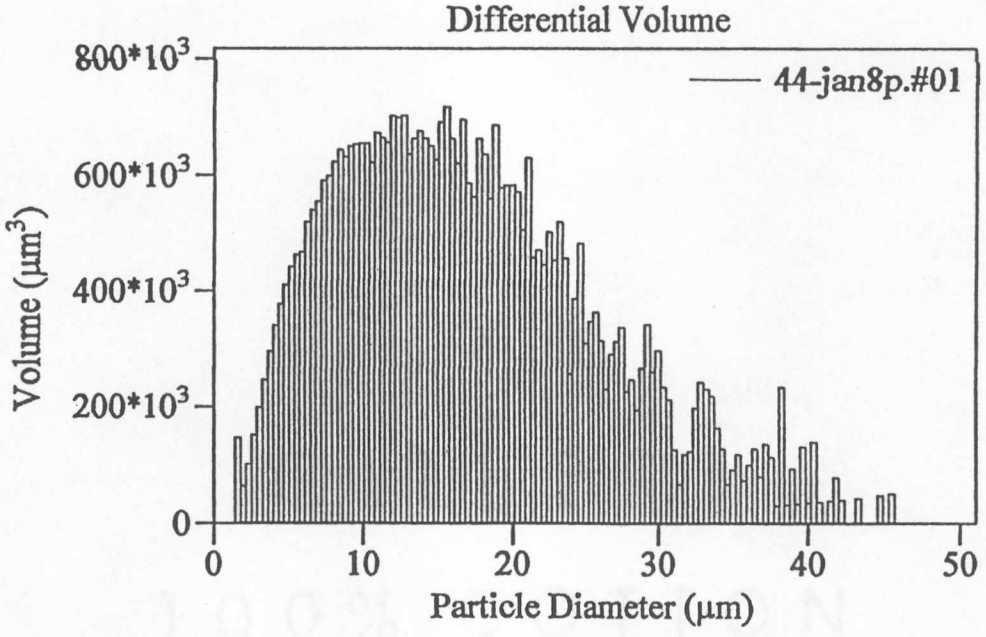
Volume:	63.44e6 µm³		
Mean:	16.63 µm	95% Conf. Limits:	16.60-16.67 µm
Median:	15.42 µm	S.D.:	8.396 µm
Mean/Median Ratio:	1.078		

% <	10	25	50	75	90
µm	6.539	10.11	15.42	22.11	28.64

COULTER AccuComp® 2.01

22 Sep 1999

File name:	44-jan8p.#01	Sample Number:	3
Group ID:	jan8p2b2.#01		
Sample ID:	1/08/99		
Comment:	2nd. run,2 pass processed sample with 5% delvo		
Operator:	NHA		
Raw Count:	415,582	Coinc. Corr. Count:	1
Acquired:	13:40 15 Jun 1997		



Volume Statistics (Arithmetic) 44-jan8p.#01

Calculations from 1.436 µm to 41.28 µm

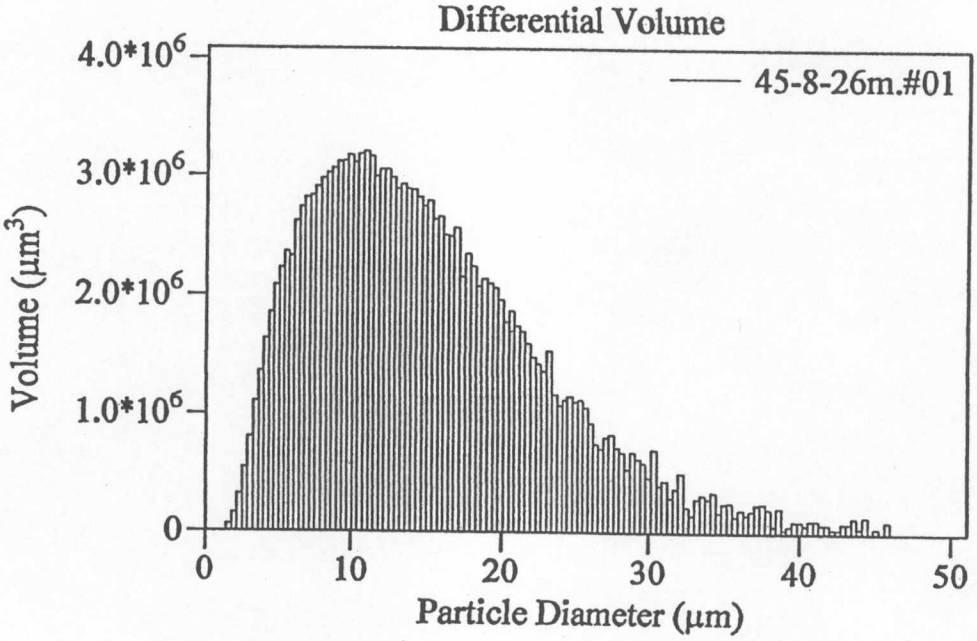
Volume:	42.67e6 µm³		
Mean:	17.00 µm	95% Conf. Limits:	16.97-17.04 µm
Median:	15.93 µm	S.D.:	8.647 µm
Mean/Median Ratio:	1.067		

% <	10	25	50	75	90
µm	6.489	10.21	15.93	22.55	29.41

COULTER AccuComp® 2.01

22 Sep 1999

File name:	45-8-26m.#01	Sample Number:	1
Group ID:	Type I Portland cement Slurry		
Sample ID:	8-26.#01		
Comment:	After the mill w/5% Delvo		
Operator:	NHA		
Raw Count:	1,001,066	Coinc. Corr. Count:	1
Acquired:	13:26 14 Jun 1997		
Background Run:	8-26MB.#01		



Volume Statistics (Arithmetic) 45-8-26m.#01

Calculations from 1.436 µm to 41.28 µm

Volume:	161.8e6 µm^3		
Mean:	14.95 µm	95% Conf. Limits:	14.94-14.97 µm
Median:	13.77 µm	S.D.:	7.645 µm
Mean/Median Ratio:	1.086		

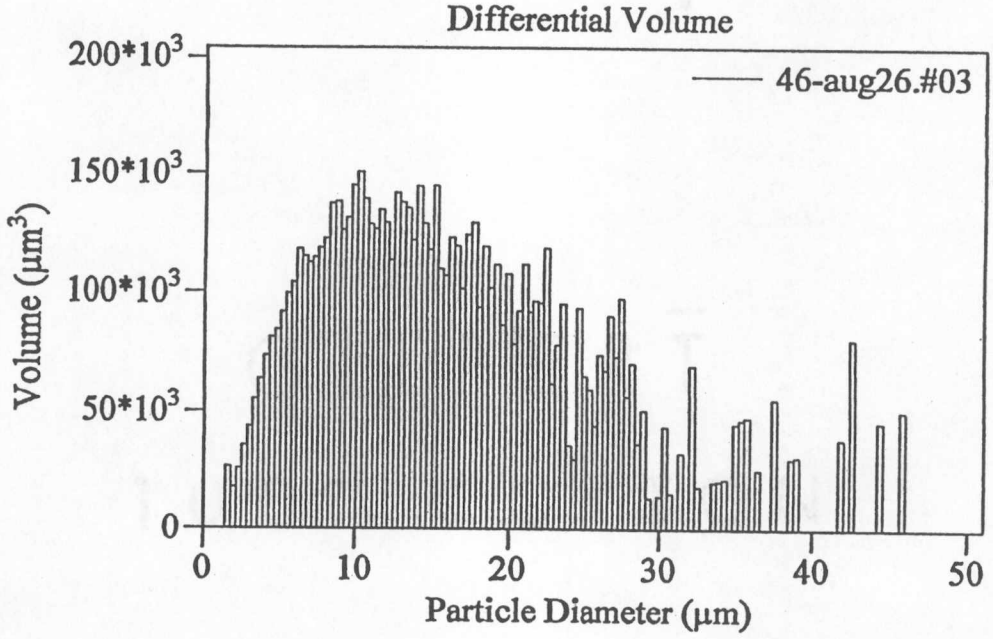
% <	10	25	50	75	90
µm	5.997	9.041	13.77	19.63	25.61

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AccuComp® 2.01

22 Sep 1999

File name:	46-aug26.#03	Sample Number:	1
Group ID:	Type I Portland cement Slurry		
Sample ID:	Aug26.#03		
Comment:	After the mill w/5% Delvo		
Operator:	NHA		
Raw Count:	107,130	Coinc. Corr. Count:	1
Acquired:	14:16 14 Jun 1997		
Background Run:	AUG26MSB.#03		



Volume Statistics (Arithmetic) 46-aug26.#03

Calculations from 1.436 µm to 41.28 µm

Volume:	8.020e6 µm³		
Mean:	16.13 µm	95% Conf. Limits:	16.06-16.21 µm
Median:	15.01 µm	S.D.:	8.310 µm
Mean/Median Ratio:	1.075		

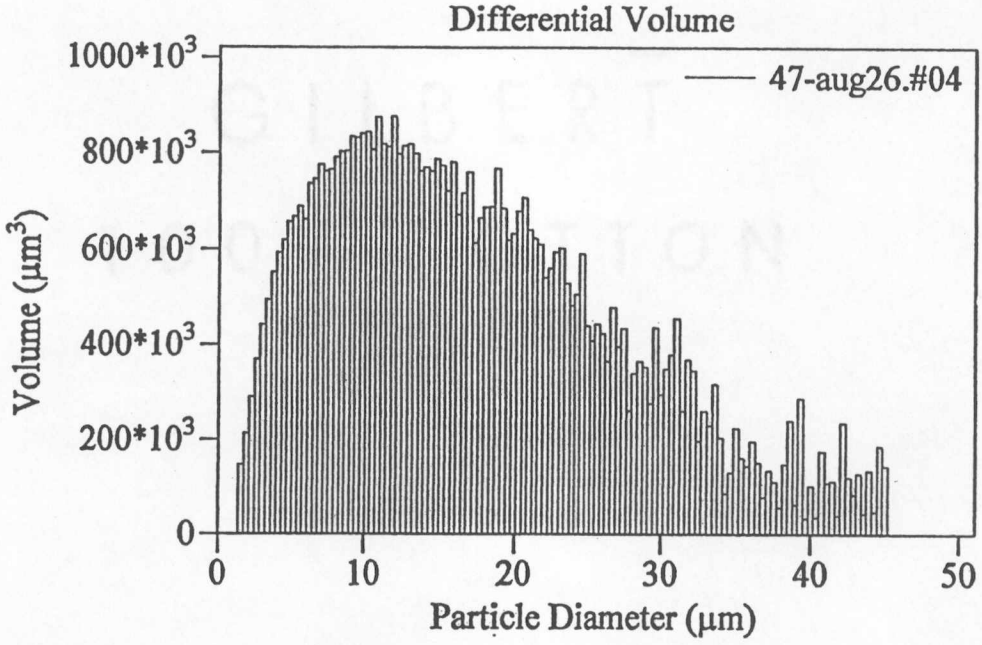
% <	10	25	50	75	90
µm	6.100	9.606	15.01	21.65	27.49

COULTER

AccuComp® 2.01

22 Sep 1999

File name:	47-aug26.#04	Sample Number:	1
Group ID:	Type I Portland cement		
Sample ID:	Aug26.#04		
Comment:	After the mill w/5% Delvo		
Operator:	NHA		
Raw Count:	509,299	Coinc. Corr. Count:	1
Acquired:	14:39 14 Jun 1997		
Background Run:	AUG26MSB.#04		



Volume Statistics (Arithmetic) 47-aug26.#04

Calculations from 1.436 µm to 41.28 µm

Volume:	55.65e6 µm³		
Mean:	17.00 µm	95% Conf. Limits:	16.97-17.03 µm
Median:	15.74 µm	S.D.:	9.248 µm
Mean/Median Ratio:	1.080		

% <	10	25	50	75	90
µm	5.653	9.543	15.74	23.29	30.61

APPENDIX D

MORTAR CUBE COMPRESSIVE STRENGTH TEST RESULTS

Compressive Strength Test Data for Trial Run Number 3 (W/C = 1.0)

Group I.D.	Sampled Date	Break Date	Age (Days)	Load in Lbs	Area (Sq. inch)	Strength PSI	Group Average	Remarks
N	2/17/99	2/18/99	1	1450	4	362.5	356	Type I cement
	"	"	"	1400	4	350		Non-Processed
P	"	"	"	1000	4	250	256	Type I cement
	"	"	"	1050	4	262.5		Processed
N	"	2/20/99	3	3500	4	875	919	Type I cement
	"	"	"	3850	4	962.5		Non-Processed
P	"	"	"	2400	4	600	550	Type I cement
	"	"	"	2000	4	500		Processed
N	"	2/24/99	7	5000	4	1250	1181	Type I cement
	"	"	"	4450	4	1112.5		Non-Processed
P	"	"	"	3300	4	825	881	Type I cement
	"	"	"	3750	4	937.5		Processed
N	"	3/17/99	28	8500	4	2125	2075	Type I cement
	"	"	"	8100	4	2025		Non-Processed
P	"	"	"	7200	4	1800	1756	Type I cement
	"	"	"	6850	4	1712.5		Processed

Compressive Strength Test Data for Trial Run Number 4 (W/C = 0.50)

Group I.D.	Sampled Date	Break Date	Age (Days)	Load in Lbs	Area (Sq. inch)	Strength PSI	Group Average	Remarks
1N	1/8/99	1/9/99	1	9300	4	2325	2350	Type I cement
	"	"	"	9500	4	2375		Non-Processed
1P1	"	"	"	6800	4	1700	1844	Type I cement
	"	"	"	7950	4	1987.5		Processed
2N	"	"	"	7800	4	1950	2006	Type I cement
	"	"	"	8250	4	2062.5		Non-Processed
2P1	"	"	"	8900	4	2225	2188	Type I cement
	"	"	"	8600	4	2150		Processed
1N	1/8/99	1/11/99	3	15050	4	3762.5	3631	Type I cement
	"	"	"	14000	4	3500		Non-Processed
1P1	"	"	"	13600	4	3400	3300	Type I cement
	"	"	"	12800	4	3200		Processed
2N	"	"	"	13000	4	3250	3188	Type I cement
	"	"	"	12500	4	3125		Non-Processed
2P1	"	"	"	12250	4	3062.5	3031	Type I cement
	"	"	"	12000	4	3000		Processed
1N	1/8/99	1/15/99	7	18200	4	4550	4575	Type I cement
	"	"	"	18400	4	4600		Non-Processed
1P1	"	"	"	16000	4	4000	3938	Type I cement
	"	"	"	15500	4	3875		Processed
2N	"	"	"	15650	4	3912.5	3913	Type I cement
	"	"	"	15650	4	3912.5		Non-Processed
2P1	"	"	"	15650	4	3912.5	3844	Type I cement
	"	"	"	15100	4	3775		Processed
1N	1/8/99	2/5/99	28	28500	4	7125	6688	Type I cement
	"	"	"	25000	4	6250		Non-Processed
1P1	"	"	"	26250	4	6562.5	6719	Type I cement
	"	"	"	27500	4	6875		Processed
2N	"	"	"	26000	4	6500	6625	Type I cement
	"	"	"	27000	4	6750		Non-Processed
2P1	"	"	"	23000	4	5750	5375	Type I cement
	"	"	"	20000	4	5000		Processed

VITA

Nabeel H. Abusadah was born in a little village called Allar in Palestine on November 1st, 1961. He was moved to the State of Kuwait when he was one month old, where he attended grammar, middle, and high school in the State of Kuwait Public School System. He graduated from Abraq Khitan high school. He immigrated to the United States of America in 1979. He attended Alabama A& M University and received his Bachelor of Science in Civil Engineering in December 1983. After graduating, he returned to Kuwait and joined the National Housing Authority as a Civil Engineer for about seven years. He returned to the USA just prior to the Gulf War and worked in Houston, Texas with consulting firm as a Construction Materials Engineer until joining the Tennessee Department of Environmental and Conservation. In 1994, he decided to pursue the Master of Science degree in Civil Engineering at the University of Tennessee. He, his wife, and their three children currently live in Knoxville, Tennessee.