DESIGNING LOST CIRCULATION PILLS FOR POLYMER BASED DRILL-IN FLUIDS

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DESIGNING LOST CIRCULATION PILLS FOR POLYMER BASED DRILL-IN FLUIDS

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ABSTRACT

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Specially designed non-damaging lost circulation pills (LCP) are being effectively applied for drilling depleted zones worldwide. Optimizing the LCP compositions stop the lost circulation effectively and protect the production zone from liquid and solids invasion significantly. Shape, particle size distribution and concentration of the lost circulation materials (LCM) are key parameters determining the effectiveness of LCP. In this study, the Permeability Plugging Apparatus (PPA) is utilized to evaluate effectiveness of various LCM's in curing the lost circulation. Sized calcium carbonates are used as LCM in different concentrations and in different particle size distribution. Lost circulation zones are simulated using the ceramic disks and slotted disks. Ceramic disks with nominal pore sizes 20, 35, 60, 90, and 150 microns are characterized in terms of pore size distribution using the computerized image analysis technique. Filter cake quality, spurt loss and filtrate volume are basic parameters to be evaluated in this study. Tests are performed at 75 F° and 300 psi of differential.

Key words: *Drilling, drilling fluid, drilling mud, lost circulation, lost circulation pill, lost circulation material, calcium carbonate, micronized cellulose, viscous pill*

ÖZ

POLİMER ESASLI RESRVUAR SONDAJ SIVILARI İÇİN KAÇAK TAPASI TASARIMI

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Formasyonu tahrip etmeyen özel olarak tasarlanmış kaçak tapaları dünyanın her yerinde basıncı düşmüş zonların delinmesinde etkin bir şekilde kullanılmaktadır. Kaçak tapalarının optimizasyonu kaçağı etkin bir şekilde durdurmakta ve üretim zonunu sıvı ve katı girişiminden dikkate değer bir şekilde korumaktadır. Kaçak kontrol malzemelerinin şekli, tane boyutu dağılımı ve konsantrasyonu kaçak kontrol tapasının etkinliğini belirleyen başlıca parametrelerdir. Bu çalışmada Geçirgenlik Tıkama Cihazı kullanılarak değişik kaçak kontrol malzemesinin kaçağı önlemedeki etkinliği değerlendirilecektir. Değişik boyutta ve konsantrasyondaki mikronize selüloz ve boyutlandırılmış kalsiyum karbonat kaçak maddesi olarak değerlendirilecektir. Kaçak zonları seramik diskler ve delikli diskler kullanılarak simüle edilecektir. Gözenek çapı 20, 35, 60, 90, and 150 mikron olan seramik disklerin gözenek boyut dağılımı bilgisayar destekli görüntüleme tekniği kullanılarak belirlenecektir. Kek kalitesi, ilk filtrat hacmi ve toplam filtrat hacmi bu çalışmada değerlendirilen başlıca parametrelerdir. Testler 75 F° ve 300 psi basınç farkında gerçekleştirilecektir.

Anahtar kelimeler: *Sondaj, sondaj sıvısı, sondaj çamuru, kaçak, kaçak tapası, kaçak kontrol malzemesi, kalsiyum karbonat, mikronize selüloz, viskoz tapa*

To My Family

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CHAPTER 1

INTRODUCTION

During the drilling of highly permeable, highly fractured and low pressured zones; loss of the drilling fluid because of the migration through formation is known as lost of circulation. During the last century, lost circulation has presented great challenges to the petroleum industry, causing great expenditures of cash and time to fighting the problem. Trouble cost for mud losses, wasted rig time, ineffective remediation materials and techniques, and in the worst cases – for lost holes side tracks, bypassed reserves, abandoned wells, relief wells and lost petroleum reserves have continued in this century. The risk of drilling wells in areas known to contain these problematic formations is a key factor in decisions to approve or cancel exploration and development projects.

Lost circulation problem is very commonly anticipated in various regions of Turkey (South-East West and South Anatolia).

As stated before, lost circulation is one of the most troublesome and costly problem encountered in drilling industry. Classical drilling process for drilling the highly fractured, low pressure and highly permeable formations is not only difficult but also expensive and risky. It has historically been one of the primary contributors to high mud cost. Pilehvari and Nyshadham (2002) has characterized lost circulation by a "Reduction in the rate of mud returns from the well compared to the rate at which it is pumped downhole during a lost circulation an appreciable part or the entire volume of drilling fluid can be lost into the formation. This may happen while drilling is in progress, due to excessive hydrostatic and annular pressure drop, or during trips, when pressure surges occur due to lowering of drill pipe or casing to the hole. ".

Mainly lost circulation occurs in one of two basic ways; invasion or fracturing.

Invasion occurs to the formations that are cavernous, vugular, fractured or unconsolidated. In the dynamic filtration process the pore invasion increases. That is because; the pressure differential increases, the fluid viscosity decreases because of the shear effect and mud cake erodes during trip and circulation. Fracturing is initiated and lost circulation occurs when some critical fracture pressure is reached or exceeded. Once a fracture is created or opened by an imposed pressure, it may be difficult to repair and may never regain the original formation strength. Lost circulation may persist even though the pressure is later reduced.

In some cases it is appreciable to reduce the amount of loss instead of loss of the entire volume during drilling. Lost circulation can be classified in three groups as; seepage loss (1-10 bbl/hr), partial loss (10-500 bbl/hr), and complete loss (over 500 bbl/hr) (Nayberg and Petty 1986).

There are plenty of studies made to solve the problem of lost circulation. At the present day, drilling industry is emphasizing underbalanced drilling (UBD) technology with increasing frequency. The reason is the advantage of reducing formation damage and minimizing lost circulation problem which reduce the production of oil and gas reservoirs.

Due to the inaccuracy of pore pressure prediction and the complex nature of water, oil, gas, and solid multiphase flow in the underbalanced system, it is very hard to remain in the underbalanced drilling condition. On the other hand, mechanistic models, rather than empirical correlations are being used for design of multiphase production system. Based on this trend of improvement, the application of mechanistic models to predict wellbore pressure and two-phase flow parameters seems to be the solution to increase the success of UBD operations by improving such prediction.

However in present day, underbalanced drilling technology needs special equipment and well educated drilling crew. Technical limitation during the appliance of this method is another disadvantage.

Because of reasons stated above, there are plenty of studies made for solving the lost circulation problems during classical drilling. Pumping LCM as discrete pills is one of the methods to combat lost circulation. Pumping lost circulation plug (LCP) functions, either by forming bridges or by increasing viscosity of the fluid to limit fluid migration in to the formation. There are plenty of lost circulation materials used in the industry; such as oil-soluble resins, fibers acid soluble particulates, graded salt slurries, high concentrated linear and cross-linked biopolymers and non-biopolymers (Samuel et al., 2003).

Bridging agents are often used to combat severe fluid loss, and calcium carbonate ($CaCO_3$) is the most commonly used. It is the most appropriate granular type of material because of its mechanical and chemical properties. $CaCO_3$ is resistant to pressure differentials and swap and surge impacts in the wellbore. Its acid solubility allows using it in producing zones. In this work, $CaCO_3$ will be tested as LCM with different particle sizes. The range of particle size distribution of LCM used in this study will be based on the range of materials available in market.

In this study compositions and rheology parameters are tested depending on; pressure-temperature difference, particle size, pore throat size of lost circulation zones.

This study is intended to conduct an experimental investigation on determining the methodology to be followed and optimum lost circulation pill design criteria to combat lost circulation problem anticipated while drilling the highly fractured, low pressure and highly permeable formations.

CHAPTER 2

LITERATURE REVIEW

Authorities estimated lost circulation affect up to 75% of all drilled wells. For drilling industry lost circulation is a critical issue because of economic reasons. Not only the fluid cost, but also its effects on drilling operations increase the cost of wells. The industry spends million of dollars a year to combat lost circulation and other effects on drilling operation like; non productive time, differential stuck, blow out and abandonment of expensive wells. Moreover, lost circulation has even been blamed for minimized production in that losses have resulted in failure to secure production tests and samples, while the plugging of production zones have to led to decrease productivity (Bugbee, 1953).

In 80's J. F. Gockel and M. Brinemann demonstrated a common misconception concerning the probability of the occurrence of lost circulation is based in the idea that if the mud weight does not exceed the fracture pressure of a given zone, no loss is likely to occur. For subnormal pressure zones it is not the right way of thinking. Subnormal pressure zones can occur naturally or can be the result of production depletion of a zone. In either case the fracture gradient can be normal, but the pore pressure is not able to withstand the equal circulating pressure of mud.

Commonly four types of formation are responsible for lost circulation can be seen on *Figure 1-1*:

a) High permeability unconsolidated sands and gravel
b) Cavernous or vugular zones in carbonates (limestone or dolomite).
c) Natural fractures, faults and transition zones in carbonates or hard shales
d) Induced fractures from extensive pressure.

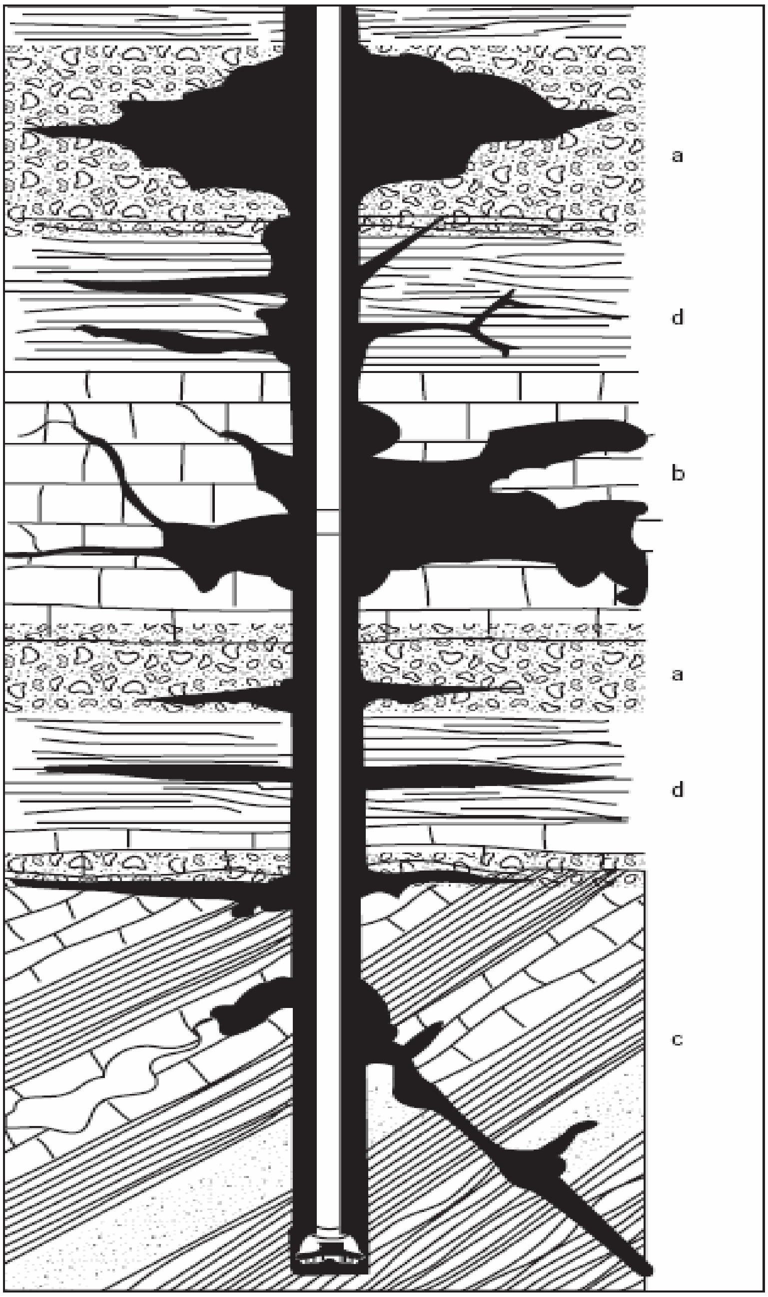

Figure: 2-1: Lost Circulation Sections

(MI Drilling Fluids Engineering Manual, Copyright 1998)

Unconsolidated sands and gravels have high permeability. Because of that, whole mud can invade through the formation. The high permeability resulting lost circulation can be present in shallow sands. In these type of formations lost circulation cause a cavity. Producing formations in the same field, especially in sands, subnormal pressure may be observed because of extraction of fluids. In such a case unless mud weight is controlled mud may invade to the formation. Moreover low pressure depleted formation can cause differential stuck. Another potential lost zone is cavernous or vugular zones with low pressure in carbonate or volcanic formations. Vugs are created due to the continuous water flow through dissolved part of the rock matrix in limestone. Caverns and vugs can also develop during the cooling of magma. The amount of lost circulation depends on the degree to which the vugs are interconnected. Mud loss also occurs to fissures or fractures where no coarsely permeable or cavernous formation exists. These fissures or fractures may occur naturally, or may be initiated or extended by hydraulically imposed pressures. The last potential lost zone is induced fractures occurring from extensive pressure. The reason of this type of fracturing is the exceeding of the critical pressure of the formation.

Especially, while drilling in depleted zones, lost circulation is the most probable and fatal problem. This is because of the greater length of formation exposed, the narrow operating window between pore pressure and fracture gradient (*Figure 2-2*). After fluid loss well control and stability problems can occur. Moreover if it is a productive zone fluid loss may cause productivity loss.

Loss of circulation into the productive zones is highly damaging. When invasion of the pay zones occurs, fluid-fluid and fluid- rock interactions are caused. These interactions are due to the invasion of mud filtrate, mud solids and in some cases, whole mud to the porous media. Solid invasion to the porous media can be classified into three main types: surface bridging, shallow plugging, and deep invasion. During the migration of fine solids through the rock, they begin to accumulate in the pore throats, forming an internal cake that irreversibly blocks the hydraulic flow channels. To avoid this internal blocking, it is necessary to create a surface mudcake in the pore in the near wellbore.

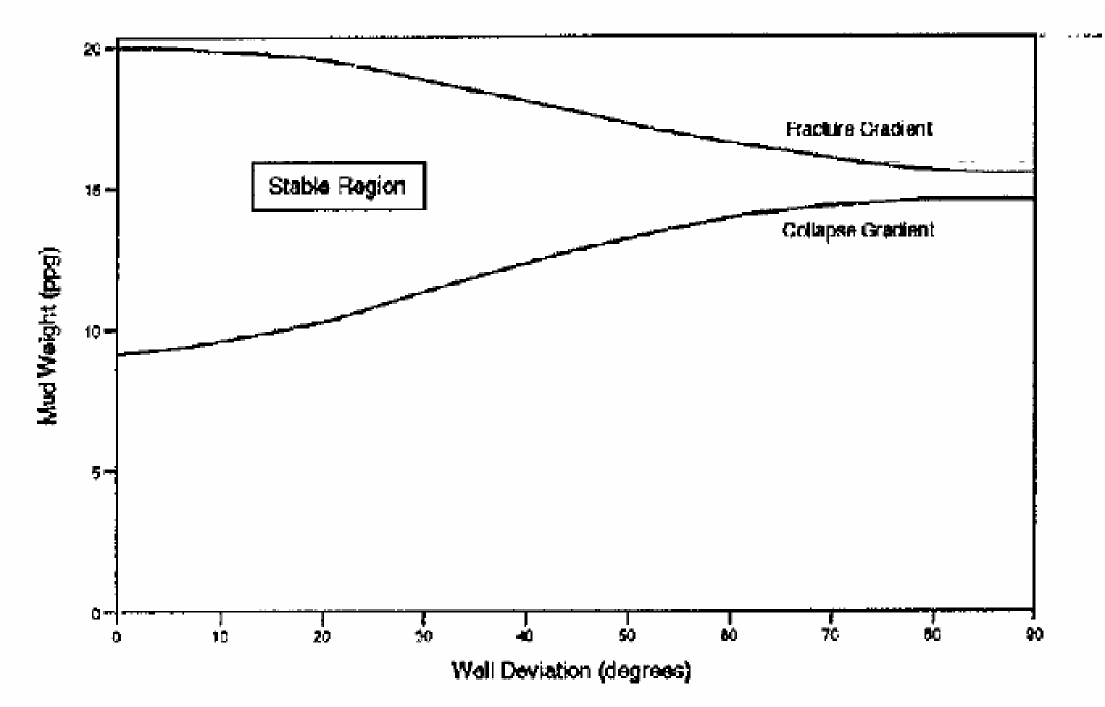

Figure 2-2: The influence of well deviance on wellbore stability (McLean and Addis, 1990)

J.C. Rojas et al.(1997) stated 7 options for controlling fluid loss which are related planning and execution stages of well;

Wellbore and drillstring geometry: Hole size and annular clearances have a big impact on frictional pressure loss. The sensitivity of equivalent circulating density on hole size, casing design and drill- string geometry should be determined at the planning stage of an extended reach well. It is also possible to use bi-centered bits to further increase the annular gap and hence reduce equivalent circulating density.

Casing design: Where possible it is advisable to select casing seats to minimize the exposure time of potential loss zones. Consideration should also be given to running drilling liners rather than full casing strings back to surface. This can help reduce equivalent circulating densities, although the benefit must be balanced against the potential downside of poorer hole cleaning in the wider annular gap created above the drilling liner.

Mud rheology and flow rate: Mud rheology and flow rate influence equivalent circulating density. The mud properties and flow rate need to be optimized to provide adequate hole cleaning as well as fulfilling the hydraulic requirements of downhole equipment (e.g. motors, MWD and bit nozzles), while at the same time minimizing equivalent circulating density.

Mud weight selection: The appropriate selection of mud weight is critical for extended-reach wells where wellbore stability issues exist. In practice it is often not possible to reduce equivalent circulating density by lowering the mud weight because of the increased risk of wellbore collapse.

Sealing capacity of drilling mud: The physical properties of the mud can influence the tendency to lose circulation. In particular the inclusion of sized bridging material can be used to control losses to high permeability formations and small fractures.

Hole cleaning efficiency: Effective cuttings transport is critical for extended-reach wells. Failure to transport cuttings effectively to surface will result in a cuttings accumulation in the annulus. This will further increase the ECD. In addition, cuttings beds which form on the low side of the hole will further restrict the annular gap. This will lead to higher frictional pressure drops in the annulus and hence higher circulating densities.

Lost circulation treatment: As well as treating the mud to reduce its potential for lost circulation, it is possible to pump LCM's in the form of discrete pills. These function either by forming physical bridges or by using increases in fluid viscosity to limit fluid penetration into the formation.

Most of them are related with stabilize and optimize equivalent circulating density and precaution for lost circulation. In spite of these options loss circulation may occur. Then lost circulation treatment should be applied. There are several methods to combat loss of circulation;

2.1 Cement Plug

Cement plug is one of the methods to combat lost circulation. Cement plugs are often effective against complete losses and severe complete losses. Especially in geothermal wells cement plug is the cure for lost circulation zones. Cement plus bentonite plug is also used in industry and it is capable of plugging vugular lost zones. Bentonite cement formed by adding cement to prehydrated bentonite. The slurry formed has lower density and higher gel strength.

The main handicaps of the cement plugs that it is acceptable for non productive zone losses. It is not able to wash out by these plugs any method. Its contamination with mud must be avoided.

2.2 Underbalanced Drilling (UBD)

Although at the present day, drilling industry is emphasizing underbalanced drilling technology, since 1970's significant progress has been made in understanding the nature of two phase flow in pipes and production systems, which can be seen as the base studies of underbalanced drilling. This progress has led to development of two phase flow models to simulate pipelines and wells under steady state as well as transient conditions. (Perez et al. 2002).

Underbalanced Drilling which is relatively considered as new sophisticated technology is progressing rapidly because of its advantages of reducing the formation damage, greater rates of penetrations, increasing bit life and minimizing lost circulation. It is basically maintaining wellbore pressure between boundaries depending on the formation pressure, wellbore stability and surface flow equipment (Alajmi and Schubert 2003).

The accuracy of UBD mainly depends on the prediction of wellbore pressure. The prediction is important for both designing the operation and predicting the changes during the operation. Whereas most of the approaches to predict wellbore pressure used in practice are empirical correlations and they have been shown to fail in predicting the correct pressure. Consequently, the current trend is toward increasing the use of prediction methods based on mechanistic models and may be the solution to increase the success of UBD operations.

However, UBD is not a reasonable solution for most of the wells dealing with lost circulation for now because of complex and unique nature of hydraulic systems of UBD operations, prediction of wellbore condition with damage mechanism and comprehensive planning stage which increase cost and safety issues.

2.3 Spotting LCM Pill

From years of experience fighting lost circulation LCM has an important role. LCM should be effective in sealing unconsolidated formations and fractures or vugs in hard formations. For different differential pressure conditions LCM should form a useful seal. During drilling, trips and casing runs swap or surge pressure would be applied. LCP should withstand both of this kind of effect and workable in oil-, synthetic or water-based system (Lummus, 1966).

Fluid-loss control is very important in successful well completion operations. It is best to avoid the use of fluid-loss control pills by incorporating mechanical fluid-loss control devices into the completion string whenever possible. However, in the absence or failure of such devices, or in situations where they cannot be used, chemical fluid-loss pills are required. The use of a pill is normally required before and after sand control treatments and after perforating. In these treatments, the pill is spotted into the perforations or against the sand control screens. In addition, fluid-loss control pills are required in several workover operations that need temporary zonal isolation.

One of the important features of any fluid-loss pill is its ability to maintain viscosity under bottom-hole conditions especially at high temperatures. The viscosity reduction of gel at high temperatures is either due to the degradation of polymer or reduced molecular interactions. The viscosity will not be regained on cooling if there is molecular degradation. (Samuel et al., 2003)

There are several types of LCM's used in drilling industry. They have different sizes, shapes, and characteristic. The use of LCM type is chosen by the influence of economic and technical limitations. For purposes of classification, LCM's can be divided in to fibers, flakes, granules and mixtures. The fibrous LCM's are used mainly in drilling mud to lessen mud loss through large face and within fractures or vugular formations, whereas flaky type LCM's can plug and bridge many types of porous formation to stop the mud loss or establish an effective seal over many permeable formations. The granular type of LCM's form bridges at the formation face and within the formation matrix, thus providing an effective seal, which depends primarily on proper particle size distribution to build a bridge having decreasing permeability, as it is being laid down. Finally blended LCM's are combination of granular, flake and fibrous materials that will penetrate fractures, vugs or extremely permeable zones and seal them off more effectively. (Pilehvari and V. R. Nyshadham ,2002)

Abrams in 1977 established the fundamental aspect of particle sealing technology and effect of mud physical properties and its effect on mud loss. The results of the study can be briefly stated as;

- Particle size: For effective sealing the mud should contain a wide range of particles

- Particle shape: It is not critical for sealing capability
- Particle concentration: The high concentration provides the better sealing.
- Particle density: Low density LCM can be used if mud weight resections exist
- Continuous Treatment: Having sealing agents continuously is more effective than spotting pills.
- Fracture Characteristic: Micro cracks are easy to seal than porous zones.
- Formation Damage: Calcium carbonate is preferable while drilling productive zones. Because of acid solubility.
- Economics: The lower cost of calcium carbonate, make its use preferable.

2.3.1 Cross-Linked Polymers

The definition of cross linking is the linking of to independent polymer by cross-linking agent. Chemically activated cross-linked pills (CACP) may be activated by cross-linking agent time and temperature and produce rubbery, ductile and spongy substance to plug lost circulation zones (*Figure 2-3*). CACP is used as plug to the lost of circulation zones. Operation time is controllable by using retarders or accelerators for the operating conditions.

Three generation of CACP is available. First and second generations carries fibrous type of LCM s whereas, third generation carry granular materials. All of three generation can be used with any water, oil or synthetic-base fluid systems. They can be squeezed to the lost circulation zones. The CACP should not be applied near production zones because they are not thermally and chemically degradable (Bruton, Ivan and Heinz, 2001).

Figure 2-3: Second generation CACP-firm, rubbery and ductile plug (Bruton, Ivan and Heinz, 2001)

2.3.2 Micronized Cellulose Fibers

In early 1980's micronized cellulose fibers *(Figure 2-4)* have become widely used as LCM worldwide for seepage or whole mud losses. They can be classified as flaky type of materials. Losses of great volume of fluids have been considered as acceptable in the history because of the thinking of it is not harmful for producing zones. Whereas nowadays it is better to achieve a rapid and near surface sealing, even if it is 100% damaging, at least minimize any invasion of fluids or solids (Verret et al., 2000).

Micronized cellulose fibers are flexible, highly compressible and swellable so that they have a rapid sealing characteristic than brittle and incompressible particles like calcium carbonates. The compressibility allows micronized cellulose fiber extrude into pore throats or fractures. They can form near surface seals and prevent damaging fluid and solid invasion into producing zones during drill-in, completion and workover operation. Near surface sealing of bulk micronized cellulose fibers can be removed by flushing the formation face with production.

Another advantage of micronized cellulose fiber is that, they have wide range of particle sizes. Moreover fibers tend to drag the slowest flow in dynamic conditions which allows to migration to lost zones and better sealing.

Figure 2-4: Micronized Cellulose Fibers
(Verret et al. 2000)

Micronized cellulose fibers have a limitation of usage in producing formation. Because of their lack of acid solubility and high sealing capacity micronized cellulose fiber may damage productive zones. However Veret et al. (2000) stated that micronized cellulose fibers are highly soluble (98-99%) in concentrated alkaline solutions which have proven to be as effective in removing cellulose fibers as conventional acid is removing calcium carbonate.

Moreover, alkaline solution increases the permeability of some types of formation like shales and clays.

2.3.3 $CaCO_3$

Bridging materials are often used in combat of massive lost circulation to the formation and formation damage because of fine solid invasion. Regular mud systems have large quantities of fine solids that invade to the formation causing damage in productive zones. The main components of these drilling muds are barite and bentonite. When barite invades the productive area an internal block within the formation is created which cannot be removed (Marqez 1996).

During formation of an external filter cake, especially fine solids are forced into the formation, building an internal filter cake. An internal filter cake plugs the near surface pore and reduces the formation permeability. Fine particles penetrate deeper into the pores and are not easily removed by back flushing. Invasion of larger particles is usually localized to near surface. Studies conducted by Bailey et al. (1999) show a strong correlation between invasion and damage. Because of that, minimizing of internal filter cake and quickly forming of external cake is very important for both fluid loss and formation damage control. A semi permeable slicker external filter cake can significantly reduce the invasion of the solids and the filtrate.

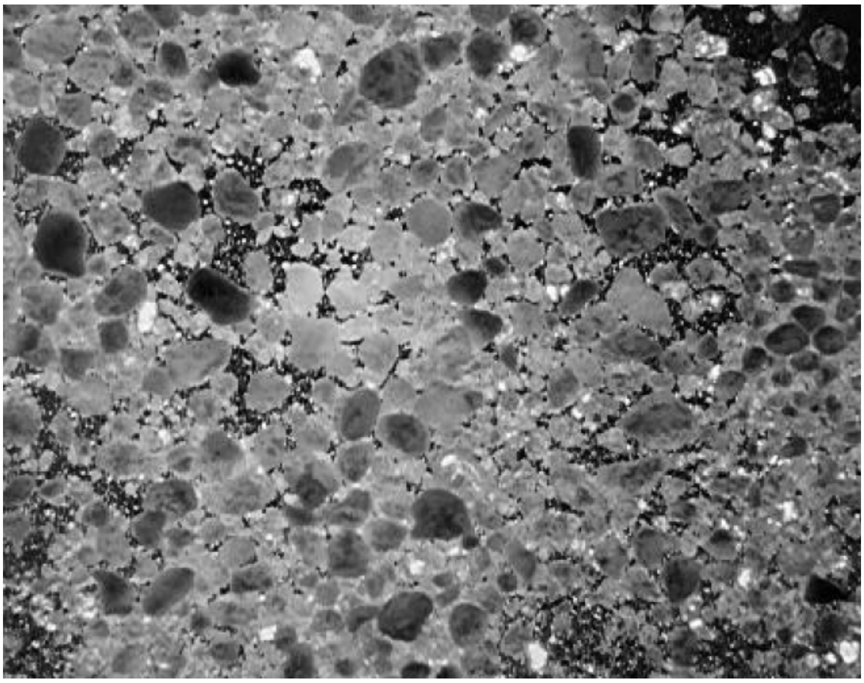

Figure 2-5: Calcium Carbonate
(Verret et al., 2000)

$CaCO_3$ (*Figure 2-5*) is the most commonly used, granular type of LCM's. It is a bridging material with proper mechanical and chemical characteristics to be utilized in the production zones. Its thermal and mechanical resistance makes the formed mud cake in the wellbore to have mechanical consistency that stands impact and high-pressure differentials. $CaCO_3$ particulates, if not removed will remain in the wellbore or formation and permanently impair the productivity of well. Hence, additional treatments should be applied to remove these particulates. Chemically, it is acid soluble so that it can be removed from the porous matrix to recover the permeability of the rock by HCl washes.

Salt pills are also granular type of material and they do not need additional treatment like acidizing unlike $CaCO_3$ pills. However they are less effective in controlling losses and are more difficult to design due to solubility issues (Rosato and Supriyono, 2002).

Correct fluid composition, bridging material selection and good maintenance of the drilling fluid are the keys to success for better productivity. Careful design is required to minimize spurt loss and solid invasion during drilling and completion operations.

There have been several studies with granular particles, especially $CaCO_3$, conducted to see the effect on lost circulation. G.E.Loeppke et al. (1990) studied high-temperature and fracture dominated loss zones. They stated that dimension of particle should be larger than the fracture if the dimension is normal for single particle bridging. J.C. Rojas et al. (1998) had discussed the effect of particle size and particle concentration of the $CaCO_3$ and concluded that for effective plugging of pores the mud should contain wide range of particles size and largest particles should be at least as large as the fracture width. They concluded that; high concentration provides better plugging. Cargnel and Luzardo (1999) conducted a detailed study on the particle size and concentration. This study was based on the study of Abrams' Median Particle-Size Rule (Abrams 1977). They concluded that as the range of particle size is between 1/7 and 1/3 of average pore size better sealing is performed which yields a small invasion of solids into the porous media and particle concentration is optimum at 25 lb/bbl for effective plugging. R.D. Cargnel stated that; "The predominant size of particles in the sample does not keep the geometric relationship to form a matrix that can avoid the filtration invasion. That way, a thicker cake is formed and, with the higher filtrate volume, the amount of particles in the cake is larger."

Nowadays, in horizontal wells open hole completions are frequently used. More attention needs to be paid to the cake forming properties of reservoir drilling fluids. Solid invasion is one of the primary causes of formation damage caused from drilling fluids.

Dick et al.(2000) proposed Ideal Packing Theory. Ideal that can be defined as the full range of particle size distribution required to effectively seal all voids, including those created by bridging agents' results in a tighter and less invading cake (Dick et al., 2000).The Ideal Packing Theory uses a graphical approach to determine the ideal size of particle range and optimum size distribution to better sealing. This theory expands the $D^{1/2}$ rule (Kaeuffer, 1973). $D^{1/2}$ rule and the Ideal Packing Theory states that ideal packing occurs when the percent of cumulative volume vs. the $D^{1/2}$ forms a straight-line relation ship, where $D^{1/2}$ is square root of the particle diameter. While Abrams' rule defines the particle size required to initiate bridging, the ideal packing theory defines the total particle range required to seal all pores, even those created by bridging agents.

CHAPTER 3

STATEMENT OF PROBLEM

Lost of circulation is one of the primary problems in drilling industry. There are several ways for treatment of lost of the circulation. Using LCM is the most popular way for treating lost circulation because of simplicity of usage and economic reasons. It is important to determine the type, composition, particle size, and the rheology of the fluid successfully.

The aim of this work is to determine a suitable composition of LCP, by using calcium carbonate, for fractured and high permeable zones. During the study different; pore sizes and particle sizes has been used. Because the ceramic is not well characterized in terms of pore throat size, these data are observed with microscope. The results have been compared and a suitable composition has been determined for loss circulation zones with different permeability.

CHAPTER 4

EXPERIMENTAL SET-UP AND PROCEDURE

4.1 Experimental Set-Up

During the experiments PPA is used as filtration device, the SEM is used to provide information about ceramic disks those are used as filtration medium, and LEICA microscope is used for particle size determination.

4.1.1 PPA

PPA (*Figure 4-1*). is a high pressure and high temperature (HPHT) filtration device is used in the experiments. Ceramic disks (*Figure 4-2*) are used as a filtration medium. They have wide range of permeability and porosities to simulate fractured zones accurately. The equipment's limitation is 5000 psi for pressure and 500 °F (260 °C) for temperature. If the test temperature exceeds 200 °F, back pressure must be applied to the cell to prevent boiling. For back pressure non-flammable, CO_2 is used. The filtration cell has a capacity of 300 ml. Hydraulic Pressure is applied to the cell by a hand pump. The filtration is collected out on the top. Collecting the filtrate out on the top avoid faulty readings due to the settling of particles during the static test from contributing to the build up of filtration cake.

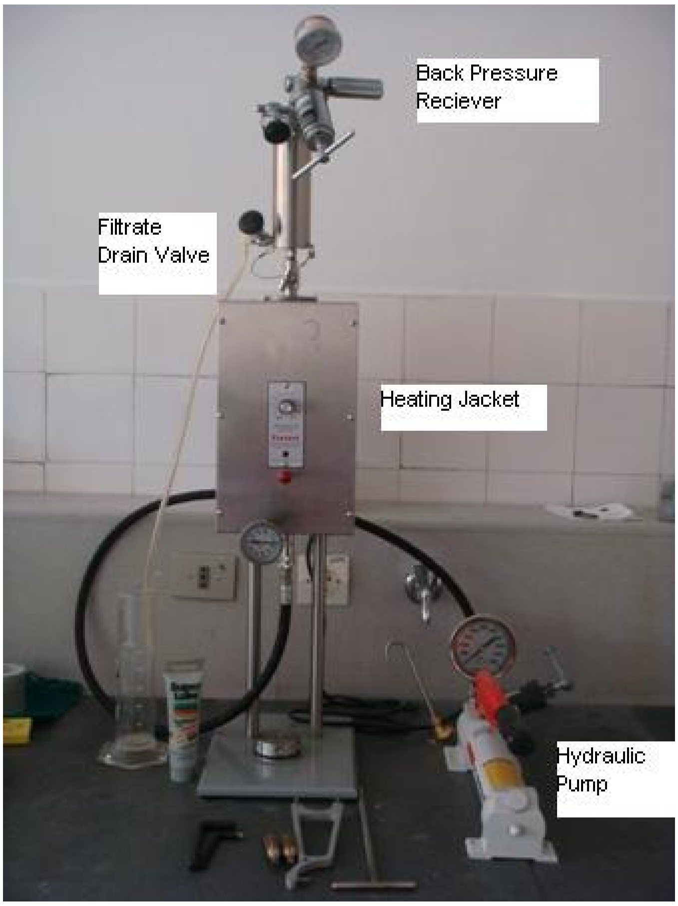

Figure 4-1: Permeability Plugging Apparatus (PPA)

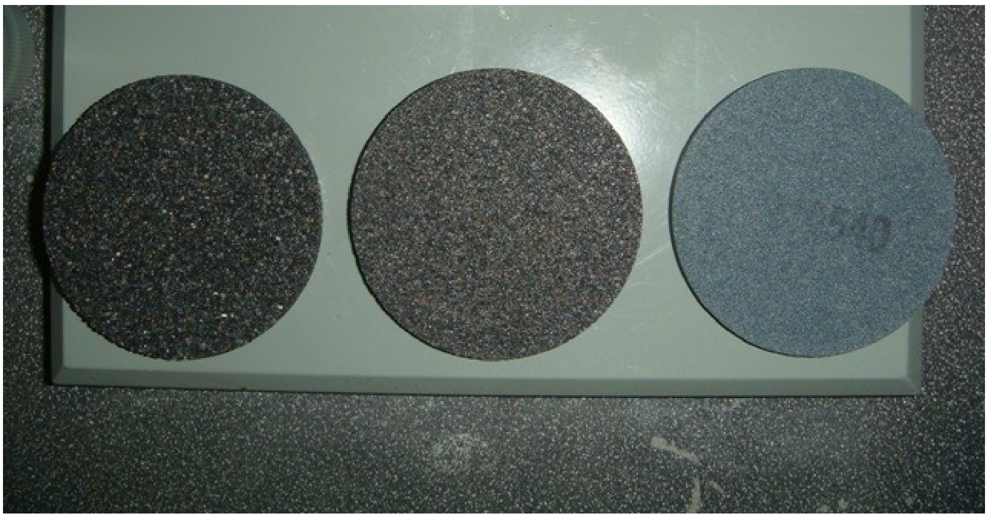

Figure 4-2: Ceramic Disks (left to right 35µ, 60 µ, and 90 µ)

4.1.2 SEM (Scanning Electron Microscope)

The SEM is used to provide information about; particle size distribution and shape of lost circulation materials and pore throat size distribution of the ceramic disks used to simulate formation. It functions by projecting a beam of electrons through magnetic focusing lenses at a specimen and recording secondary electrons excited by the primary beam. The amount of secondary electron emission portrays the topography of the specimen because more secondary electrons are emitted from high points than from low points.

The property of the SEM that is important in this study is its great depth of focus. The depth of focus results from using electrons rather than light.

Figure 4-3: Scanning Electron Microscope (SEM)

4.2 Experimental Procedure

In experimental work there are several steps need to be followed. First is to find particle sizes of LCM and pore size distributions of the ceramic disks, to determine the combinations of LCP and ceramic disks combination for experiments. Following this step LCPs' are prepared and permeability plugging tests are run. Finally the ceramic disks and filtration tests results are examined to see the effect of the selected LCP and pore sizes. For particle size and pore throat size determination, SEM is used to take photographs of both ceramic disks and $CaCO_3$. These photographs are analyzed in the Image J software (http://rsb.info.nih.gov/ij/download.html; accessed at October 2008) to discover the particle sizes for $CaCO_3$ and pore throat sizes for ceramic disks. For permeability plugging tests, PPA is used.

4.2.1 Determination of Pore Throat Size

Determination pore throat size of ceramic disks is consisting three steps. In the first step pore size distributions of ceramic disks are evaluated. By using SEM photographs of ceramic disks are taken (Figure 4-4).For this purpose thin sections of the ceramic disks of 3 cm diameter are prepared in polyester resin. The samples are grounded on a steel lap until they appeared flat, then transferred to a Granton-Vander-bilt polishing machine for final polishing with diamond abrasive. The samples are cleaned to remove all abrasive materials from the pores.

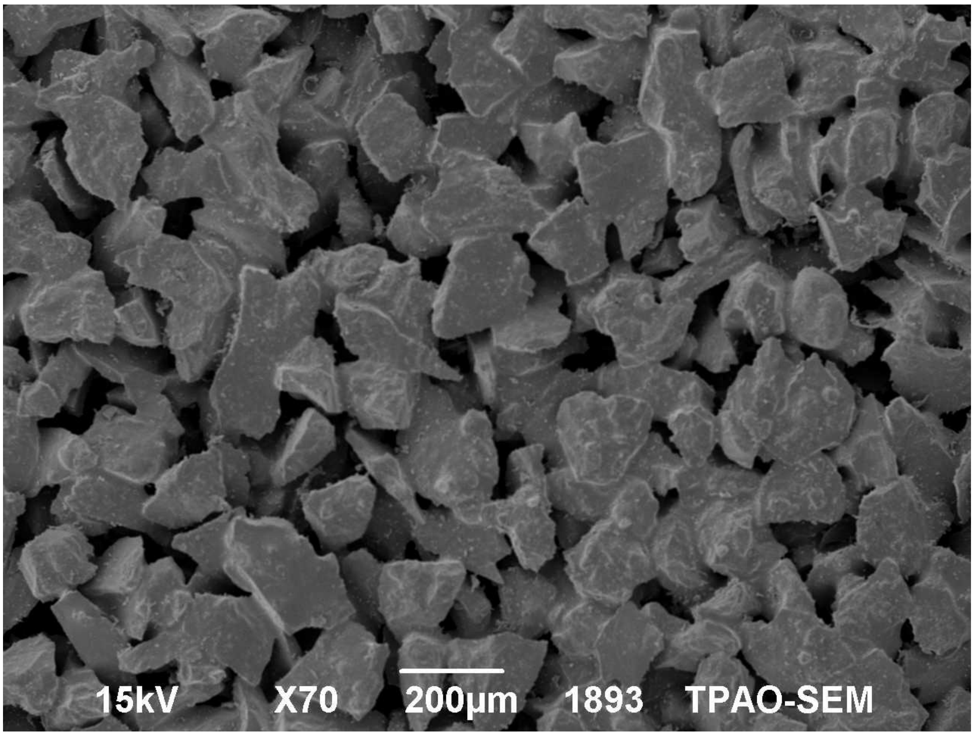

Figure 4-4: SEM photograph of 35 µ Ceramic Disk 70 X Focus

Than the photographs is analyzed using Image J software by adjusting threshold option. (Figure 4-5).The areas of the pores (black areas) are evaluated by software.

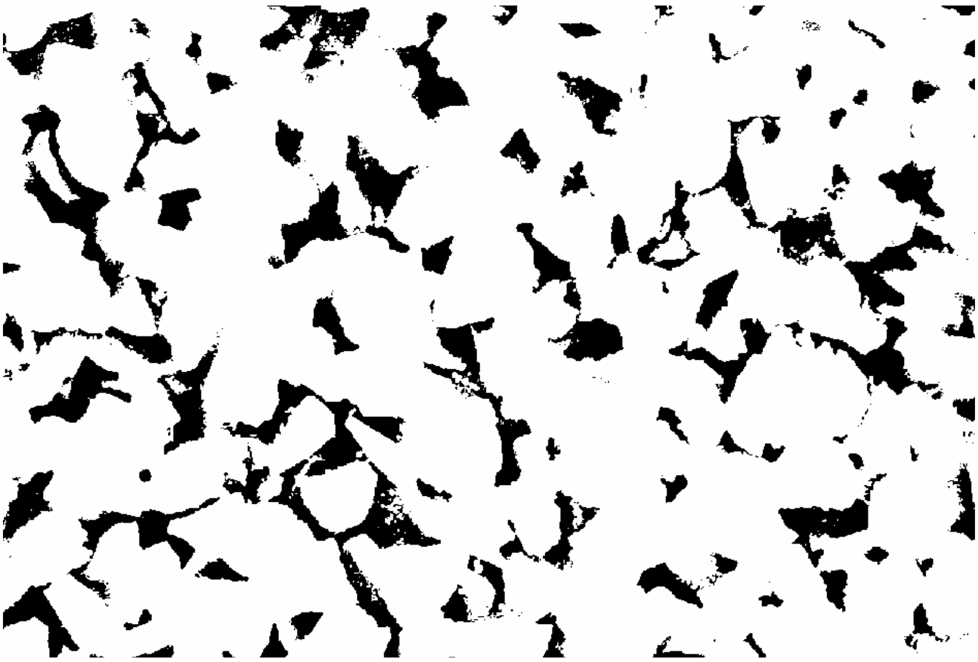

Figure 4-5: Areas of Pores for 35 µ Ceramic Disk 70 X Focus

Finally, diameter of each pore has been calculated assuming that a pore has a circular body. These data are recorded as mean pore size diameters. The minimum pore sizes are evaluated through Image J. Both data are used to plot histograms of the pore throat sizes. The figures can be seen on Appendix A for ceramic disks of different pore sizes. Both mean and minimum pore throat sizes are calculated. The results show a lack of consistent base line data matching the manufacturer's specs.

4.2.2 Determination of Particle Size

Same procedure in chapter 4.2.1 is followed for determination of particle size distributions of $CaCO_3$. By using LEICA microscope, photographs of $CaCO_3$ are taken. The photographs are then analyzed using Image J software to evaluated the particle size distributions using the aforementioned procedure. The results can be seen on chapter 5.1 for different size of $CaCO_3$. Finally particle size distribution graphs is plotted.

4.2.3 Preparing LCP

Solids free viscous pills are used in the experiments. FLO-PRO^{TM} is used for LCP. The LCP is prepared with 350 cc pure water, 1 lb/bbl XCD polymer, 4 lb/bbl modified starch and of $CaCO_3$. First, XCD polymer and modified starch are added to the fresh water and mixed for 30 minutes. $CaCO_3$ is added and mixed for 5 more minutes. Hamilton beech mixer is used for preparation of LCP.

4.2.4 PPA Filtration

After preparing LCP, PPT is run;

- Specific ceramic disk is placed in the PPA cell and pour 275 ml of LCP into the cell.
- Adjust the pressure to 300 psi and temperature to 75 F°.
- If the test is run above the boiling point the back pressure must be applied.
- Collect filtrate for 30 minute. Record the reading at 7,5 minute as spurt loss $EV_{7,5}$ and after 30 minute as total volume EV_{30}.
- Disassemble the cell and remove the ceramic disk.

After finishing the PPT estimation filtration parameters can be calculated (Davis et al., 1999) from the data collected at 7,5 and 30 minute intervals. Spurt loss Total filtration and static filtration parameters are calculated by using the equations below:

$$\text{Spurt Loss, ml} = 2 \times [EV_{7,5} - (EV_{30} - EV_{7,5})] \tag{4.1}$$

$$\text{Total Fluid Loss} = 2 \times EV_{30} \tag{4.2}$$

$$\text{Static Filtration Rate mL/min}^{1/2} = 2 \times (EV_{30} - EV_{7,5}) / 2{,}739 \tag{4.3}$$

CHAPTER 5

RESULTS AND DISCUSSION

Petrophysical characterization and pore geometry determination of the rock are important step while determining the pore size distribution of bridging material. The main scope is minimizing the invasion. The Drill-in fluid should contain bridging solids of a specific particle size distribution that is able to plug for the formation of different pore sizes. As said before $CaCO_3$ is the most commonly used granular bridging material. There are various rules in the industry. Some of them are stated in chapter 2. These rules of thumbs are used to choose the particle size of bridging materials that can form an efficient external filter cake and minimize formation damage.

- A median particle size of bridging additive equal to or slightly greater than one third of the median pore size of the formation (Abrams, 1977).
- The concentration of the bridging agents must be at least 5% by volume of the solids in the final mud mix (Abrams, 1977).
- The D90 of the particle size distribution of the bridging agents should be equal to the pore size of the rock (Hands et al., 1998).
- D 1/2 rule states that ideal packing occurs when the percent of cumulative volume vs. the D 1/2 forms a straight line relationship, where D1/2 is square root of the particle diameter (Kaeuffer, 1973; Dick et al., 2000).

5.1 LCM Size Distribution

Three different sizes of $CaCO_3$ are used. These commercially available $CaCO_3$s' named as $CaCO_3$ A, B and C. Particle size distributions (PSD) of $CaCO_3$ have been determined by analysis through Image J software by using photographs taken from LEICA microscope.

Figure 5-1: Photograph of $CaCO_3$ B 40 X

By using threshold analysis areas of the particles in two dimensions has been evaluated. Diameters of each particle have been calculated through equation 5.1.

$$AREA = \pi \frac{D^2}{4} \tag{5.1}$$

By using the data gathered, cumulative particle percentage vs. particle size graphs have been plotted. Particle size distribution graphs of the selected $CaCO_3$

are shown in figures 5.2, 5-3, 5-4. The S-shape curves indicate the range of particle presents.

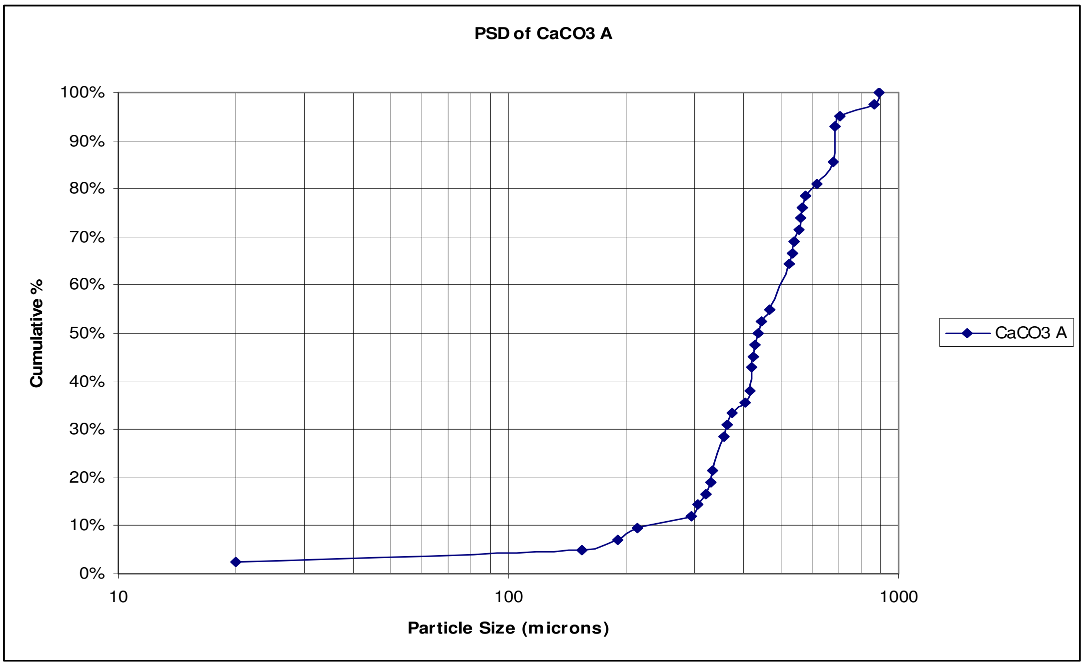

Figure 5-2: PSD of $CaCO_3$ A

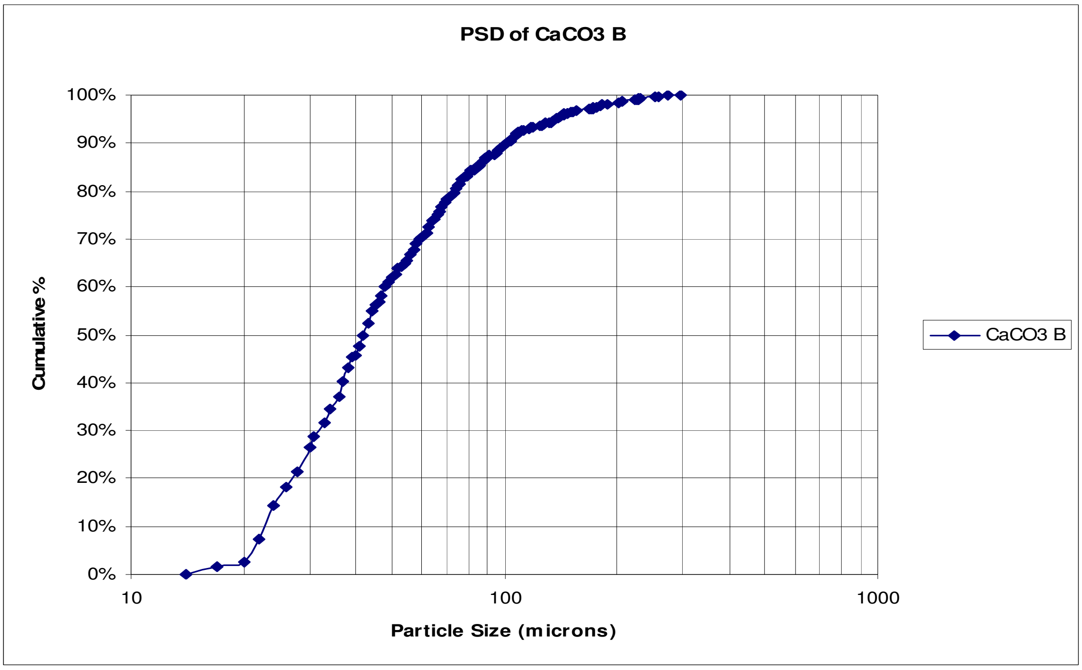

Figure 5-3: PSD of $CaCO_3$ B

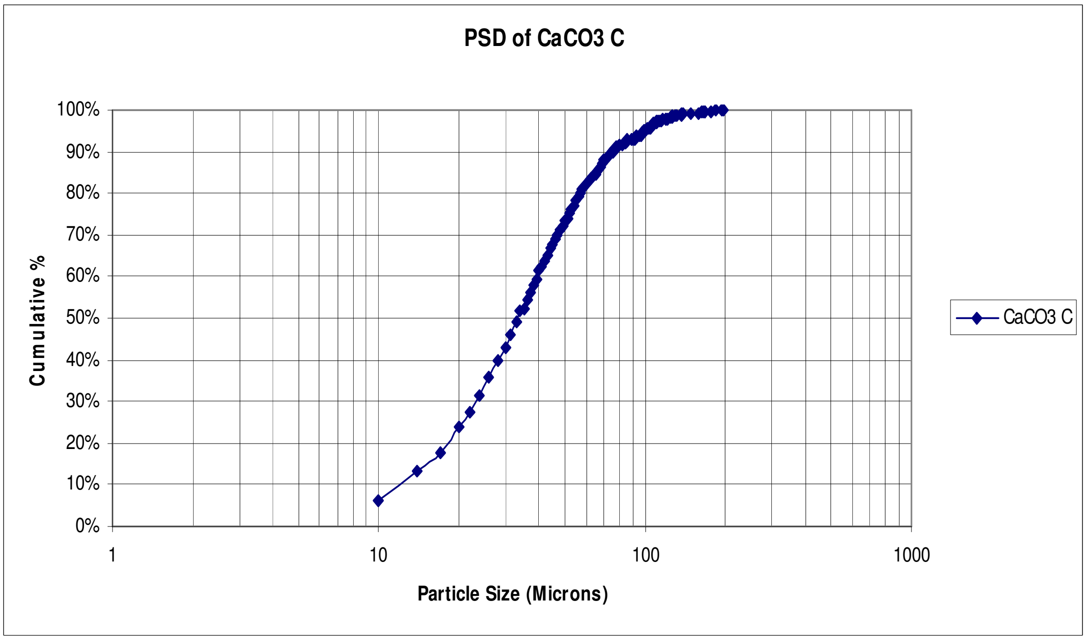

Figure 5-4: PSD of $CaCO_3$ C

The cumulative particle size distribution of selected $CaCO_3$ is shown on Table 5-1.

Table 5-1: Particle Sizes of Calcium Carbonates

	D90 (μ)	D50 (μ)	D10 (μ)
$CaCO_3$ A	688.5	438.3	291.6
$CaCO_3$ B	102.0	42.4	23.8
$CaCO_3$ C	76.0	33.6	13.7

As stated before derivation of D_{10}, D_{50} and D_{90} values are used in test matrix preparation. After PPT, their effects are studied for analyzing the relation between pore size distribution of ceramic disks and particle size distribution of LCM. The test results obtained from PPT and the post study with SEM will also be helpful in this analyses.

5.2 Pore Throat Size Distribution

There are presently many types of filter medium for PPA. In field usually ceramic disks are used. In this work also ceramic disks are used too. However, Davis et al. (1999) stated the concerns over the use of ceramic disks because of considerable variability between disks' pore throat sizes. It is viewed that a lack of consistent base line data, particularly for the coarser discs, may be due to random variations in pore size distributions and permeability of disks which make matching sizes to a given formation and repeatability of the tests difficult. Finally, no directions were issued by the PPA manufacturers with regard to the use of alternate filtering media.

By using SEM and Image J software, pore size distributions of ceramic disks are analyzed which was stated at Chapter 4. By using the SEM, photographs of the ceramic disks have been taken at varied focuses to check the consistency.

These photographs are analyzed by Image J and the parameters are calculated as; area, perimeter and minimum pore throat sizes. The study is based on several steps. In the first step mean diameters and their relationship will be discussed. In the second step minimum diameters will be discussed. Mean while rules of thumbs that are stated above, will be compared.

In computation stage of Image J software, several assumptions have been made. First assumption was used for calculating mean diameter of pore throats that is the pores are circular. Mean diameters are calculated from area. Second assumption was that the ceramic disks do not have double porosity and pore sizes should become dense at a particular point. By using threshold analysis areas evaluated. Using the histograms and the assumptions written above noise values has been removed and the actual mean diameters have been calculated.

Ceramic disks are used for simulating formation are 20 μ, 35 μ, 60 μ, 90 μ, 150 μ and 190 μ pore throat size. Histograms are re-plotted after removing noise data. Pore geometry determined by image processing, and the results can be seen on Appendix A.

Pore size values are shown on Table 5-2.

Table 5-2: Ceramic disks pore throat size distributions

20 µ Ceramic Disk			
	D90 (µ)	**D50 (µ)**	**D10 (µ)**
50X Focus (mean)	76,55	29,78	11,68
70X Focus (mean)	52,03	25,74	13,1
100X Focus (mean)	63,94	25,91	11,71
50X Focus (min.)	51,88	22,08	7,55
70X Focus (min.)	37,59	18,66	8,37
100X Focus (min.)	39,04	20	8,35
35 µ Ceramic Disk			
	D90 (µ)	**D50 (µ)**	**D10 (µ)**
50X Focus (mean)	101,5	41,21	14,8
70X Focus (mean)	103,43	41,86	15,38
100X Focus (mean)	77,22	41,62	23,3
50X Focus (min.)	72,11	28,59	9,45
70X Focus (min.)	75,83	30,04	10,83
100X Focus (min.)	60,47	29,95	15,1
60 µ Ceramic Disk			
	D90 (µ)	**D50 (µ)**	**D10 (µ)**
50X Focus (mean)	150,69	70,17	13,02
70X Focus (mean)	150,66	75,44	11,75
50X Focus (min.)	111,71	45,14	9,1
70X Focus (min.)	119,19	50,35	7,83
90 µ Ceramic Disk			
	D90 (µ)	**D50 (µ)**	**D10 (µ)**
30X Focus (mean)	252,73	96,88	37,91
30X Focus (min.)	176,82	65,54	25,46
150 µ Ceramic Disk			
	D90 (µ)	**D50 (µ)**	**D10 (µ)**
16X Focus (mean)	361,74	179,24	68,59
16X Focus (min.)	258,43	124,23	41,57
190 µ Ceramic Disk			
	D90 (µ)	**D50 (µ)**	**D10 (µ)**
16X Focus (mean)	491,19	182,07	51,66
16X Focus (min.)	389,92	140,3	41,73

5.3 Permeability Plugging Tests

In order to show the effect of $CaCO_3$ on filtration properties, PPA filtration tests were conducted. 65 experiments in total were carried out during this study. Tests are conducted at 75 F° of constant temperature and 300 psi of constant differential pressure. The results can be seen on Appendix B.

PPT 7.5 min. = Total Filtrate collected in 7.5 minutes

PPT 30 min. = Filtrate collected in 30 minutes

PPT illustrative equipment for studying the effect of lost circulation plugs. The values shows repeatability and plugging effects also can be seen.

Spurt loss is most important parameter for indicating the plugging performance of the LCM. It indicates the rate of plugging. For LCM a rapid sealing characteristic is needed. Total filtration and static filtration rate mostly reveals the quality of semi-permeable filter cake.

The result of the tests is examined in two subtitles. The first one is the effect of concentration variation on plugging capability of the LCP. Than using the PSD and pore throat sizes their size relationship is investigated. Meanwhile rules of thumbs also are evaluated during the observation of the test values.

After PPT some of the ceramic disks have been photographed by SEM to investigate the plugging quantity of LCP. Best filtration result of 60 μ ceramic disk is shown on figure 5-5 and 5-6.

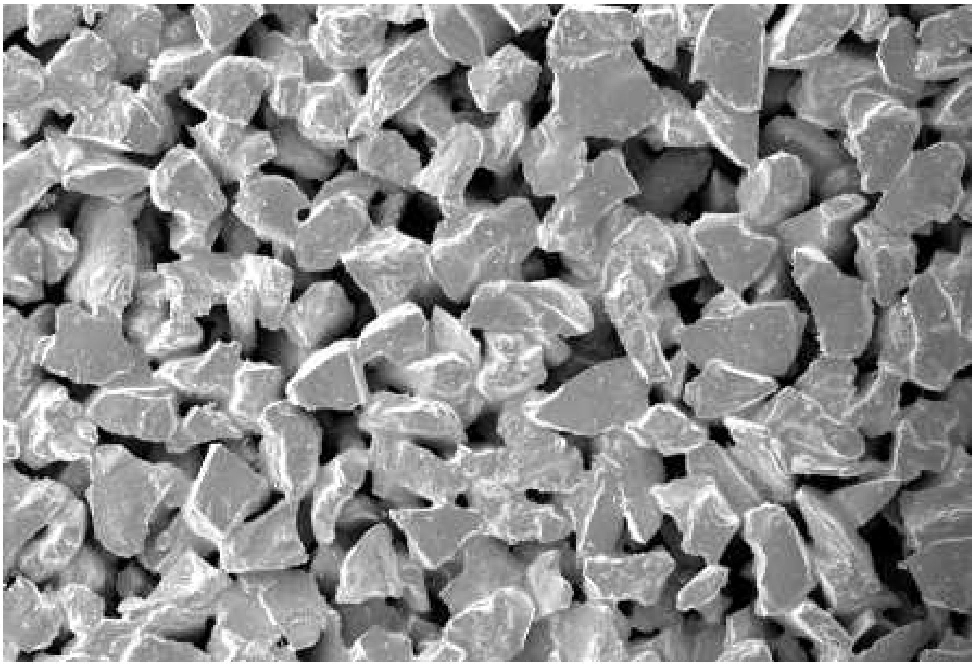

Figure 5-5: 60 μ Ceramic Disk before PPT

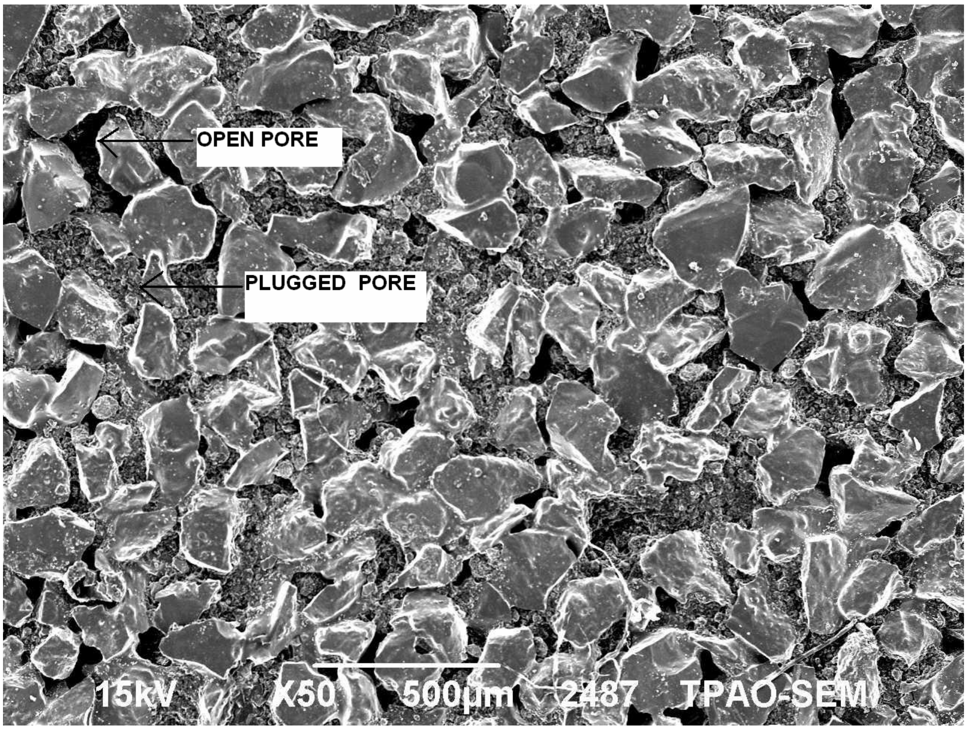

Figure 5-6: 60 μ Ceramic Disk after PPT (30 lb/bbl of $CaCO_3$ C)

5.3.1 Effect of $CaCO_3$ concentration

For determining the effect of the $CaCO_3$ concentration, bridging material was varied from 10 lb/bbl to 60 lb/bbl for different ceramic disks. The properties of the fluids are shown on table 5-3.

Table 5-3: Properties of Lost Circulation Plugs

	$CaCO_3$ C (10 ppb)	$CaCO_3$ C (20 ppb)	$CaCO_3$ C (30 ppb)	$CaCO_3$ C (40 ppb)	$CaCO_3$ C (60 ppb)	$CaCO_3$ B (30 ppb)
PV, cP	7	7	7	7	9	8
YP, lb/100ft^2	15	16	17	16	17	17
Gel 10 sec. lb/100 ft^2	8	8	10	9	9	9
Gel 10 min. lb/100 ft^2	10	10	12	11	11	11
Density, ppg	8.5	8.65	8.8	8.9	9.2	8.8
API Filtrate, ml	7.8	7.2	7.4	7.6	7.8	7.2
pH	8	8	8	8	8	8

Concentration of $CaCO_3$ has a significant effect on spurt loss and total filtration and static filtration rate. The test results are demonstrated by spurt loss, total filtration and the static filtration rate vs. concentration graphs for pore sizes and bridging materials. The results are shown on figures 5-7, 5-8 and 5-9. The test of 90 µ ceramic disk and 10 lb/bbl of $CaCO_3$ B failed in plugging. The reason is the concentration of particle sizes above pore throat size is 90%.

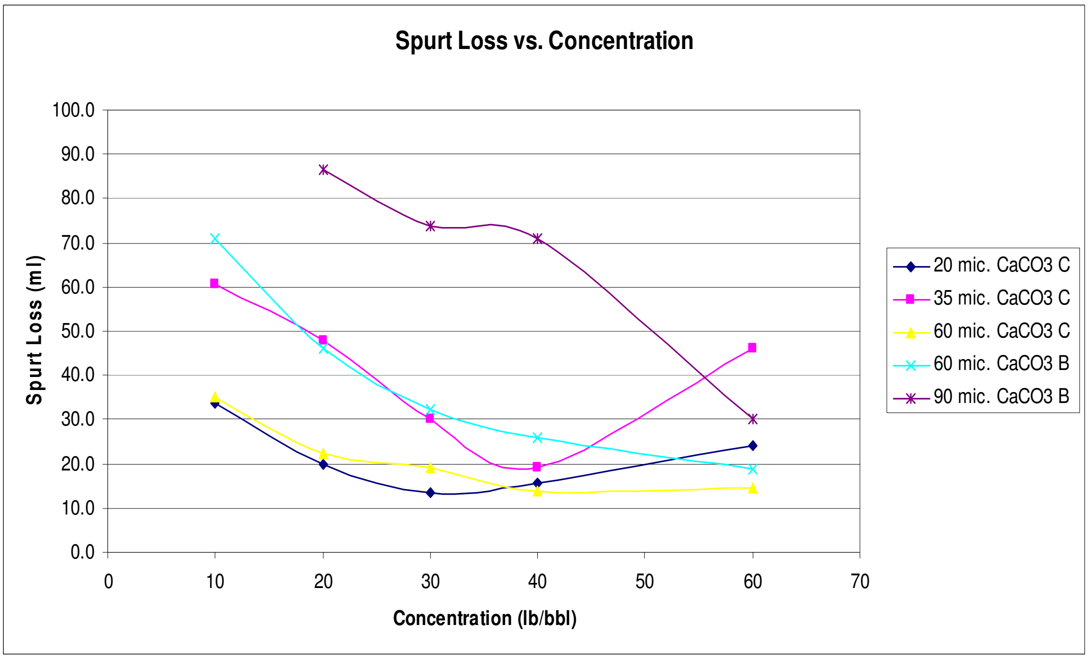

Figure 5-7: Spurt Loss vs. Concentration

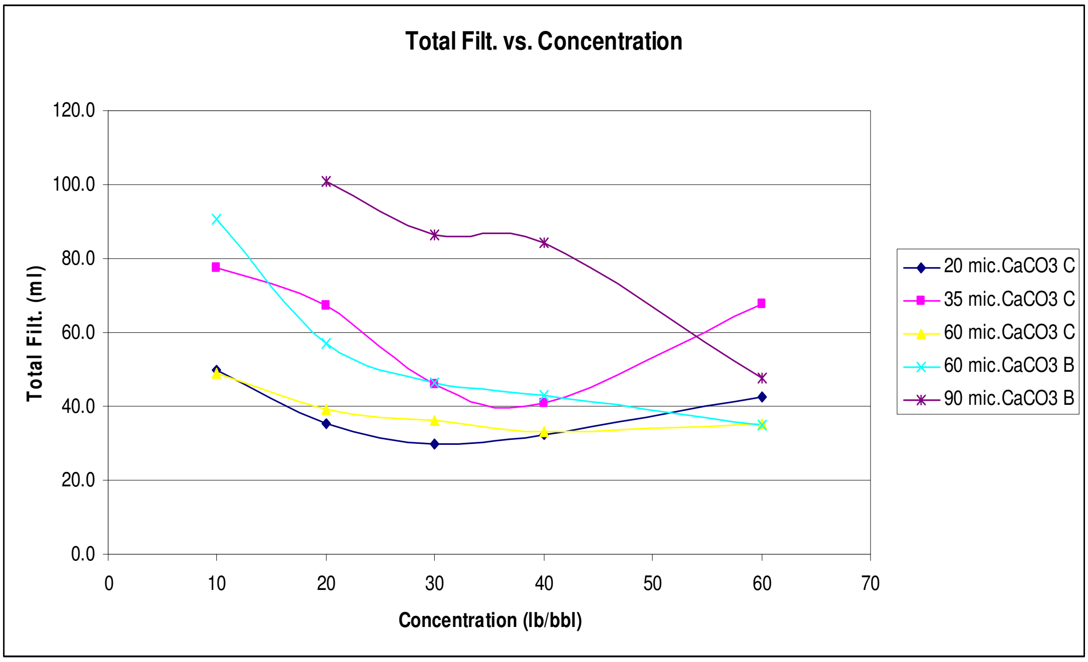

Figure 5-8: Total Loss vs. Concentration

For 60 µ and 90 µ ceramic disks, the higher concentration has better sealing capacity moreover as the pore size increases effect of concentration is clearer.

On the other hand, for 20 μ and 35 μ ceramic disks filtration parameters increases as the concentration of the bridging material is increased from 30 lb/bbl to 60 lb/bbl. The reason is the particle size of the $CaCO_3$ is bigger than the mean pore size diameter of ceramic disks. From the Figure 5-2 and Figure 5-3 it can be seen that, 36.8% of $CaCO_3$ C larger than 35 μ mean pore throat size. This is 62 % for 20 μ. From the graph it can be seen that changes in concentration has influence on 35 μ ceramic disk more than 20 μ. It is because 64.2 % of particles are smaller than mean pore size where as it is 38 % in 20 μ disk.

Increasing the concentration 30 lb/bbl to 60 lb/bbl higher spurt loss and total filtration are observed. These values verifies the statement of Cargnel (1999) "The predominant size of particles in the sample does not keep the geometric relationship to form a matrix that can avoid the filtration invasion. That way a thicker cake is formed and with the higher filtrate volume the amount of particles deposited in the cake is larger." Thicker cake is seen for the entire test with increasing concentration.

For 90 μ ceramic disk total loss is observed for all concentrations of $CaCO_3$ C and 10 lb/bbl concentration of $CaCO_3$ B. When particle sizes and pore throat sizes are compared for 90 μ ceramic disk and $CaCO_3$ C; the cumulative amount of bridging materials is 5.7% which exceeds 97 microns which is the median pore size of ceramic disks. Detailed consideration of the particle size effect will be evaluated at section.

The test results of $CaCO_3$ B and 90 μ ceramic disk reveals that; not only the choosing the suitable particle size, but also appropriate concentration has an important role in plugging the lost circulation zones especially for coarser pore sizes. For all of the ceramic disk reduction of concentration from 30 lb/bbl to 20 lb/bbl, an increase in total filtration and spurt loss is occurred. The deposition of thin cake also shows the lack of bridging particles to form a suitable cake.

As the static filtration rates are observed an increase is observed till 30 lb/bbl of concentration is exceeded. The reason is same as spurt loss and total filtration that a thicker filter cake is formed.

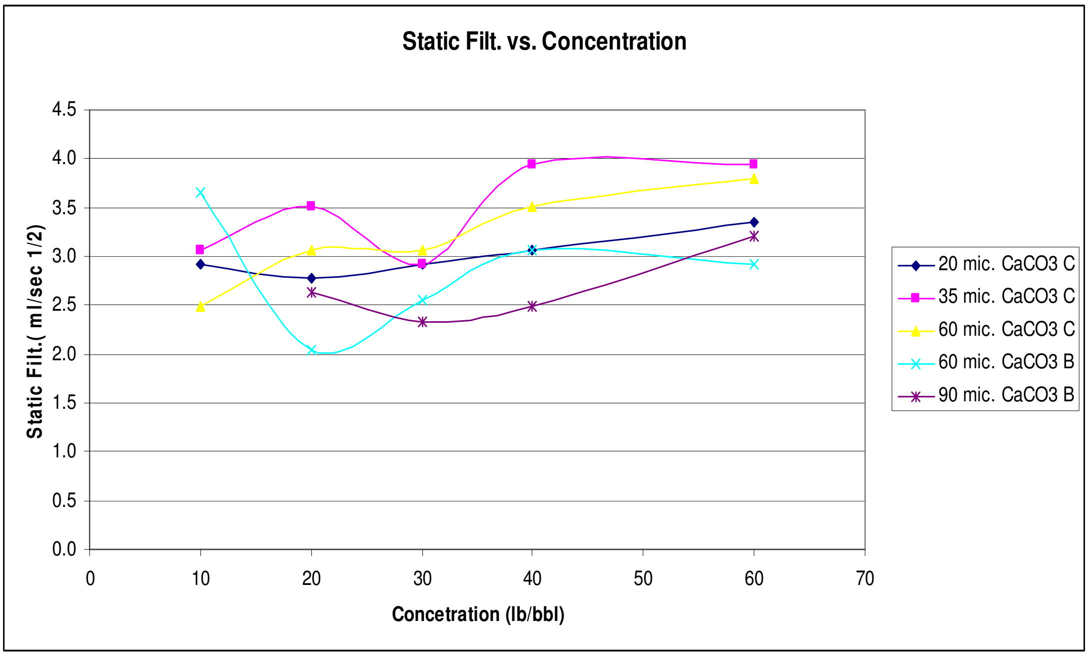

Figure 5-9: Static Filtration vs. Concentration

Except for 90 μ and $CaCO_3$ B, 30 lb/bbl of bridging agent concentrations are appropriate for spurt loss, total filtration and static filtration rate for 20, 35 and 60 micron ceramic disks. Thin, impermeable mud cake is formed. Although it does not provide lowest values of filtration, it is the optimum amount when filter cake properties and static filtration rates are considered. Because a small amount of decrease in filtration is observed while increasing the concentrations from 30 lb/bbl to 40 lb/bbl. So, 30 lb/bbl concentration is the optimum concentration for these cases.

For 90 μ ceramic disk on the other hand 60 lb/bbl concentration should be preferred for rapid sealing in spite of an increase in static filtration rate. The lowest spurt lost and total filtration is observed for this particle size distribution of the bridging material.

By assuming a specific gravity of $CaCO_3$ as 2.7 concentration of bridging materials in LCP s' calculated as:

$$\text{Solid \%} = \frac{Vsolid}{Vfluid} \times 100 \tag{5.2}$$

$$Vmud = \frac{m}{\rho} \tag{5.3}$$

By using the equations, 10 lb/bbl concentration is equal to 1% of bridging material by volume in mud mix. The test results of 20 μ, 35 μ and 60 μ ceramic disks show that 4 % of bridging material by volume is suitable and at least 2% is needed for a semi-permeable filter cake. For 90 μ ceramic disks the particle concentration of 6% shows better results.

Though it can be said that; it is not necessary to have at least 5% of bridging materials by volume in the final mud mix for all cases as Abrams (1977) stated before. But the suitable concentration of the bridging materials for effective sealing is mostly depends on the characteristic of the formation and bridging materials used.

5.3.2 Effect of $CaCO_3$ size

Equivalent diameter of pore throats is calculated from area that is derived from image J software by assuming the pores are circular. Minimum diameters are measured by image J software. The results have been gathered from image analysis ceramic disks and particle size distribution of $CaCO_3$. The following results have been derived.

35 μ & 20 μ ceramic disks: Cumulatively 37.5 % of $CaCO_3$ C and 50 % of $CaCO_3$ B is larger than 41 microns which is mean diameters of 35 μ ceramic disks. This is 62% and 80 % respectively for 20 μ which has a mean diameter of 27 microns.

60 μ ceramic disks: Cumulative 11.5 % of $CaCO_3$ C and 20 % of $CaCO_3$ B is larger than 72.5 microns the mean diameter of these ceramic disks.

90 μ ceramic disks: Cumulative 5% of $CaCO_3$ C and 11% of $CaCO_3$ B is larger than 98 microns which is the mean diameter of these ceramic disks.

150 μ ceramic disks: For this size of ceramic disks none of the $CaCO_3$ C and 3% of $CaCO_3$ B is larger than mean diameter of 179 microns.

The D90, D50 and D10 values of ceramic disks and the cumulative percentage of $CaCO_3$ below these values for each of the ceramic disks is shown on table 5-4.

During PPT full lost is occurred for several combinations. For 60 μ and 90 μ ceramic disks plugs with $CaCO_3$ A, for 150 μ ceramic disks $CaCO_3$ A and $CaCO_3$ C are failed to initiate plugging. Moreover, for 60 μ disks the plug prepared with bridging materials fewer than 44 microns also failed to initiate plugging.

Table 5-4: Cumulative Particle Sizes below Ceramic Disk pore sizes

20 μ	***D90 μ***	***D90>***	***D50 μ***	***D50>***	***D10 μ***	***D10>***
$CaCO_3$ C%	*64.2*	83	*27.1*	38	*12.2*	10
$CaCO_3$ B%	*64.2*	72	*27.1*	19.9	*12.2*	0
35 μ	***D90***		***D50***		***D10***	
$CaCO_3$ C%	*102.5*	95.6	*41.5*	63.2	*17.8*	17
$CaCO_3$ B%	*102.5*	90	*41.5*	48.8	*17.8*	2
60 μ	***D90***		***D50***		***D10***	
$CaCO_3$ C%	*150.7*	100	*72.5*	88.2	*12.4*	10
$CaCO_3$ B%	*150.7*	96.5	*72.5*	79.2	*12.4*	0
90 μ	***D90***		***D50***		***D10***	
$CaCO_3$ C%	*252.7*	100	*96.9*	94.3	*37.9*	57.8
$CaCO_3$ B%	*252.7*	100	*96.9*	88.9	*37.9*	43
150 μ	***D90***		***D50***		***D10***	
$CaCO_3$ C%	*361.7*	100	*179.2*	100	*68.6*	87
$CaCO_3$ B%	*361.7*	100	*179.2*	97.8	*68.6*	77

For 35 μ and 20 μ ceramic disks the best filtration parameters has been observed from the blends of different sizes of particles. A blend of $CaCO_3$ A-B-C provides the smallest total filtration volume and $CaCO_3$ B-C provides the smallest spurt loss. Because the mud contains wide range of particles so filter cake quality is improved.

Cumulatively 62.5 % of $CaCO_3$ C and 50 % of $CaCO_3$ B is below 41 microns which is mean diameters of 35 μ ceramic disks.

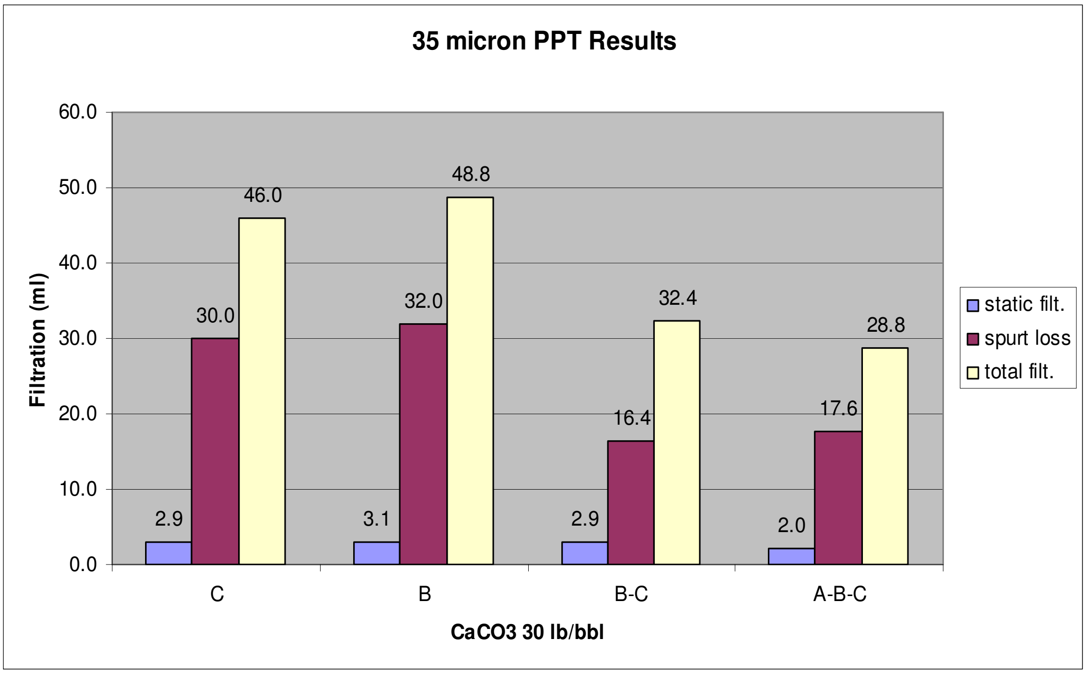

Figure 5-10: Effect of particle size for 35 µ ceramic disks

For 35 µ ceramic disks both $CaCO_3$ B and $CaCO_3$ C filtration results are similar. It is same for 20 µ ceramic disks for test 1 and test 2 (appendix B). Plugging is not the primary reason of the reduction of filtration values for single $CaCO_3$ plugs and blend plug, but also the filter cake quality. Larger filter cake has formed for the test of $CaCO_3$ B and $CaCO_3$ C than the blends. As a result the tendency is an increase in total filtration volume and filtration rate. Reduction of filtration through the 35 µ to 20 µ ceramic disk is because of the smaller pore sizes.

On the other hand comparing the results for 35 µ and 60 µ ceramic disks; although 60 µ has larger pore sizes, it has showed better results than 35 µ ceramic disks for the tests of $CaCO_3$ C and similar results for $CaCO_3$ B plugs. These results prove that plugging occurred for both of the bridging materials and $CaCO_3$ C is more suitable for 60 µ ceramic disks

Cumulative 88.5 % of $CaCO_3$ C and 80 % of $CaCO_3$ B is smaller than the mean pore size of these ceramic disks.

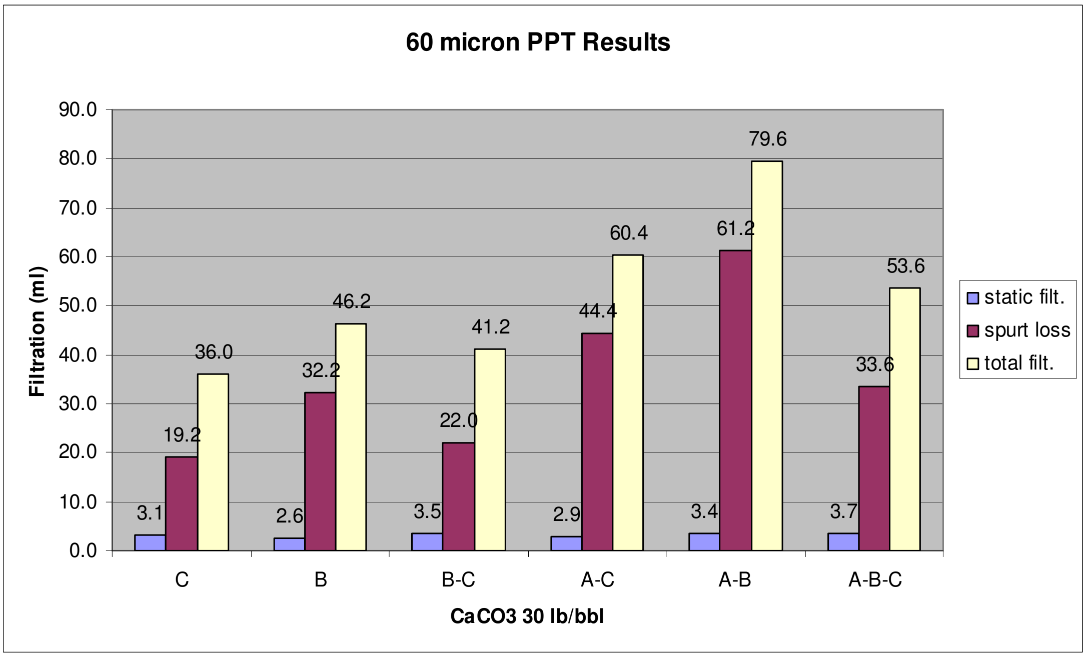

Figure 5-11: Effect of particle size for 60 µ ceramic disks

From Figure 5-11 it can be seen that $CaCO_3$ A has a negative effect in filtration parameters because, its particle sizes are larger than the pore throats. It has no effect on plugging. Adding $CaCO_3$ A works as reduction in concentration and mud additives because of encapsulation.

It is again obvious that $CaCO_3$ C has better effect on 60 µ filtration medium. Low in filtration values for six sets of experiments of 30 lb/bbl concentration reveals the suitability of $CaCO_3$ C.

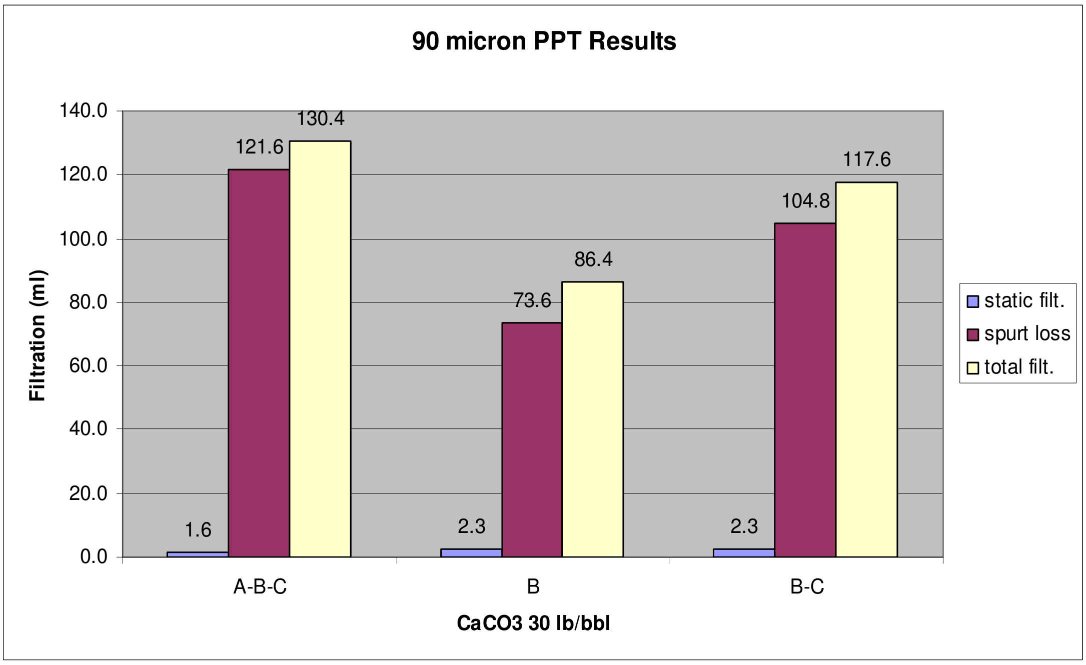

Figure 5-12: Effect of particle size for 90 µ ceramic disks

The results of 90 µ ceramic disks shows as the pore size diameters are increased the filtration results also increased. Moreover fine solids invasion is observed during tests. The spurt loss is high but static filtration rates are decreased. The reason is as the pore throats are extended, LCM has difficulties in plugging pore throats. Reduction in static filtration rate is because of the bridging materials invade through the pore throats but not completely pass through it but stuck inside and can not able to have a surface plugging.

For 60 µ ceramic disks $CaCO_3$ C and for 90 µ ceramic disks $CaCO_3$ B has provided better solution than other sizes of bridging agents. D90 of $CaCO_3$ C is 76 microns and D90 of $CaCO_3$ B is 102 microns. Plugging occurred successfully satisfied the requirement of, D90 particle size of $CaCO_3$ is equal or at least close to mean pore size of ceramic disks as Hands et al. stated.

For testing mean and minimum pore throat size and their relationship with sized particles a set of test has made. By using 200 mesh (74 microns) and 325 mesh

(44 microns) sieves 3 sized of particles are separated from $CaCO_3$ C. 74 micron is the mean and 44 micron is the minimum pore throat size of 60µ ceramic disk.

Table 5-5: Sieve size and particle diameter

Sieve Number	Opening, mm	Wire Diameter, mm
200	**0.074**	**0.053**
325	**0.044**	**0.036**

(Taken from http://www.iyte.edu.tr)

Different amount of these particles are blended stay in the range of mean and minimum pore size diameters. The concentrations of particles are equal for both the above of the mean throat size and below of the minimum throat size. Amount of particles has changed between mean pore size and minimum pore size as the percentage seen on the table 5-4 for the total concentration of 30 lb/bbl. For example for test 36, 12 lb/bbl of particles between 74 microns and 44 microns blended with 9 lb/bbl of particles above 74 microns and 9 lb/bbl of particles below 44 microns have blended.

The results can be seen from table. These results are taken from appendix B.

Table 5-6: Effects of Concentration Difference of Particle Sizes between Mean & Minimum Pore Diameters

No:	Particle Size	7,5 min (ml)	30 min (ml)	Static Filtration Rate (ml/sec$^{1/2}$)	Spurt Loss (ml)	Total Filtration (ml)
23	$CaCO_3$ C	13.8	18	3.1	19.2	36.0
24	$CaCO_3$ B	19.6	23.1	2.6	32.2	46.2
35	74 µ>20%>44 µ	12.2	16.2	2.9	16.4	32.4
36	74 µ>40%>44 µ	14.8	19	3.1	21.2	38.0
37	74 µ>60%>44 µ	14	17.8	2.8	20.4	35.6
38	74 µ>80%>44 µ	14.2	18	2.8	20.8	36.0
39	%50<44µ & 50% mean	16.8	20.6	2.8	26.0	41.2
40	>44 micron	275	275	0.0	550.0	550.0

The filtration results of the tests 23, 36, 37 and 38 are similar. But on test 35 a reduction in total and spurt loss can be seen. So the concentration of particles between the mean and minimum pore sizes does not affect the plugging at concentration of 40% and higher. For test 24 and 39 high spurt loss and total circulation is observed. Particles above the mean pore size have effect on plugging also. For an optimum value of %10 coarse particles has a positive effect on filtration parameters.

Test 40 reveals that the LCP mixture of the particles finer than minimum pore size diameter failed in plugging and full lost circulation has occurred. The best filtration result was observed in test 35. In this test maximum amount of fine particles exists as 12 lb/bbl with the combination of particles between mean and minimum pore diameters as 6 lb/bbl and higher than mean pore throat size as 12 lb/bbl. As stated before changes of particles sizes between mean and minimum pore size diameter does not have an effect on filtration parameters on the other hand increasing coarse sized particles has a increases total and spurt loss. So the best combination should be the increased amount fine particles with combination of coarser.

As said before there are various rules in the industry. The tests enable us to check accuracy some of these rules

Abrams' Rule: Abrams' rule stated that for plugging "median particle size of the bridging agent should be equal or slightly grater than 1/3 of the median pore size of the formation." And it is followed with the suggestion of the bridging material concentration should be at least 5% by volume in the final mud mix.

Table 5-7: Cumulative Particle Sizes above and below 1/3 of mean pore sizes of Ceramic Disks.

35 µ	**(cumulative%) >**	**1/3 × Mean Diameter**	**> (cumulative%)**
$CaCO_3$ C%	90	13.87	10
$CaCO_3$ B%	100	13.87	0
60 µ			
$CaCO_3$ C%	68.5	24.2	31.5
$CaCO_3$ B%	85	24.2	15
90 µ			
$CaCO_3$ C%	52	32.3	48
$CaCO_3$ B%	69.5	32.3	30.5
150 µ			
$CaCO_3$ C%	17.7	59.7	82.3
$CaCO_3$ B%	30	59.7	70

As said before10 lb/bbl concentration is equal to 1% of bridging material by volume in LCP and for the test results for 20 μ, 35 μ 6% of bridging materials has a negative effect on filtration parameters as increasing spurt loss total filtration and static filtration rates. However, for 90 μ ceramic disk a drastically decrease has been observed 4% to 6% of bridging materials used as shown in figures 5-7 and 5-8. Abrams' rules define the particle size, pore diameter and concentration relationship for initializing the sealing. "But it is not addresses an ideal packing sequence for minimizing fluid invasion and optimizing sealing."(Dick et al., 2000)

Ideal Packing Theory (IPT): Abrams' rule defines the particle size to initiate bridging. Determination of particle size of bridging material is the first step in selecting proper type of LCP. Dick et al. stated the "Ideal Packing Theory" to define the total particle range required to seal all pores even those created by bridging agents.

The IDP uses a linear type of graph (cumulative % below vs. square root of particles) to optimize particle size distribution for particular reservoirs. Optimum target lines have been plotted for square root of median pore size of used disks with particle size distributions of the selected $CaCO_3$. For ideal packing, the slope of $CaCO_3$ should be approximate to the slope of the disk. Ideal particle size distribution line of the ceramic disks and the particle size distribution of bridging materials are shown in Figure 5-13, Figure 5-14, Figure 5-15 and Figure 5-16.

For 35 μ ceramic disks the slopes of $CaCO_3$ A, B and C is on the right hand side of the ideal PSD line. It can be seen that available $CaCO_3$ sizes are larger for these ceramic disks. For 35 μ ceramic disks test results are similar for LCPs' of $CaCO_3$ B and C. This is same as 20 μ ceramic disks that increasing the size of bridging materials does not affect the filtration parameter significantly. Because the pore size of 20 μ ceramic disks has smaller pore size filtration results are smaller than 35 μ ceramic disks.

Ideal PSD line for 60 μ ceramic disks is the closest line to the slope of $CaCO_3$ C. The pre-evaluation of the results shows that 60 μ ceramic disks $CaCO_3$ C. The effect of concentration has eliminated above 30-40 lb/bbl in the filtration parameters. On the other hand, using $CaCO_3$ B as bridging material will too be appropriate for drill-in fluids for lost circulation to the formation which has pore size distribution close to 60 μ ceramic disks. During the drilling process, larger particles become smaller.

Particle size distribution of $CaCO_3$ B is the best match for 90 μ ceramic disks according to the test results of these sized ceramic disks. From the Ideal PSD line for 90 μ ceramic disks it is obvious that it is the suitable size for ideal packing theory either. Unlikely to 60 μ ceramic disks, 60 lb/bbl is the best concentration for 90 μ ceramic disks.

Finally for 150 μ ceramic disks both slope of $CaCO_3$ B and $CaCO_3$ C is on the left hand side of Ideal PSD line of the median pore size. For all of the cases of ceramic disks $CaCO_3$ A has a large particle size distribution. Although plugging is achieved with CaCO3 B large amount of particles invading through is observed both in filtrate and the above ceramic disk.

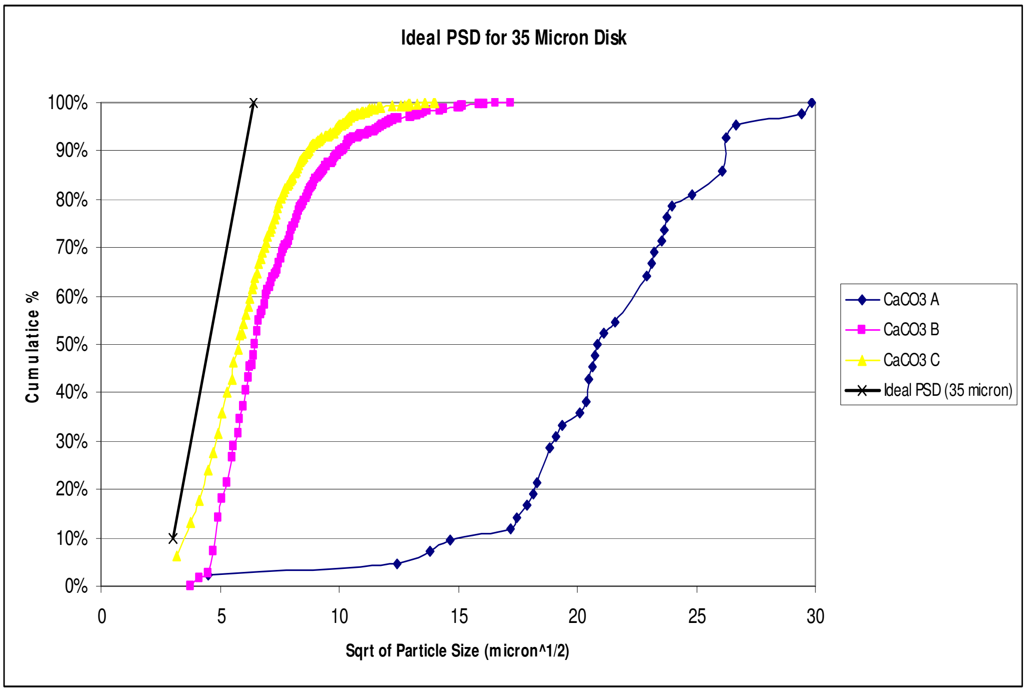

Figure 5-13: Ideal PSD for 35 μ Ceramic Disks & $CaCO_3$ Grades

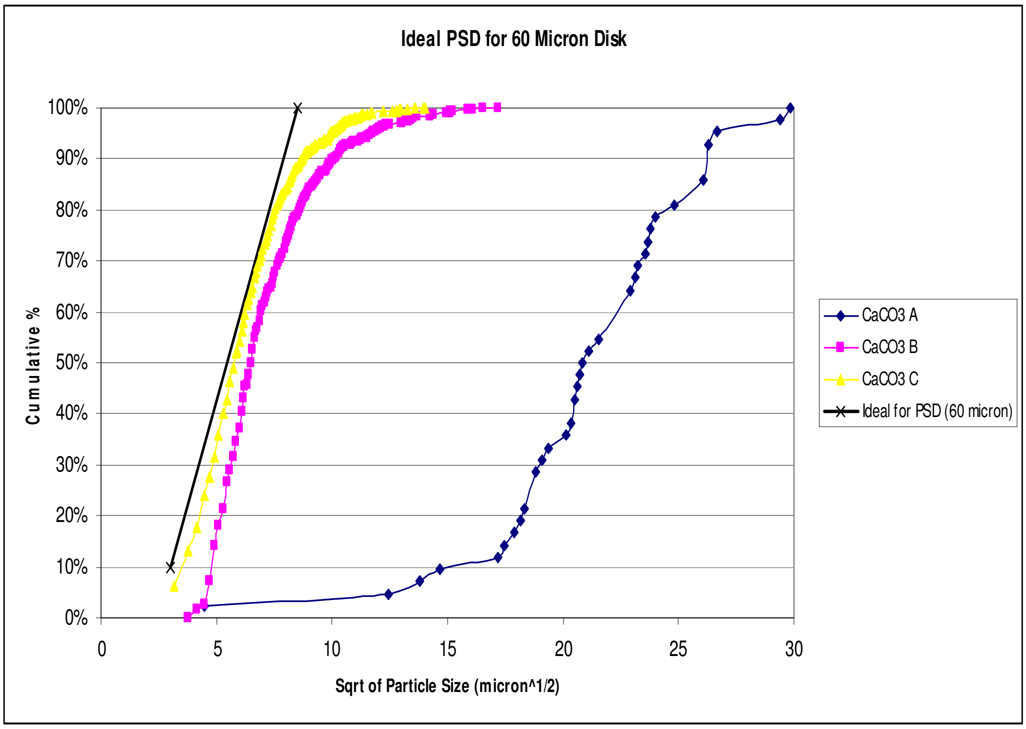

Figure 5-14: Ideal PSD for 60 μ Ceramic Disks & $CaCO_3$ Grades

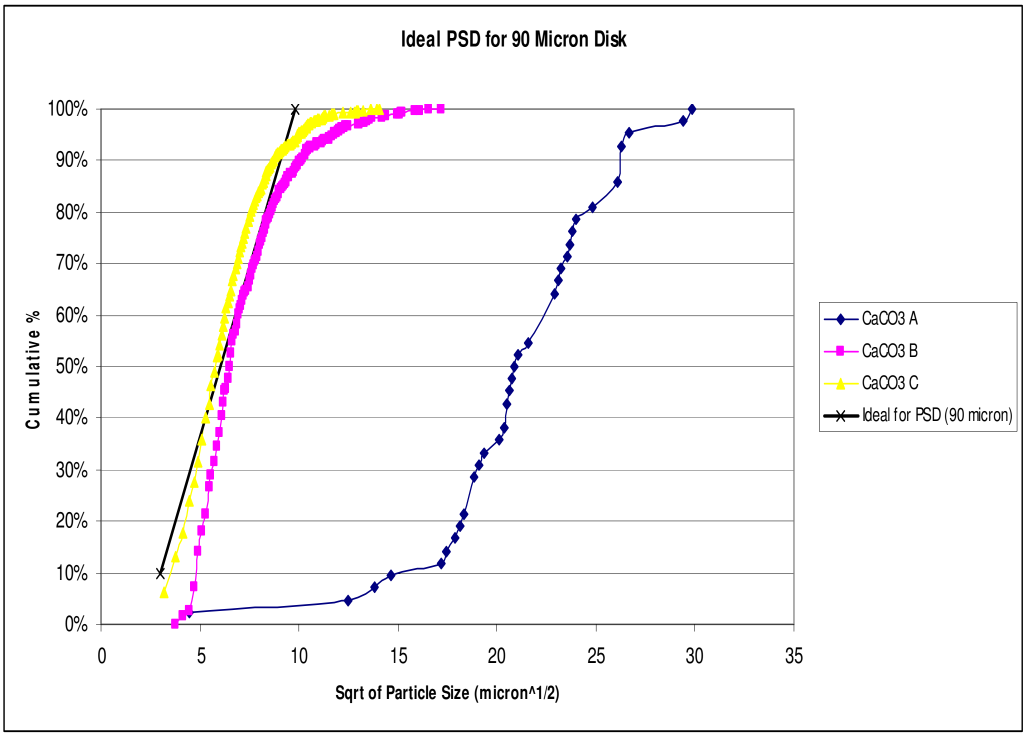

Figure 5-15: Ideal PSD for 90 µ Ceramic Disks & $CaCO_3$ Grades

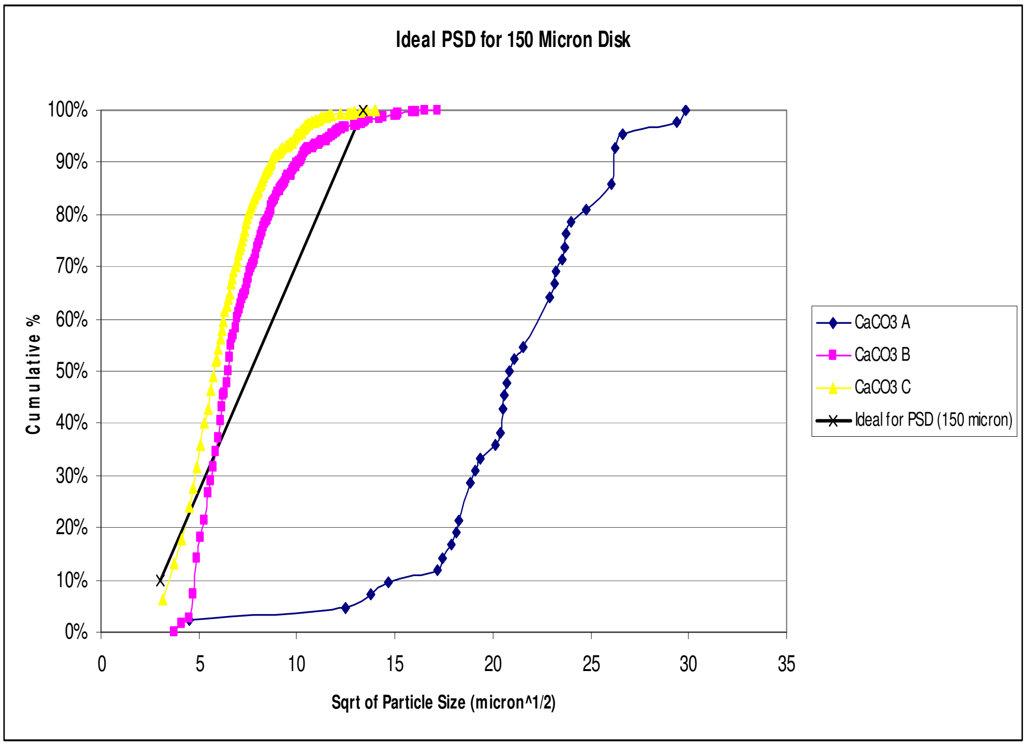

Figure 5-16: Ideal PSD for 90 µ Ceramic Disks & $CaCO_3$ Grades

CHAPTER 6

CONCLUSIONS

During this study, performance of $CaCO_3$ is tested as a LCM in polymer base mud. Different sizes and concentrations of $CaCO_3$ are tested for plugging tests using ceramic disk as filtration medium. The experiments were carried out in TPAO Research Center. The results are analyzed and the following conclusions can be drawn from the study:

- Large particle sizes have failed in plugging smaller pore throat sizes. If particle size distribution is suitable for pore throat size minimum void spaces in the filter cake is obtained.

- Concentration of the LCM is affecting sealing capacity of LCP. Whereas; it is not appropriate for all cases to state that, as the concentration of $CaCO_3$ increases, sealing capacity of LCP develops. For some cases especially smaller pore sizes with larger bridging materials concentration should be optimized.

- For 90 μ ceramic disk increasing bridging materials concentration from4% to 6% a drastically decrease has been observed. On the other hand for 20 μ and 35 μ ceramic disks 6% of bridging materials has a negative effect on filtration parameters as increasing spurt loss total filtration and static filtration rates.

- For 60 μ ceramic disks $CaCO_3$ C and for 90 μ ceramic disks $CaCO_3$ B has provided better solution than other sizes of bridging agents.

- Permeability plugging tests illustrative equipment for studying the effect of lost circulation plugs. Photographs taken after the tests reveals the smaller filtration values obtained for maximum sealing.

- Concentration and particle size distribution used for LCP for bridging materials should be determined after evaluation of the physical characteristics of porous media.

- The ideal packing theory is successful in the guidance of the particle size distribution selection for sealing the pore throats.

- Hands et al., defines another effective statement for the selection of the particle sizes used as bridging materials suitable for determined pore throat size.

CHAPTER 7

RECOMMENDATIONS

This study is an important step to understand the effect of particle size of LCM used in combat with lost circulation, the relationship of particle size distribution with pore throat size of lost circulation zones. On the other hand further studies are recommended for better understanding of LCM character;

- Although calcium carbonate is the most commonly used type of granular LCM, other LCM's (wood flour, walnut etc.) can be used with different particle sizes at least for non-productive formations.

- Flaky and fibrous types of LCM are required and combinations of all should be tested for the same conditions that were run in this study.

- In this study static condition is tested. Same compositions can be tested for dynamic conditions and compare the results may give more accurate idea about characteristic of bridging materials and pore sizes.

- Different types of drilling fluids can be tested.

- Further tests should be conducted for varying formation temperatures and pressure differentials.

REFERENCES

Abrams, A.: “Mud Design to Minimize Rock Impairment due to Particle Invasion,” Journal Petroleum Technology, May 1977, 586-592.

Alajmi, S.E. and Schubert, J. J. “Optimum selection of Underbalanced Techniques” paper SPE/IADC 85322 presented at the 2003 SPE/IADC Middle East Drilling Technology Conference and held in Abu Dhabi, U.A.E., 20–22 October 2003.

Bugbee, J. M.: “Lost Circulation-A Major Problem in Exploration and Development”, paper presented at the spring meeting of Southwestern District, Division of Production, Forth Worth, Texas (March 1953)

Cargnel, R.D. and Luzardo, J.P.: “Particle Size Distribution Selection of $CaCO_3$ in Drill-In Fluids: Theory and Applications,” paper SPE 53937 presented at the 1999 SPE Latin American and Caribbean Petroleum Engineering Conference, Caracas, Venezuela, 21-23 April 1999.

Perez, C. T., Smith, J. R., Edwards, J. K.: “A New Comprehensive, Mechanistic Model for Underbalanced Drilling Improves Wellbore Pressure Predictions” paper SPE 74426 presented at the SPE International Petroleum Conference and Exhibition in Mexico held in Villahermosa, 10-12 February 2002.

Dick, M.A., Heinz, T.J., Svoboda, C.F., Aston, M.: “Optimizing the Selection of Bridging Particles for Reservoir Drilling Fluids” paper SPE 58793 presented at the 2000 SPE International Sypmposium on Formation Damage, Lafayette, Louisiana, 23-24 Feb. 2000.

Pittman, E.D. and Thomas, J.B "Some Applications of Scanning Electron Microscopy to the Study of Reservoir Rock" paper SPE 7750 presented at thr SPE Annual Fall Technical Conference and Exhibition held in Houston, 1-2 October 1978.

Hand, N., Kowbel, K., Maikranz, S., Nouris, R.: "Drill-in Fluid Reduces Formation Damage, Increases Production Rates" Oil & Gas Journal July 13 1998.

James, R. B., Catalin, D. I., Thomas, J. H. : "Lost Circulation Control: Evolving Techniques and Strategies to Reduce Downhole Mud Losses" IADC/SPE 67735 presented at the IADC/SPE Drilling Conference Amsterdam, The Netherlands, 27 February-1 March 2001

Adachi, J., Bailey L., Houwen O.H., Meeten G.H. & P.W. Way, Schlemmer R.P.: "Depleted Zone Drilling: Reducing Mud Losses Into Fractures," paper IADC/SPE 87224 presented at the IADC/SPE Drilling Conference, Dallas, Texas, U.S.A., 2—4 March 2004.

J. C. Rojas, P, A. Bern, B. L. Ftizgerald, S. Modi, P. N. Bezant: "Minimizing Down Hole Mud Losses," IADC/SPE 39398 presented at the 1888 IADC/SPE Drilling Conference, Dallas, Texas, 3 -6 March 1998.

Gockel, J. F., Gockel, C.E., and Brinemann, M.: " Lost Circulation: A Solution Based on the Problem" paper SPE/IADC 16082 presented at the 1987 SPE/IADC Drilling Conference held in New Orleans, L.A., 15–18 March 1987.

Lummus, J. L.: "A New Look at Lost Circulation", paper presented at Annual AAODC Meeting in New Orleans, 12-14 October 1966.

McLean, M. R. and Addis, M. A.: "Wellbore Stability: The Effect of Strength Criteria on Mud Weight Recommendations." SPE 20405 presented at the 65th Annual Technical Conference and Exhibition, New Orleans, Sept 23-26, 1990.

M-I Drilling Fluids.L.C.: “Drilling Fluids Engineering Manual,” Copyright 1998.

Nayberg, T. M. And Petty, B. R.: “Laboratory study of Lost Circulation materials for use in both oil-base and water-base drilling muds” IDAC/SPE 14723 10-12 February 1986.

Davis, N., Mihalik, P., Lundie, P. R., Calloni, G.: “New Permeability Plugging Apparatus Procedure Addresses Safety and Technology Issues” paper SPE/IADC 52815 presented at the 1999 SPE/IADC Drilling Conference held in Amsterdam, Holland, 9–11 March 1999.

Pilehvari and Nyshadham, V. R.: “Effect of Material Type and Size Distribution on Performance of Loss/Seepage Control Material,” paper SPE 73791 presented at the at the SPE International Symposium and Exhibition on Formation Damage Control Lafayette. Louisiana. 20—21 February 2002.

Rosato, M.J. and Supriyono, A.: “Use of a Crosslinked Polymer Fluid-Loss Control Agent to Aid Well Cleanouts,” paper SPE 74845 presented at the 2002 SPE/ICOTA Coiled Tubing Conference, Houston, TX, 9-10 April 2002.

Ross, C.M., Williford, J. and Sanders, M.W.: “Current Materials and Devices for Control of Fluid-loss,” paper SPE 54323 presented at the Asia Pacific Oil and Gas Conference and Exhibition, Jakarta, Indonesia, 20-22 April 1999.

Verret, R.: “Fibers to Protect Producing Formations,” paper SPE 58794 presented at the 2000 SPE International Symposium on Formation Damage Control, Lafayette, Louisiana, 23–24 February 2000.

The A to Z Materials “Mesh Size Equivalents”
http://www.azom.com/details.asp?AticleID=1417 (accessed on 30.07.2008).

APPENDIX A

MEAN &MINIMUM CERAMIC DISK PORE DIAMETERS

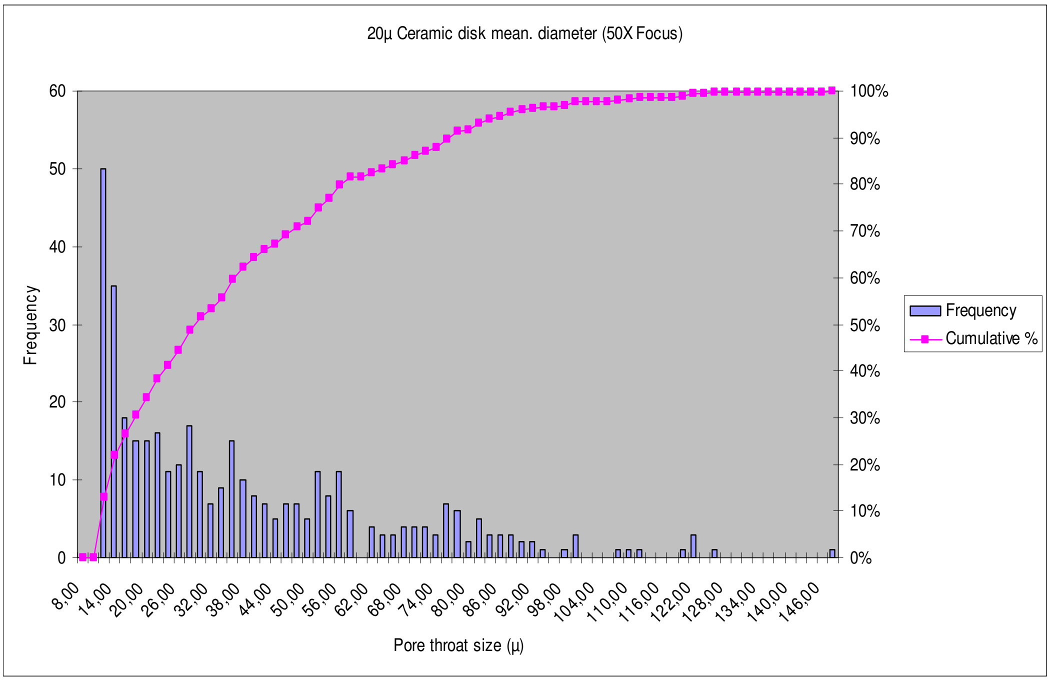

Figure A-1: 20µ Ceramic Disk Mean Diameter (50X Focus)

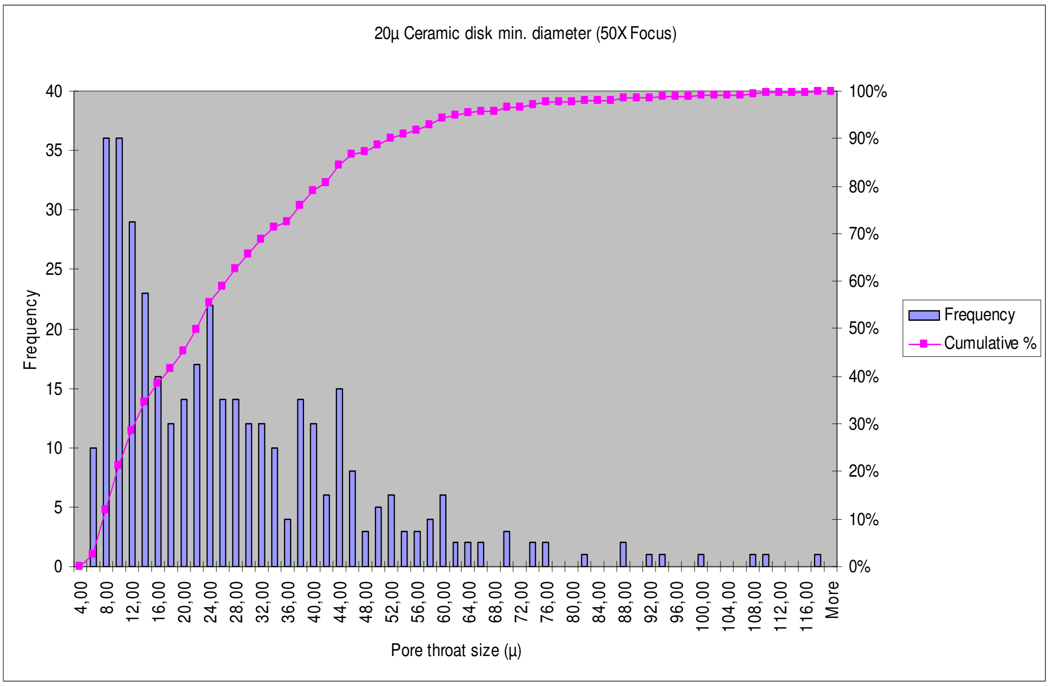

Figure A-2: 20µ Ceramic Disk Minimum Diameter (50X Focus)

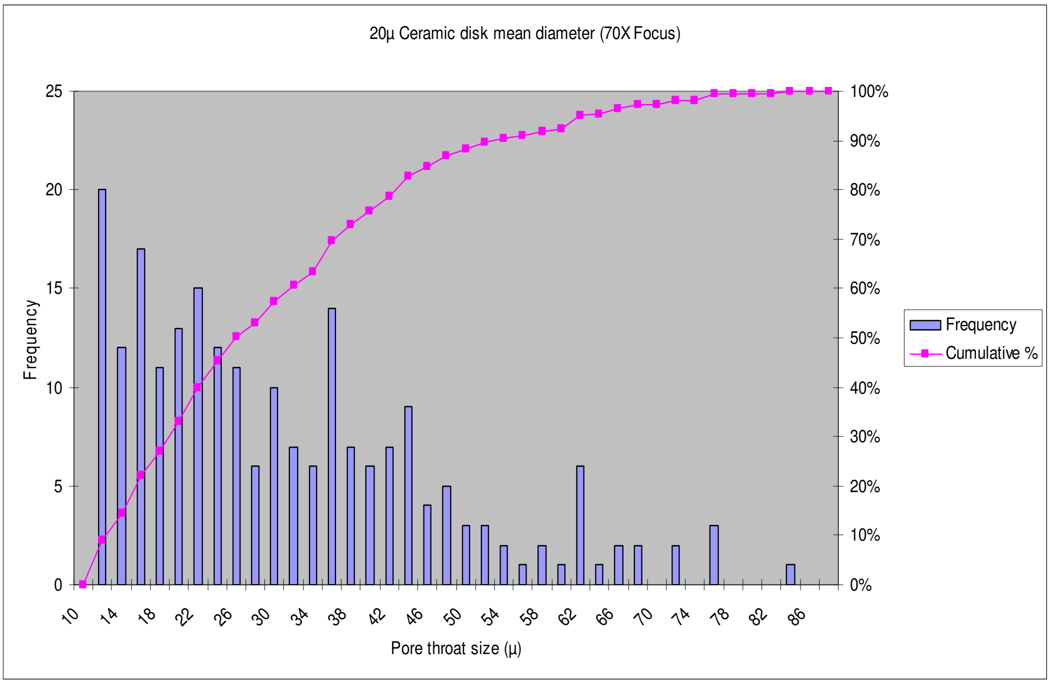

Figure A-3: 20µ Ceramic Disk Mean Diameter (70X Focus)

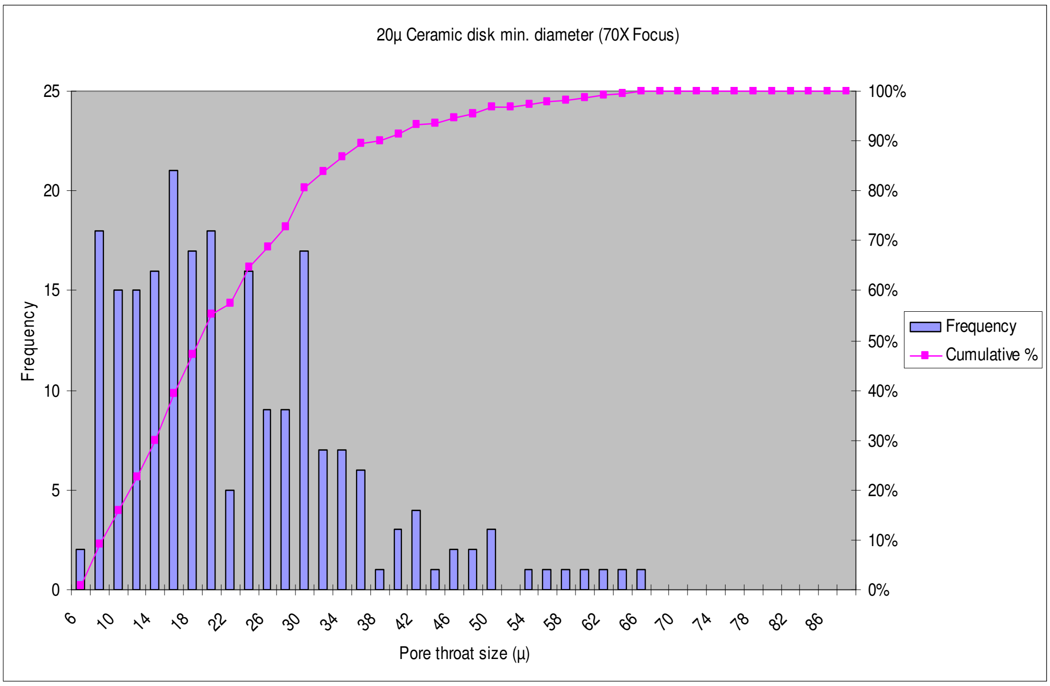

Figure A-4: 20µ Ceramic Disk Minimum Diameter (70X Focus)

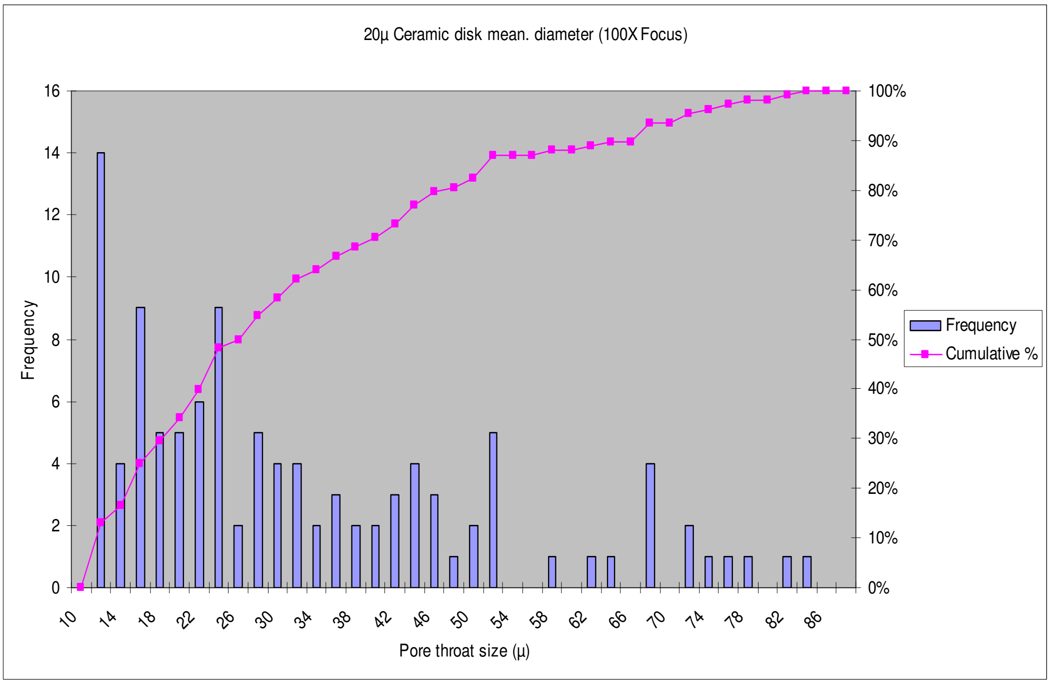

Figure A-5: 20µ Ceramic Disk Mean Diameter (100X Focus)

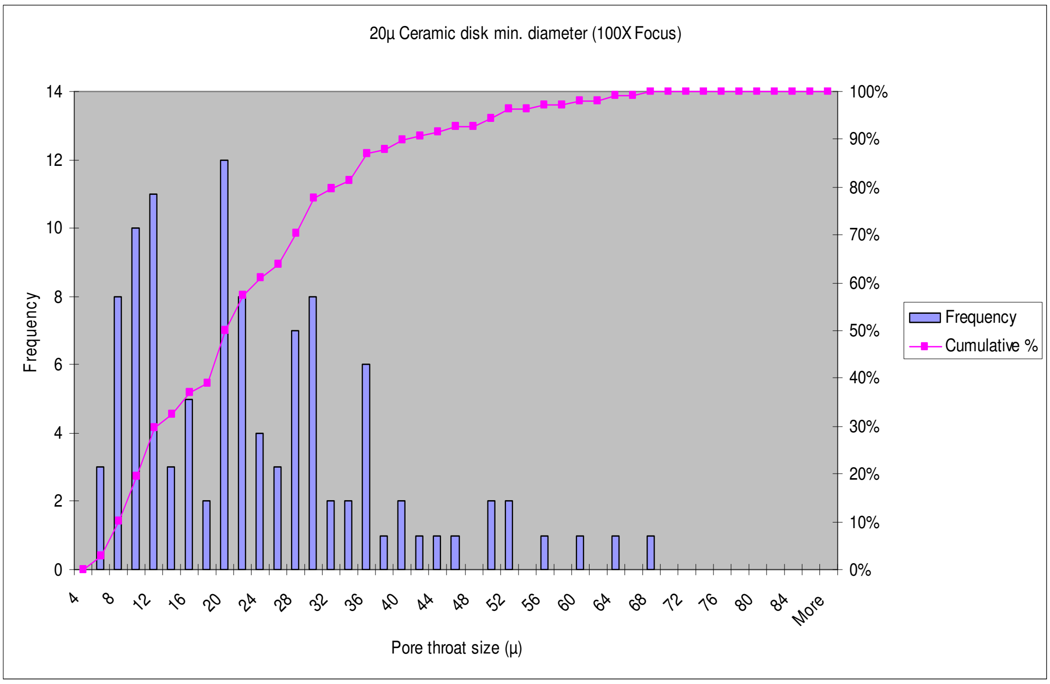

Figure A-6: 20µ Ceramic Disk Minimum Diameter (100X Focus)

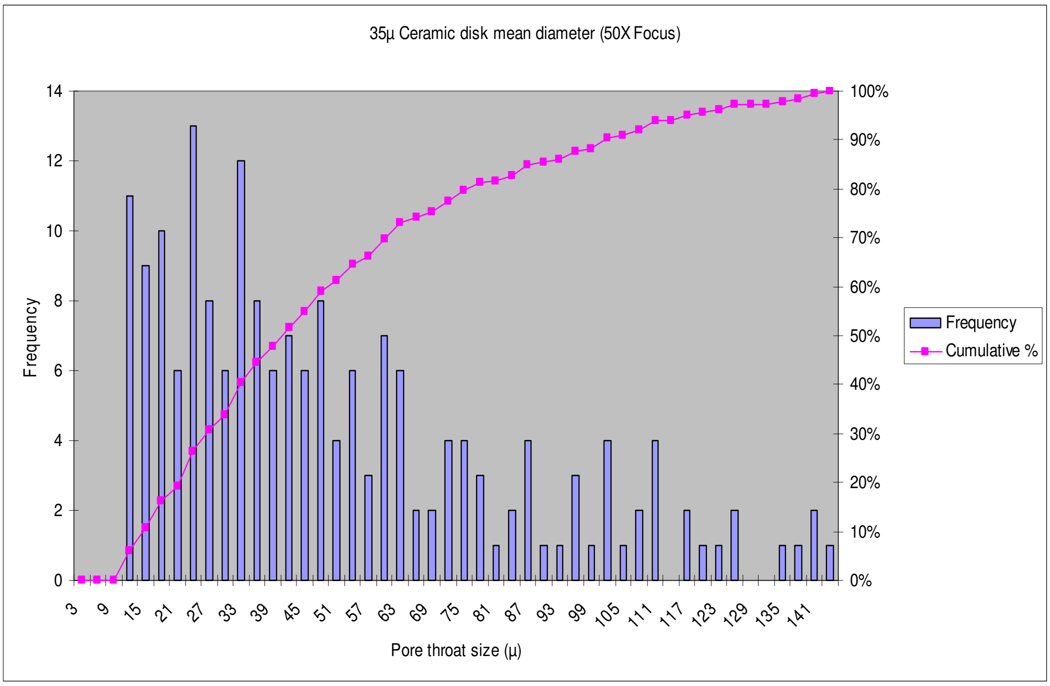

Figure A-7: 35µ Ceramic Disk Mean Diameter (50X Focus)

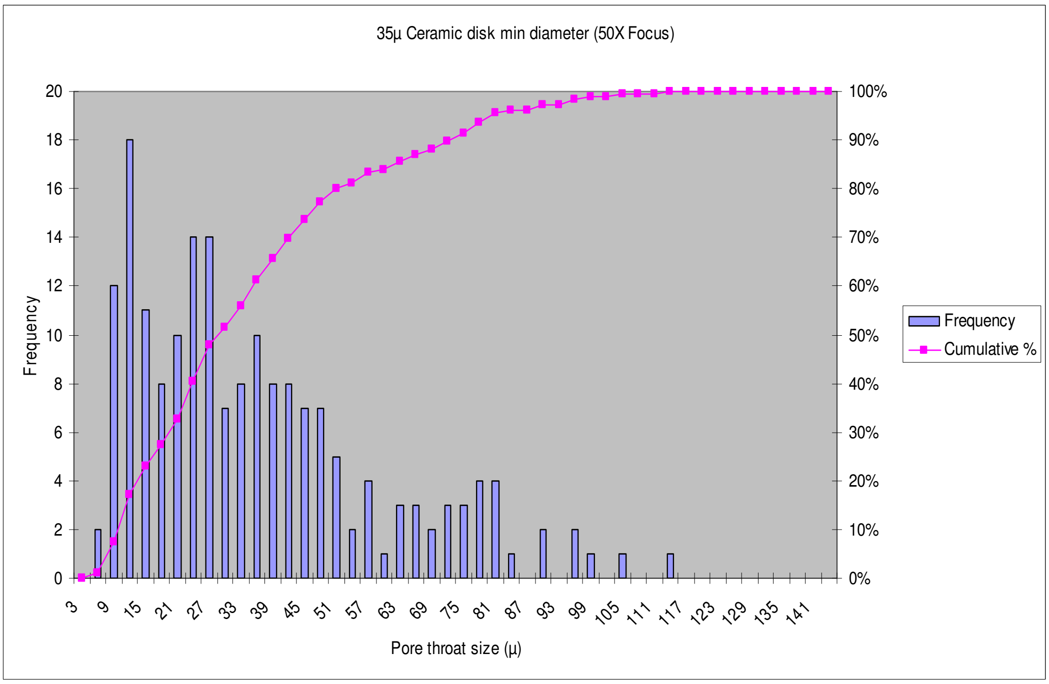

Figure A-8: 35µ Ceramic Disk Minimum Diameter (50X Focus)

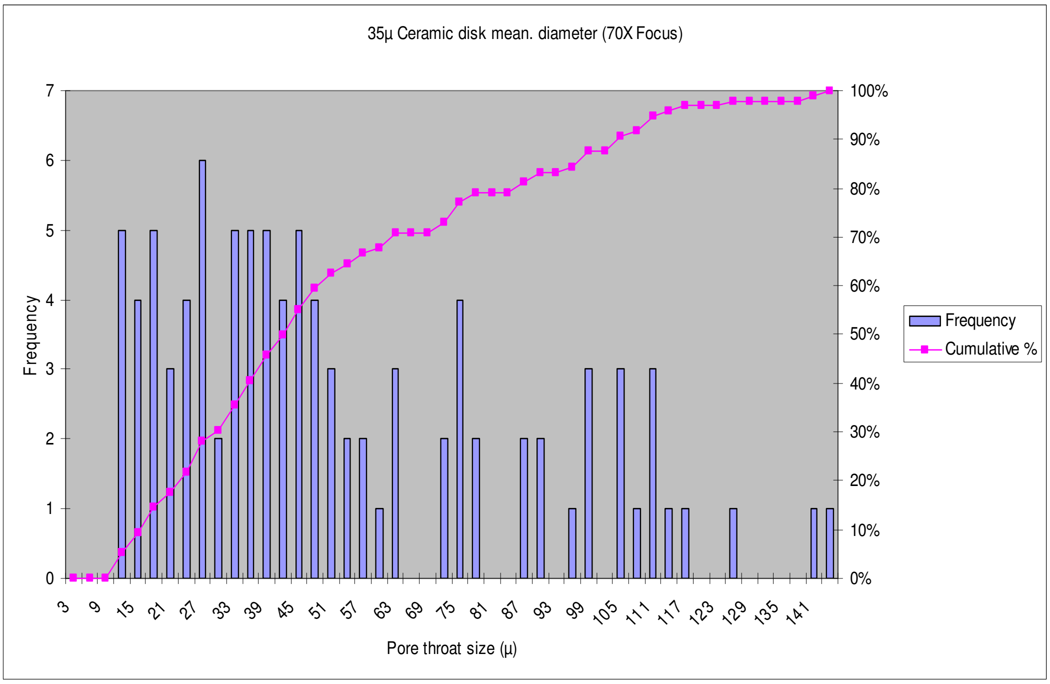

Figure A-9: 35µ Ceramic Disk Mean Diameter (70X Focus)

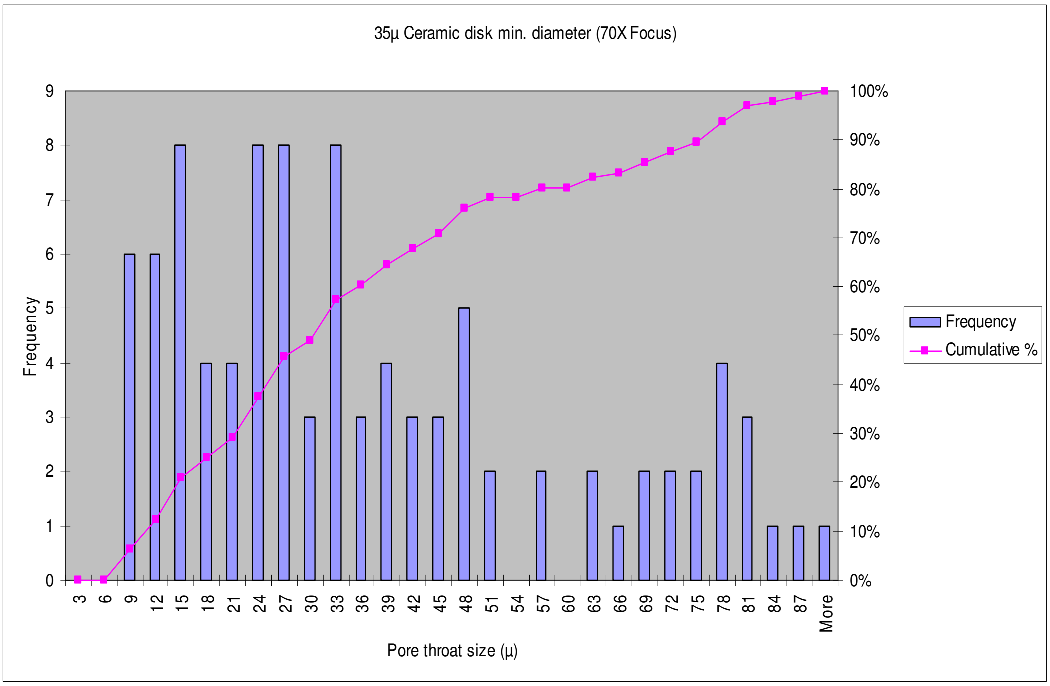

Figure A-10: 35µ Ceramic Disk Minimum Diameter (70X Focus)

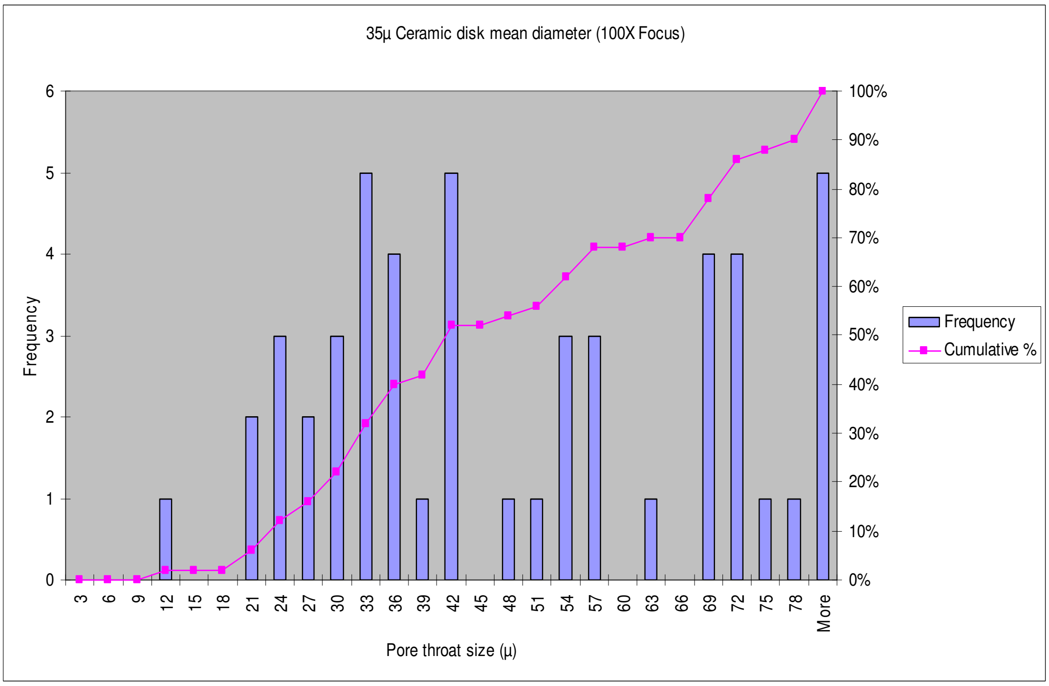

Figure A-11: 35µ Ceramic Disk Mean Diameter (100X Focus)

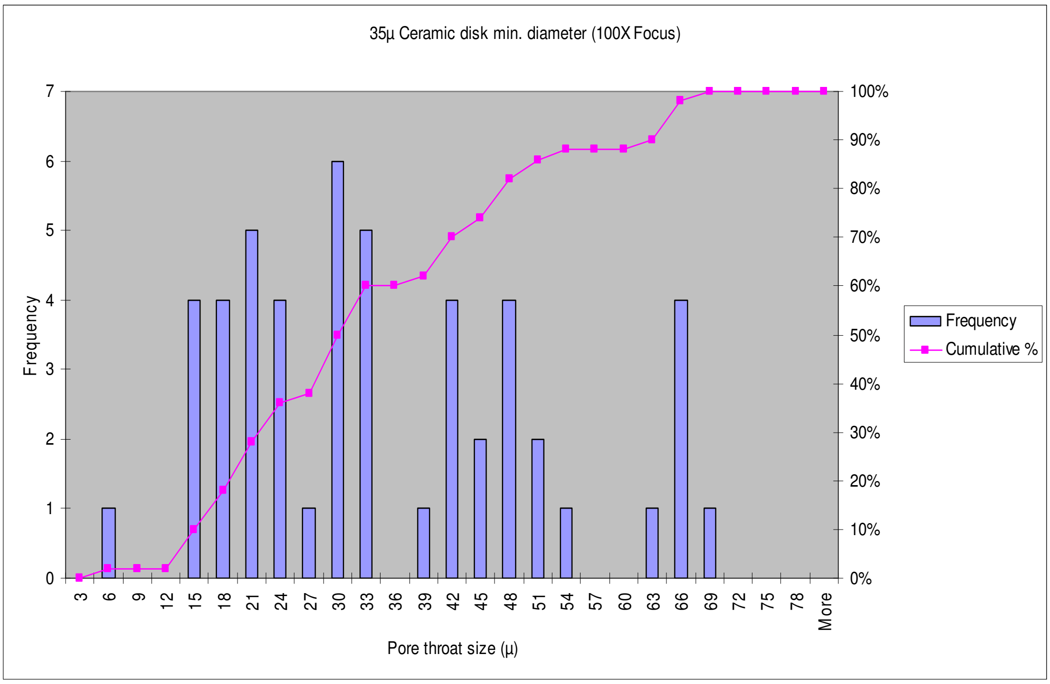

Figure A-12: 35µ Ceramic Disk Minimum Diameter (50X Focus)

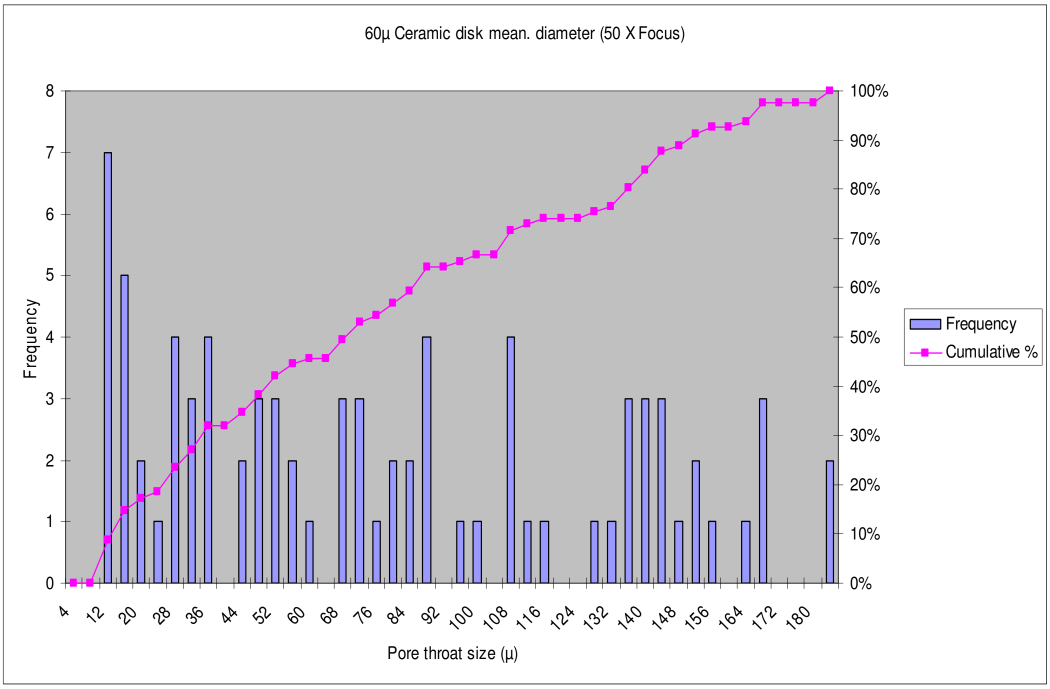

Figure A-13: 60µ Ceramic Disk Mean Diameter (50X Focus)

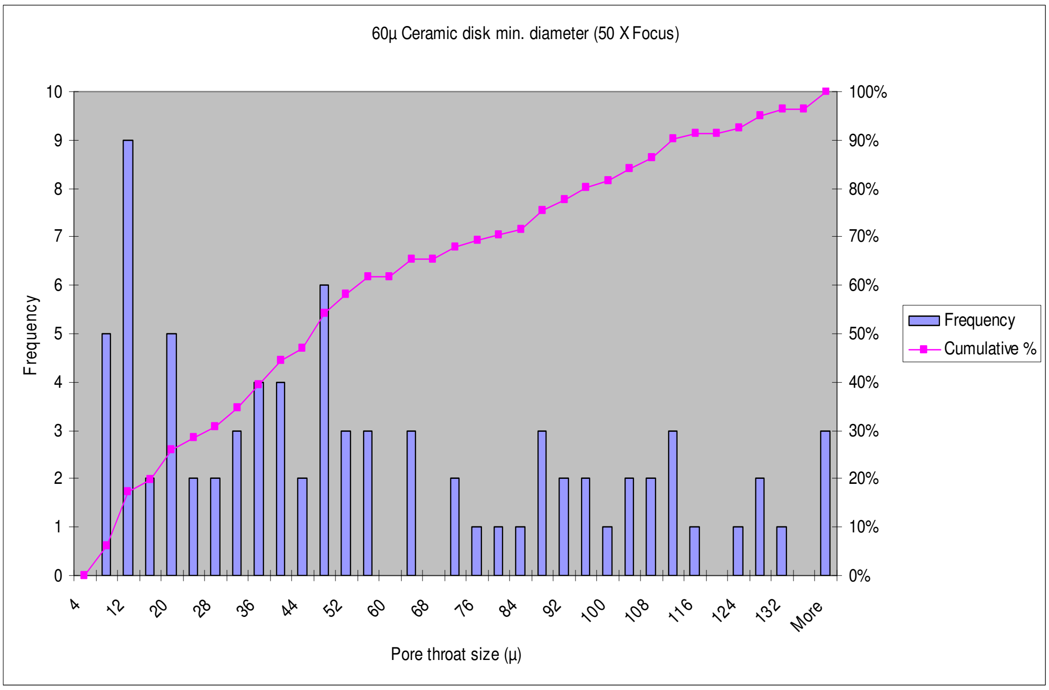

Figure A-14: 60µ Ceramic Disk Minimum Diameter (50X Focus)

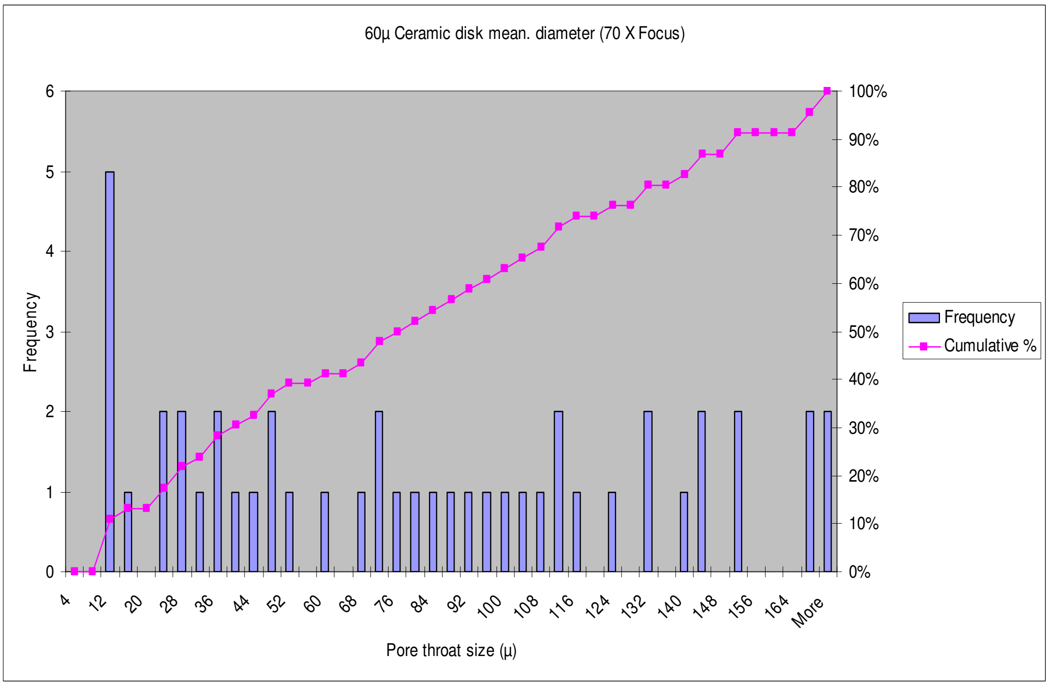

Figure A-15: 60µ Ceramic Disk Mean Diameter (70X Focus)

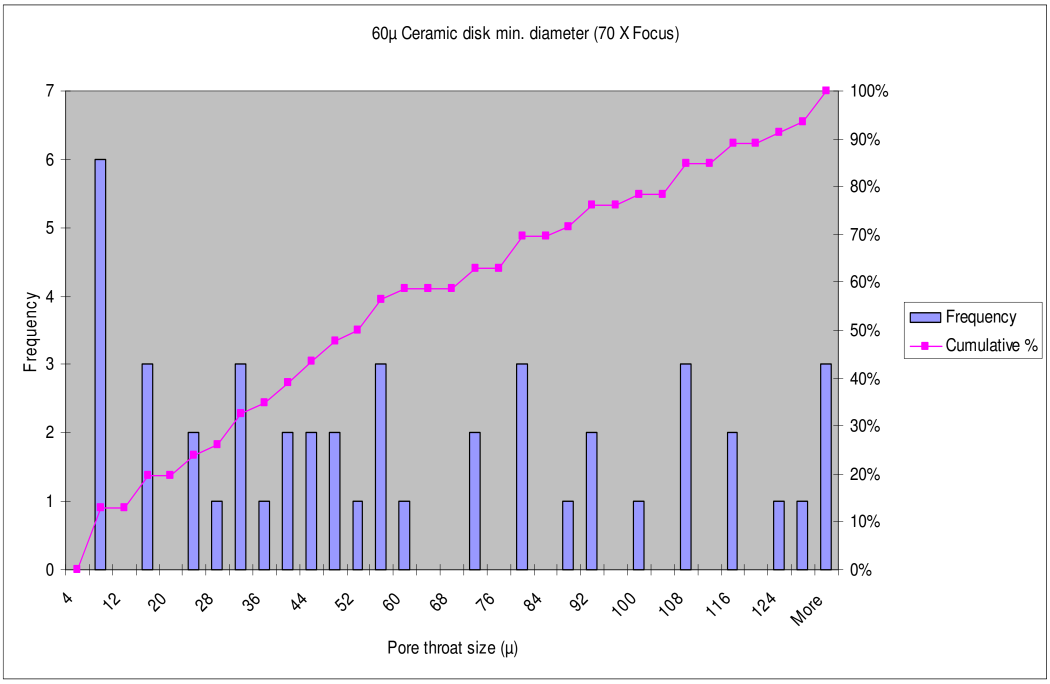

Figure A-16: 60µ Ceramic Disk Minimum Diameter (70X Focus)

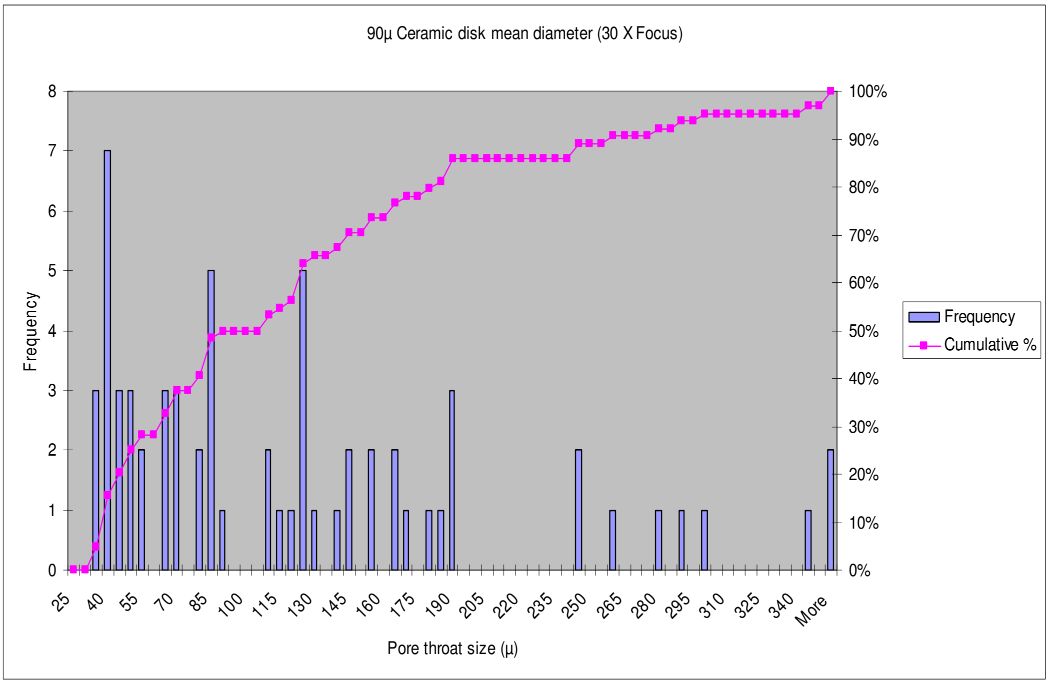

Figure A-17: 90µ Ceramic Disk Mean Diameter (30X Focus)

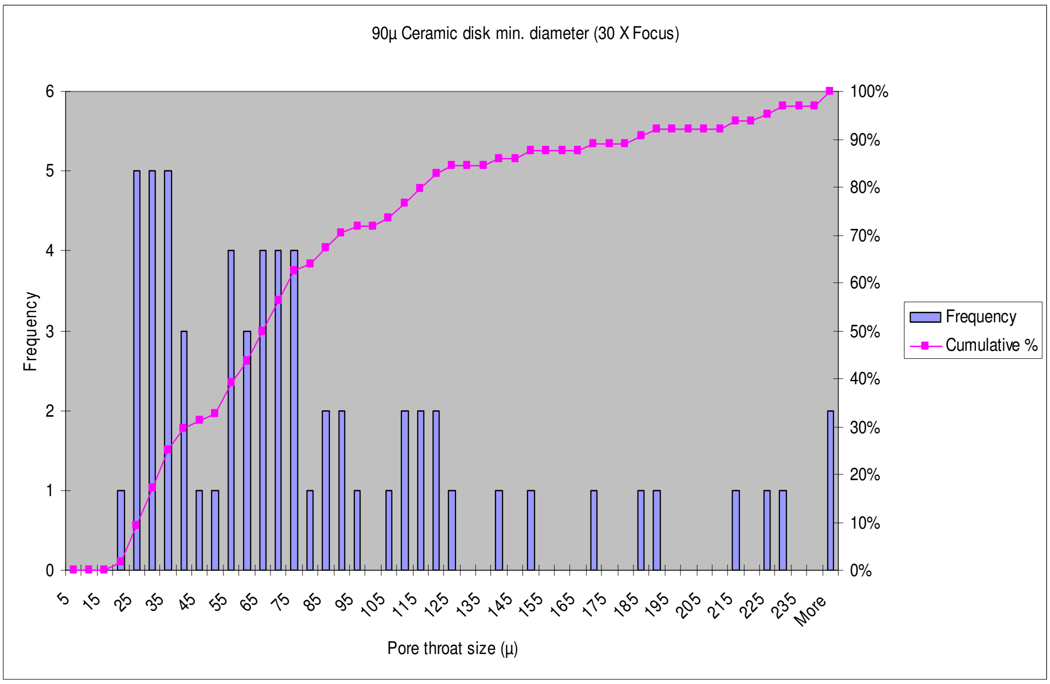

Figure A-18: 90µ Ceramic Disk Minimum Diameter (30X Focus)

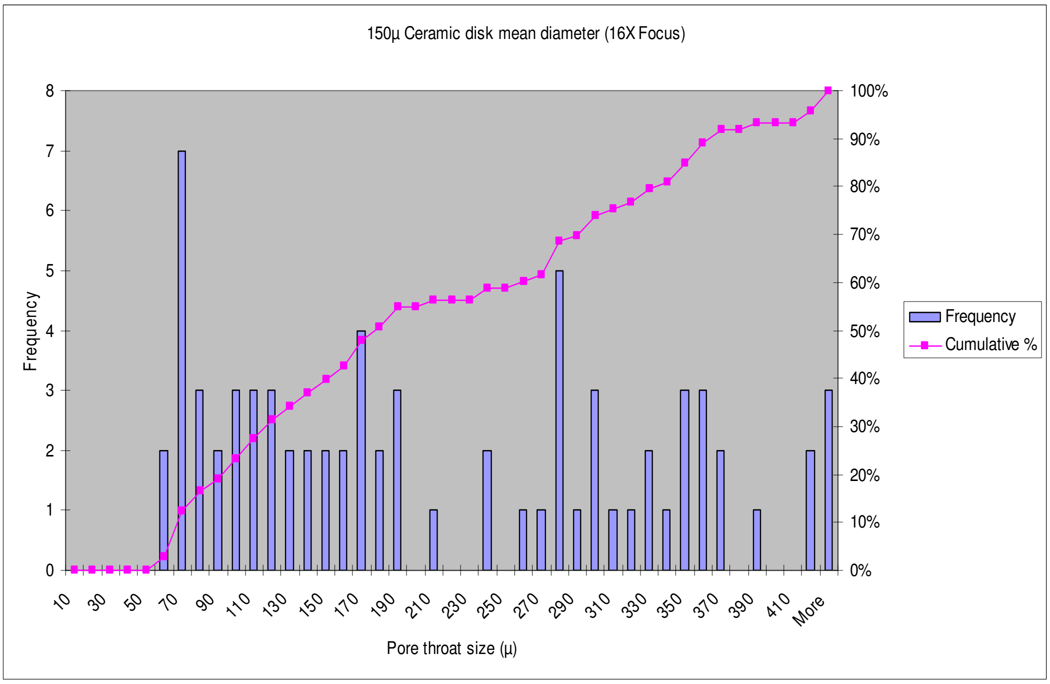

Figure A-19: 150µ Ceramic Disk Mean Diameter (16X Focus)

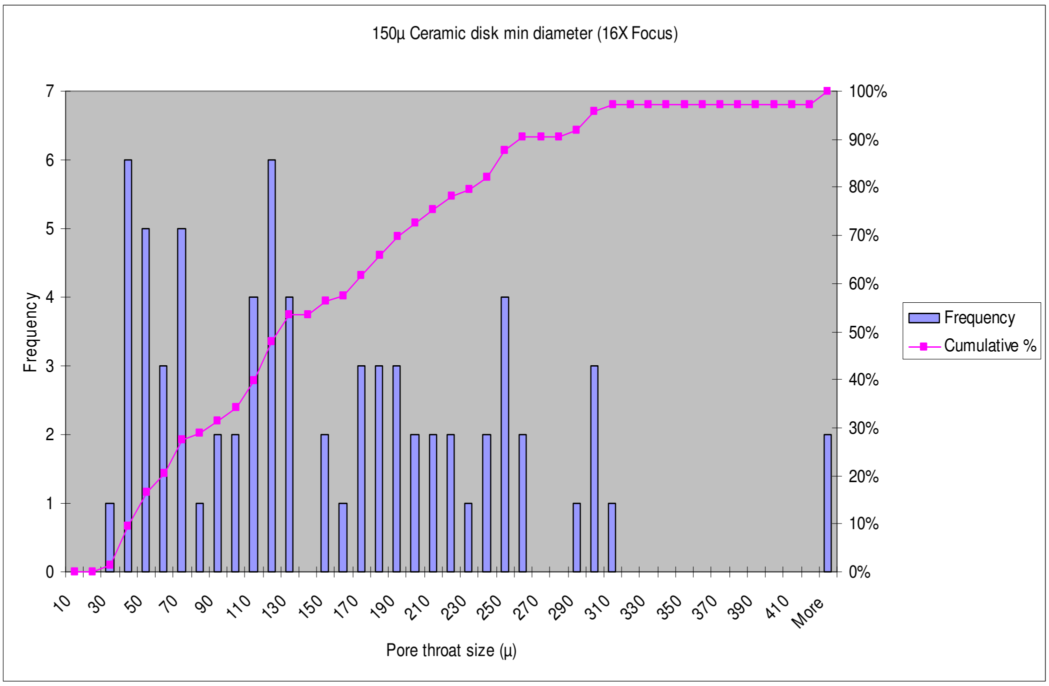

Figure A-20: 150µ Ceramic Disk Minimum Diameter (16X Focus)

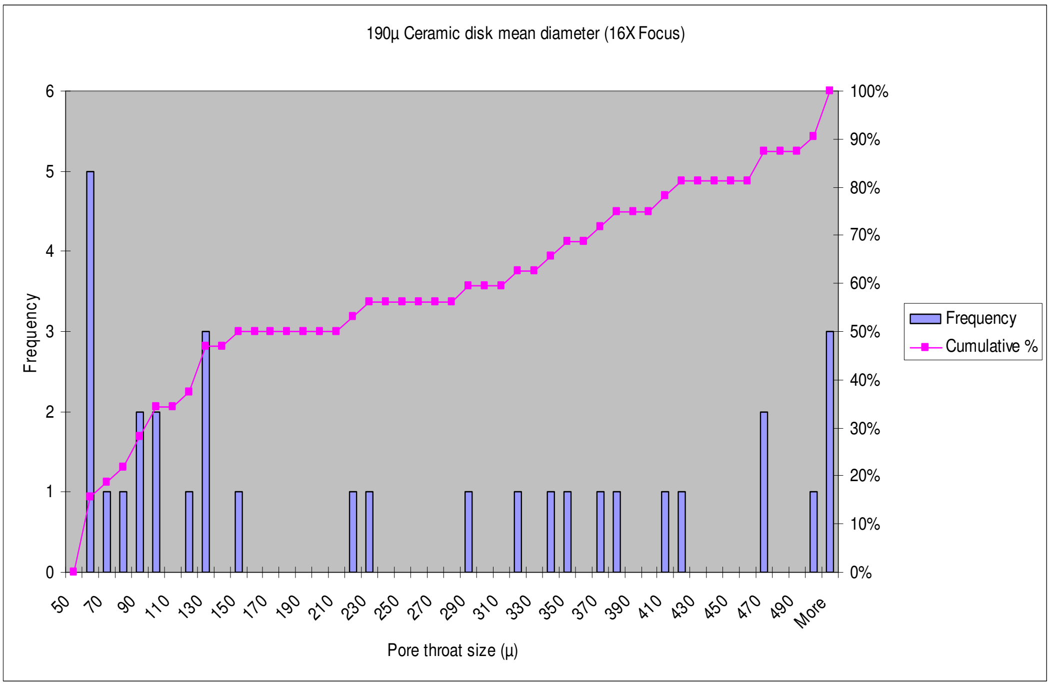

Figure A-21: 190µ Ceramic Disk Mean Diameter (16X Focus)

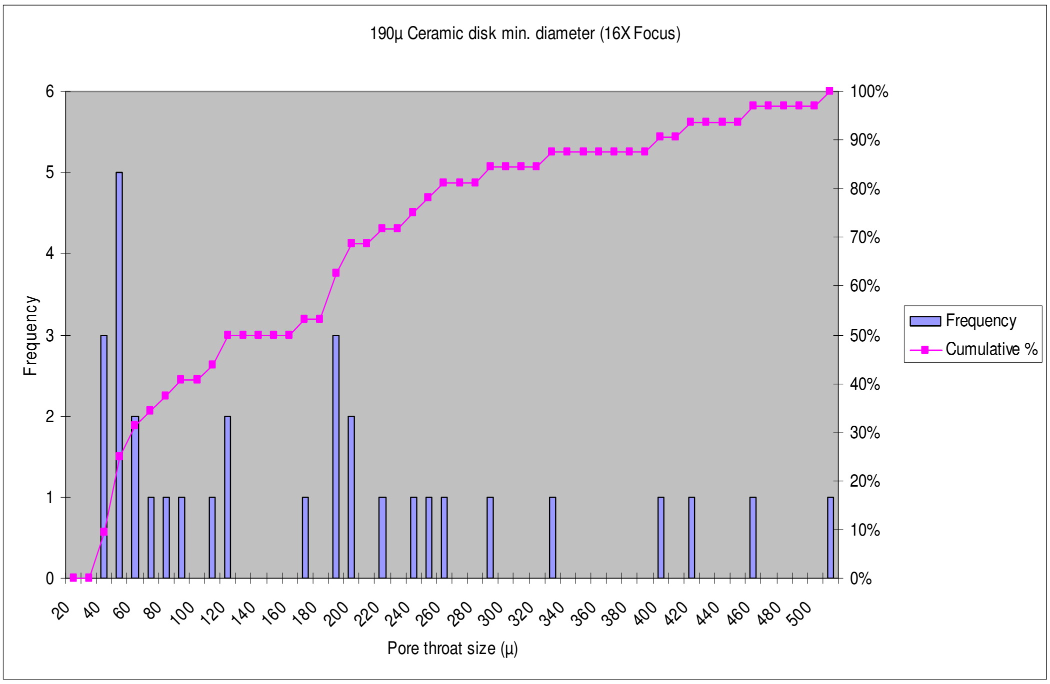

Figure A-22: 190µ Ceramic Disk Minimum Diameter (160X Focus)

APPENDIX B

PPT TEST RESULTS

Table B-1: PPT Test Results

TEST NO	Ceramic disk pore size	$CaCO_3$ particle size	Fluid	$CaCO_3$ Con. (lb/bbl)	Pressure (psi)	T (F)	PPT 7,5 min (ml)	PPT 30 min(ml)	S.Filtration Rate ml/sec$^{1/2}$	Spurt Loss (ml)	Total Filtration (ml)
1	20	$CaCO_3$ C	Flo-Pro	30	300	75	11.8	15.8	2.9	15.6	31.6
2	20	$CaCO_3$ B	Flo-Pro	30	300	75	12.8	16.5	2.7	18.2.2	33
3	20	$CaCO_3$ B&C	Flo-Pro	30	300	75	8.6	12.6	2.9	9.2	25.2
4	20	$CaCO_3$ C	Flo-Pro	10	300	75	20.8	24.8	2.9	33.6	49.6
5	20	$CaCO_3$ C	Flo-Pro	20	300	75	13.8	17.6	2.8	20	35.2
6	20	$CaCO_3$ C	Flo-Pro	40	300	75	12	16.2	3.1	15.6	32.4

Table B-1: PPT Test Results (cont')

7	20	$CaCO_3$ C	Flo-Pro	60	300	75	12.6	17.2	3.4	16	34.4
8	35	$CaCO_3$ C	Flo-Pro	10	300	75	34.6	38.8	3.1	60.8	77.6
9	35	$CaCO_3$ C	Flo-Pro	20	300	75	28.8	33.6	3.5	48	67.2
10	35	$CaCO_3$ C	Flo-Pro	30	300	75	19	23	2.9	30	46
11	35	$CaCO_3$ C	Flo-Pro	40	300	75	15	20.4	3.9	19.2	40.8
12	35	$CaCO_3$ A&B&C	Flo-Pro	30	300	75	11.6	14.4	2	17.6	28.8
13	35	$CaCO_3$ C	Flo-Pro	30	300	75	18.2	22.2	2.9	28.4	44.4
14	35	$CaCO_3$ B	Flo-Pro	30	300	75	20.2	24.4	3.1	32	48.8
15	35	$CaCO_3$ B&C	Flo-Pro	30	300	75	12.2	16.2	2.9	16.4	32.4
16	35	$CaCO_3$ C	Flo-Pro	60	300	75	28.4	33.8	3.9	46	67.6
17	60	$CaCO_3$ C	Flo-Pro	10	300	75	21	24.4	2.5	35.2	48.8
18	60	$CaCO_3$ B	Flo-Pro	10	300	75	40.4	45.4	3.7	70.8	90.8
19	60	$CaCO_3$ A	Flo-Pro	10	300	75	275	275	0	550	550
20	60	$CaCO_3$ C	Flo-Pro	20	300	75	15.4	19.6	3.1	22.4	39.2
21	60	$CaCO_3$ B	Flo-Pro	20	300	75	25.8	28.6	2	46	57.2

Table B-1: PPT Test Results (cont')

22	60	$CaCO_3$ A	Flo-Pro	20	300	75	275	275	0	550	550
23	60	$CaCO_3$ C	Flo-Pro	30	300	75	13.8	18	3.1	19.2	36
24	60	$CaCO_3$ B	Flo-Pro	30	300	75	19.6	23.1	2.6	32.2	46.2
25	60	$CaCO_3$ A	Flo-Pro	30	300	75	275	275	0	550	550
26	60	$CaCO_3$ C	Flo-Pro	40	300	75	11.8	16.6	3.5	14	33.2
27	60	$CaCO_3$ B	Flo-Pro	40	300	75	17.2	21.4	3.1	26	42.8
28	60	$CaCO_3$ A	Flo-Pro	40	300	75	275	275	0	550	550
29	60	$CaCO_3$ A&B&C	Flo-Pro	30	300	75	21.8	26.8	3.7	33.6	53.6
30	60	$CaCO_3$ B&C	Flo-Pro	30	300	75	15.8	20.6	3.5	22	41.2
31	60	$CaCO_3$ A&B	Flo-Pro	30	300	75	35.2	39.8	3.4	61.2	79.6
32	60	$CaCO_3$ A&C	Flo-Pro	30	300	75	26.2	30.2	2.9	44.4	60.4
33	60	$CaCO_3$ C	Flo-Pro	60	300	75	12.4	17.6	3.8	14.4	35.2
34	60	$CaCO_3$ B	Flo-Pro	60	300	75	13.4	17.4	2.9	18.8	34.8
35	60	20%	Flo-Pro	30	300	75	12.2	16.2	2.9	16.4	32.4
36	60	40%	Flo-Pro	30	300	75	14.8	19	3.1	21.2	38

Table B-1: PPT Test Results (cont')

37	60	60%	Flo-Pro	30	300	75	14	17.8	2.8	20.4	35.6
38	60	80%	Flo-Pro	30	300	75	14.2	18	2.8	20.8	36
39	60	%50>44m & 50% mean	Flo-Pro	30	300	75	16.8	20.6	2.8	26	41.2
40	60	>44 micron	Flo-Pro	30	300	75	275	275	0	550	550
41	90	$CaCO_3$ C	Flo-Pro	10	300	75	275	275	0	550	550
42	90	$CaCO_3$ C	Flo-Pro	20	300	75	275	275	0	550	550
43	90	$CaCO_3$ C	Flo-Pro	30	300	75	275	275	0	550	550
44	90	$CaCO_3$ C	Flo-Pro	40	300	75	275	275	0	550	550
45	90	$CaCO_3$ A&B&C	Flo-Pro	30	300	75	63	65.2	1.6	121.6	130.4
46	90	$CaCO_3$ C	Flo-Pro	30	300	75	275	275	0	550	550
47	90	$CaCO_3$ B	Flo-Pro	30	300	75	40	43.2	2.3	73.6	86.4
48	90	$CaCO_3$ A	Flo-Pro	30	300	75	275	275	0	550	550
49	90	$CaCO_3$ B&C	Flo-Pro	30	300	75	55.6	58.8	2.3	104.8	117.6
50	90	$CaCO_3$ B	Flo-Pro	10	300	75	275	275	0	550	550
51	90	$CaCO_3$ B	Flo-Pro	20	300	75	46.8	50.4	2.6	86.4	100.8

Table B-1: PPT Test Results (cont')

52	90	$CaCO_3$ B	Flo-Pro	40	300	75	38.8	42.2	2.5	70.8	84.4
53	90	$CaCO_3$ B	Flo-Pro	60	300	75	19.4	23.8	3.2	30	47.6
54	150	$CaCO_3$ C	Flo-Pro	10	300	75	275	275	0	550	550
55	150	$CaCO_3$ C	Flo-Pro	20	300	75	275	275	0	550	550
56	150	$CaCO_3$ C	Flo-Pro	30	300	75	275	275	0	550	550
57	150	$CaCO_3$ C	Flo-Pro	40	300	75	275	275	0	550	550
58	150	$CaCO_3$ A&B&C	Flo-Pro	30	300	75	63	64.4	1	123.2	128.8
59	150	$CaCO_3$ C	Flo-Pro	30	300	75	275	275	0	550	550
60	150	$CaCO_3$ B	Flo-Pro	30	300	75	48.6	50	1	94.4	100
61	150	$CaCO_3$ A	Flo-Pro	30	300	75	275	275	0	550	550
62	150	$CaCO_3$ B&C	Flo-Pro	30	300	75	275	275	0	550	550
63	190	$CaCO_3$ C	Flo-Pro	30	300	75	275	275	0	550	550
64	190	$CaCO_3$ B	Flo-Pro	30	300	75	275	275	0	550	550
65	190	$CaCO_3$ A	Flo-Pro	30	300	75	275	275	0	550	550

APPENDIX C

EFFECT OF $CaCO_3$ CONCENTRATION

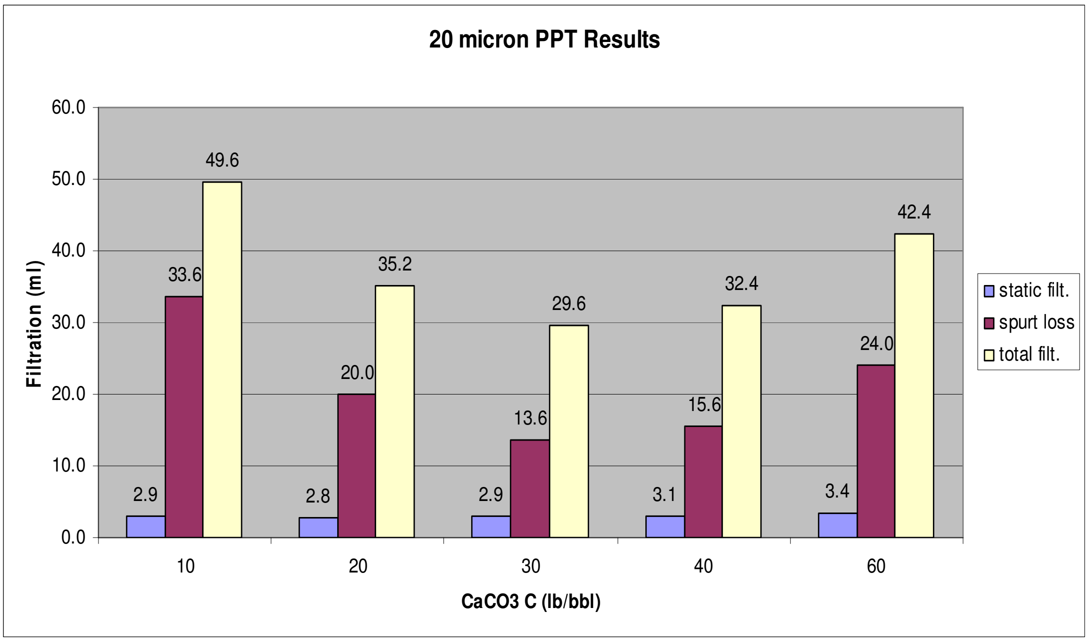

Figure C-1: Effect of Concentration of $CaCO_3$ C (20 µ Ceramic Disks)

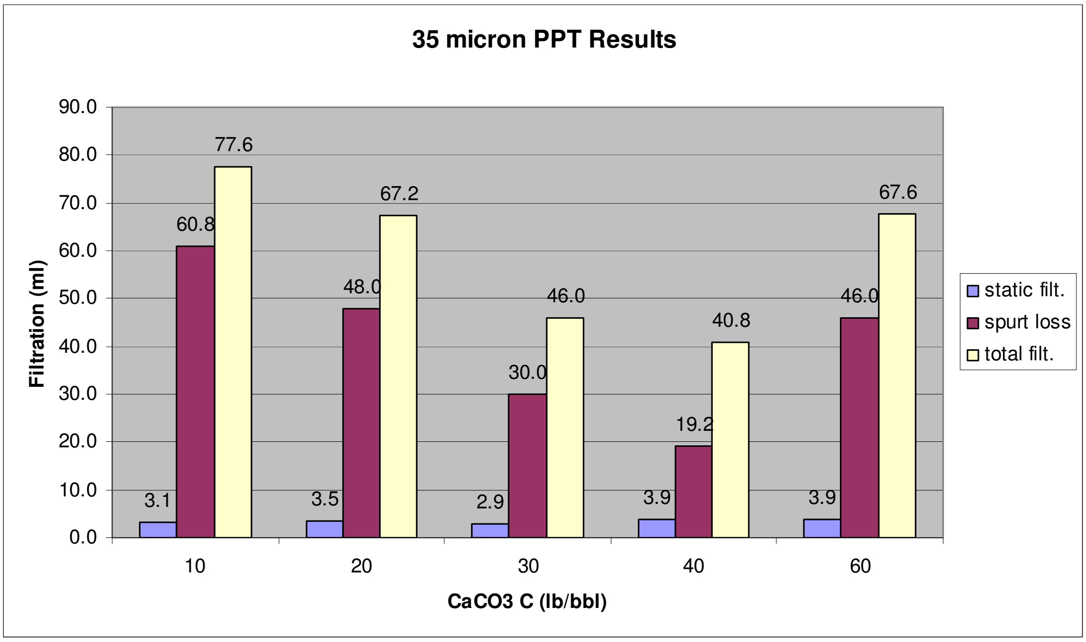

Figure C-2: Effect of Concentration of $CaCO_3$ C (35 µ Ceramic Disks)

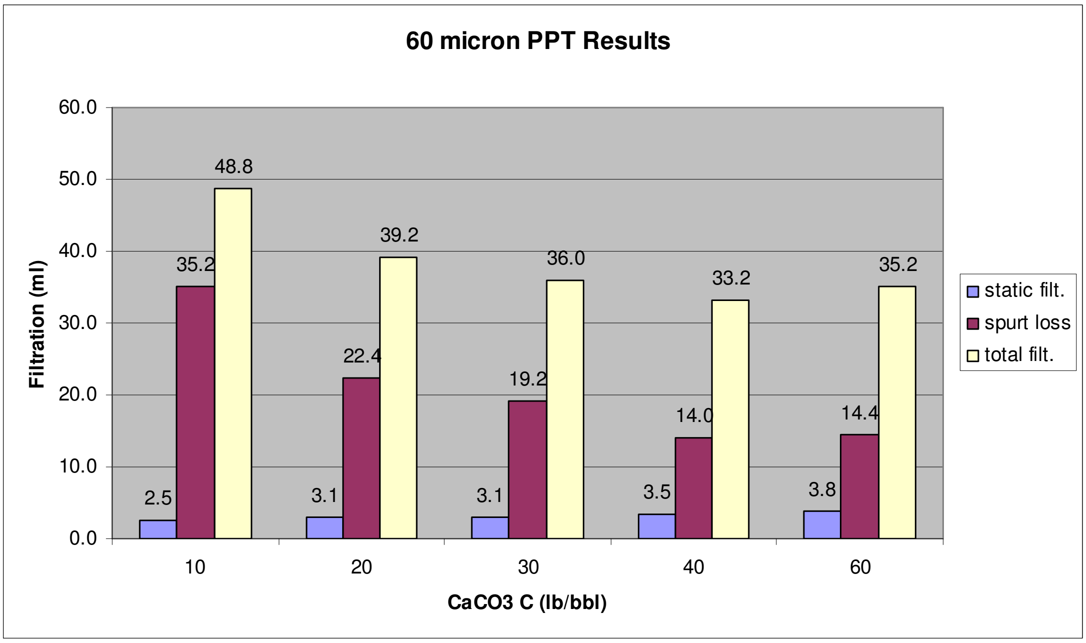

Figure C-3: Effect of Concentration of $CaCO_3$ C (60 µ Ceramic Disks)

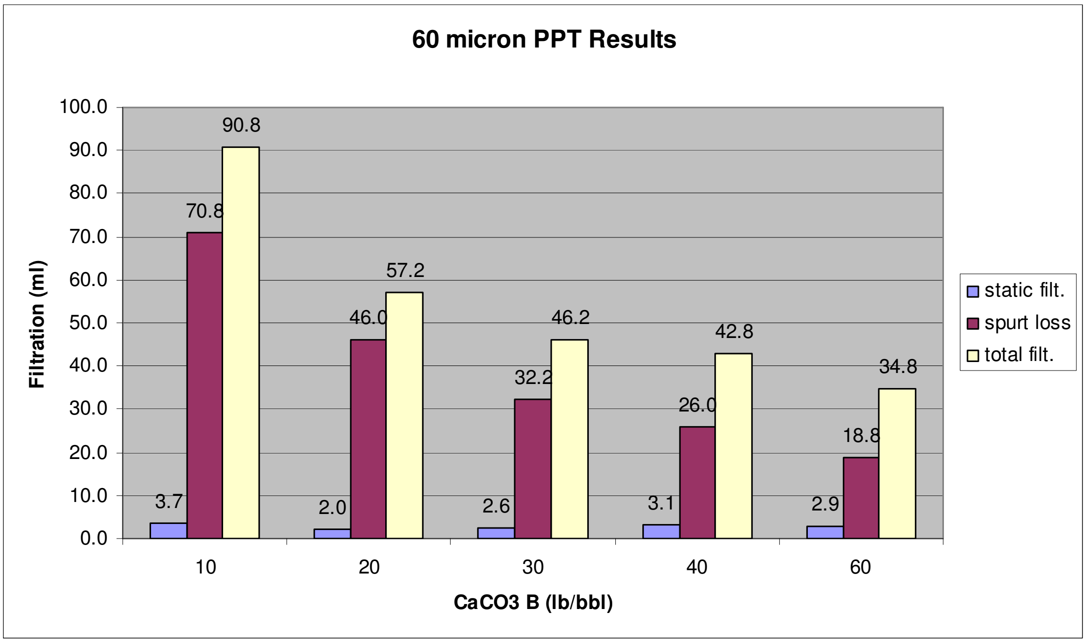

Figure C-4: Effect of Concentration of $CaCO_3$ B (60 μ Ceramic Disks)

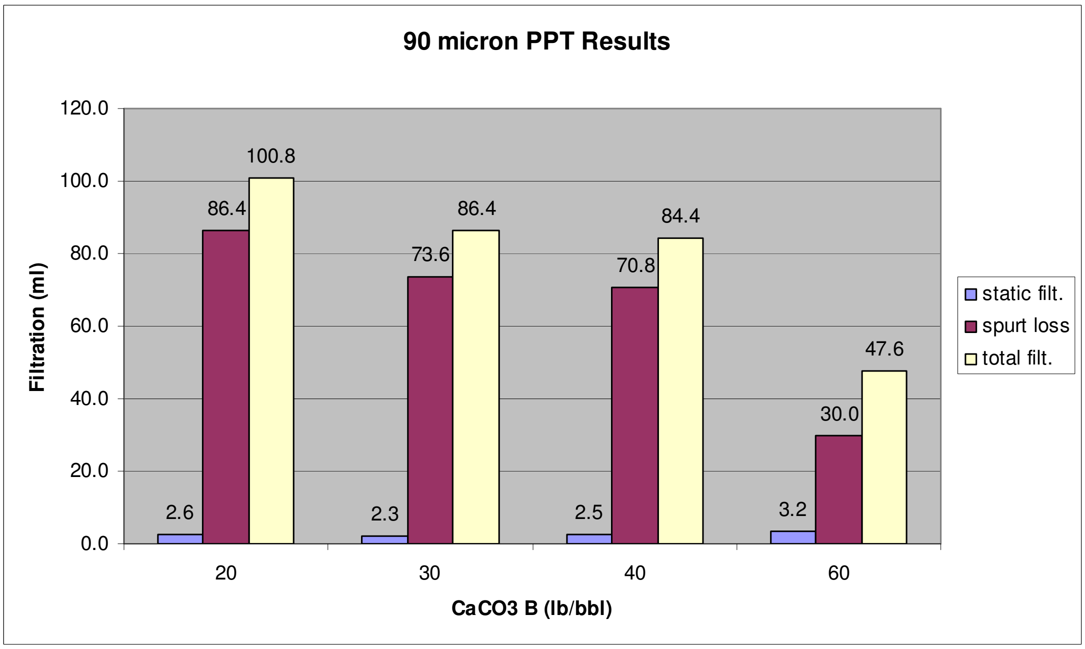

Figure C-5: Effect of Concentration of $CaCO_3$ B (90 μ Ceramic Disks)