Development and Characterization of Synthetic Rock-Like Materials for Drilling and Geomechanics Experiments

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Abstract

The Drilling Technology Laboratory (DTL) have been investigating relationships between vibrations and Rate of Penetration (ROP) using real sedimentary rocks and synthetic rocks for years. However, identical sedimentary rock samples are not easy to obtain in local area (onshore area of eastern Newfoundland). Therefore, this research is focusing on developing synthetic rocks as a substitute for real sedimentary rocks and characterizing petroleum related physical properties of synthetic rocks.

These Rock-Like Materials (RLM) are essentially fine grained concretes based on the use of Portland Cement, fine aggregate, water and related admixtures which can meet the research requirements.

A new approach has been proposed by Prasad (2009) [1] to describe drillability of rocks in a quantitative way with eight parameters which include density, porosity, compressional and shear wave velocities, unconfined compressive strength, Mohr friction angle, mineralogy and grain size. This method is adopted and modified in this research to be suitable for synthetic rocks developed previously. The eight parameters tests are conducted in the drilling technology lab to characterize the properties of the synthetic rocks and to provide the basis for the future work. In this research, standard procedures of making concrete (synthetic rocks) and standard procedures of eight parameters tests have been established with quality assurance.

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DTL	Drilling Technology Laboratory
RLM	Rock-Like Materials
P wave	Compressional Wave
S wave	Shear Sonic Wave
DTC slowness	Compressional Wave Velocity
DTS slowness	Shear Wave Velocity
UCS	Unconfined Compressive Strength
CCS	Confined Compressive Strength
XRD	X-Ray Diffraction
SEM	Scanning Electron Microscopy
QA	Quality Assurance
CREAIT	Core Research Equipment & Instrument Training Network
pVARD	passive Vibration Assisted Rotary Drilling
CI	Cutting Size Index
ROP	Rate of Penetration
WOB	Weight on Bit
BHP	Bottom Hole Pressure

1. Introduction

1.1 Background

Nowadays, petroleum is playing a pivotal role in so many aspects of our lives, not to mention its potential commercial impacts in the energy field. Researches and studies targeting on sedimentary rocks were conducted because oil occurs almost exclusively in sedimentary rocks.

Rocks are a widely varied class of materials with strengths, elastic constants, and other properties varying by one or two orders of magnitude ranging from the weakest to the strongest rock types. Our laboratory have used standard types of rock such as Carthage Marble, Crab Orchard Sandstone, etc. However, within any given rock mass, properties can be heterogeneous and isotropic and vary with both position and orientation within the same formation. The use of natural rock materials for experimental geomechanics studies in Oil and Gas at Memorial University has several difficulties:

1) Experimental studies require a high degree of reproducibility, and many natural rocks have high variability even within a small sample volume
2) Oil and Gas reservoirs are found in sedimentary rocks, and rocks of this type are not local to the onshore portion of Eastern Newfoundland.

However, concrete is a material composed of a Portland Cement based matrix and rock aggregate, and has similar material properties and failure behavior as low permeability sedimentary rocks. Therefore, concrete samples are considered as a reasonable substitute of real rock samples which can meet the following requirements in this research:

1) three different UCS values at 28-days which represent weak, medium and strong sedimentary rocks
2) the materials are easy to get and can be supplied for a long-term
3) all the samples have a high degree of reproducibility

1.2 Research Objectives

The first objective of this work was to develop highly consistent, fine grained concrete materials which can represent characteristic rock types for experimental geomechanics research. The aim was concrete mixes with three different UCS values which represent weak (20MPa), medium (50MPa) and strong (over 80MPa) rock samples respectively. These concrete materials are intended, in particular, for laboratory research, to be cast in sample sizes ranging from a few inches to a few feet in all dimensions.

The second objective of this research was to characterize the drillability of these concrete samples following procedures proposed for the drillability of rock, in particular tests proposed by Prasad (2009) and described and reviewed in detail in a later chapter. In summary, Prasad proposed that the drillability of rock can be characterized as a combination of density, porosity, compression and shear sonic wave velocities, unconfined compressive strength, Mohr friction angle, mineralogy, and grain size. This objective was pursued by studying all these eight parameter on the three concrete materials developed.

The third objective was to establish standard procedures for quality assurance in the laboratory production of the concrete materials to serve in laboratory studies as analogs for rocks.

1.3 Significance of this research

1.3.1 Convenient substitute of real rock in experiments

The research was focused on researching a number of rock analogue materials based on fine rock aggregates and Portland cement. Through parametric batch mixing and materials testing, a suite of standard materials were developed as potential replacements for actual rock that have the desired range of strength, elastic properties, abrasivity, elastic wave velocities, and failure behavior. This will provide for a standard set of materials that may represent rock types ranging from weak shales, intermediate strength siltstones and low permeability sandstones to strong crystalline rocks in experimental geomechanics. This will overcome the general lack of available weak and intermediate strength sedimentary rocks for experimental studies onshore in the province of NL and provide for a high degree of Quality Assurance and experimental reproducibility for geomechanics studies.

1.3.2 Provide basis to compare with real rock

Experimental geomechanics using real rock specimens is normally limited due to the high cost of acquiring materials and the often limited amount available (e.g. from drill cores, etc.) development and refinement of experimental facilities, data analysis procedures. Conversely, these “synthetic rock” materials would provide almost unlimited specimens which could also be cast into convenient shapes for experiments and include such features as imbedded instrumentation, internal flow channels, piezometers, etc. This also can facilitate comparison with studies at other geomechanics laboratories.

1.3.3 Establish the standard procedure for quality assurance

A reason for using concrete sample as a substitute of real rock is a high level of reproducibility of material properties can be achieved so that with the Quality Assurance (QA) playing an important role in casting consistent and reproducible concrete samples. During this research, a series of quality assurance procedures following standard practice for concrete were established to provide the confidence of repeatability studies and reproducibility studies. For instance, sieve analysis, moisture content calculation and internal vibration all became standard procedures in mixing concrete so that repeatability and reproducibility can be improved. The concept of sieve analysis, moisture content calculation and internal vibration are introduced in next three paragraphs.

Sieve analysis [2] is a standard procedure to assess particle size distribution in the concrete industry, which can control the quality of aggregates in this project. The sieve analysis results can be used to characterize the sand used in every batch of concrete cast because similar sieve analysis results provide the similar material performance.

Moisture content calculation [3] is used to calculate the moisture content in fine aggregate (sand). Fine aggregate is usually stored in an open area. Therefore the water content can be affected by the weather. Moisture content is determined by removing the water in fine aggregate through heating. The difference in weight is the water content. The water/cement ratio used in adjusted accordingly.

Internal vibration was introduced to improve the workability of mix to achieve a better consolidation. Higher workability will provide consistent rock samples compared with the poor consolidation. In high strength design, the internal vibration made a significant improvement in workability when the mix can not be consolidated without vibration.

1.4 Limitations

1.4.1 Uncertain curing conditions might affect strength of concrete

Concrete samples are usually cast in laboratory with certain procedures and strict curing environments. However, large quantity specimens may require to be cast at the field in future work. It is well known that strength of concrete is highly related to curing conditions i.e. curing temperature, humidity and time. This raises the problem of control curing environments to achieve desired UCS values outside the laboratory. In addition, curing with water saturated with calcium hydroxide ($Ca(OH)_2$) to prevent leaching of this compound (a product of hydrolysis in curing) from the concrete as ASTM Standard C511-09 suggests is difficult to implement in field condition.

1.4.2 Methods used in characterization of rock might not compatible with concrete

As we are using concrete as a convenient material to use instead of real rock, it is realized that we must characterize the concrete in a way that will make it possible to compare our drilling test results with similar results obtained in real rock, and with simulation/modeling work based on the relevant properties. However, the methods used to characterize properties of real rock cannot always be applied on concrete specimens. For example, porosity is usually calculated using water saturated method which is obviously not suitable for concrete or it will continuously react with water.

1.4.3 Variability issues that can affect the characterization of concrete

Most natural rock was formed millions of years ago, while the age of concrete under consideration here would be only being a matter of months. Whatever way concrete is produced and whatever the environment in which it exists, it is a “work in progress,” i.e. the result of incomplete chemical processes and continuing change. The specification strength of concrete is usually the strength after 28 days of curing, and that depends on the manner in which curing takes place, but strength can continue to increase and other properties continue to change over time. Thus there can be issues of variability of properties in space within concrete specimens, as well as variability over time.

2. Literature Reviews

2.1 Factors Affect the UCS Values

Engineers have developed many ways to strengthen concrete in the field, such as inserting steel bar and introducing additives. However, steel bar and coarse aggregate can not be contained in concrete sample in this research. The fundamental approach is to adjust the ratio between water, sand and cement which are the basic materials of concrete. The investigation of concrete strength affected by the ratio between water, sand and cement has been conducted, and water/cement ratio is the key point of the strength of concrete.

2.1.1 Water/cement ratio

Water/cement ratio, also described as water/binder ratio, is a critical factor affecting the compressive strength of concrete. In fact, by given sand and cementitious material, water/cement ratio affect the properties of concrete by changing the microstructure of concrete. For example, Duff Abrams found the followed relationship between water/cement ratio and compressive strength in 1919 [4]:

$$f_c = \frac{k_1}{k_2^{w/c}}$$

Where w/c represents the water/cement ratio of mix,

and k_1 and k_2 are empirical constants.

Figure 1 gives a direct view of a typical relationship between compressive strength and water/cement ratio for concrete made with a rapid-hardening Portland cement.

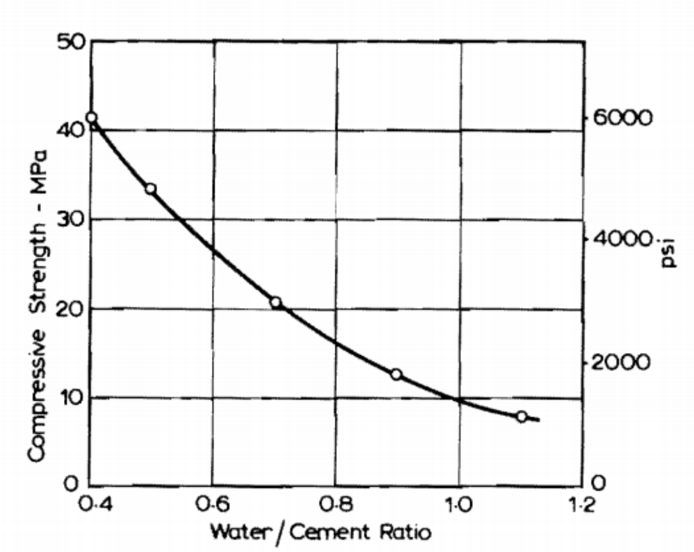


Figure 1 Relation between strength and water/cement ratio [4]

2.1.2 Material Selection

In this project to design and test custom mixes of concrete with three levels of strengths, the choice of raw materials was determined by availability and cost. Pennecon Ltd supported the project in several way including supplying cement, sand

and additives.

2.1.2.1 Sand (fine aggregate)

Aggregate also known as sand and stone, is used in concrete. In general mixing design, both coarse aggregate (＞4.75mm) and fine aggregate (≤4.75mm) were considered. Because drill bits no bigger than 2 inches are used in this project, only fine aggregate was used for materials. Although only fine aggregate was acceptable in this design, particle size distribution is critical to the performance of concrete. Sieve analysis is a practical method to assess particle size distribution in lab, which is the easy way to describe fine aggregate. Black Mountain sand is proper fine aggregate available from Pennecon. Our sieve analysis results of Black Mountain sand are shown in Figure 2 and Table 1.

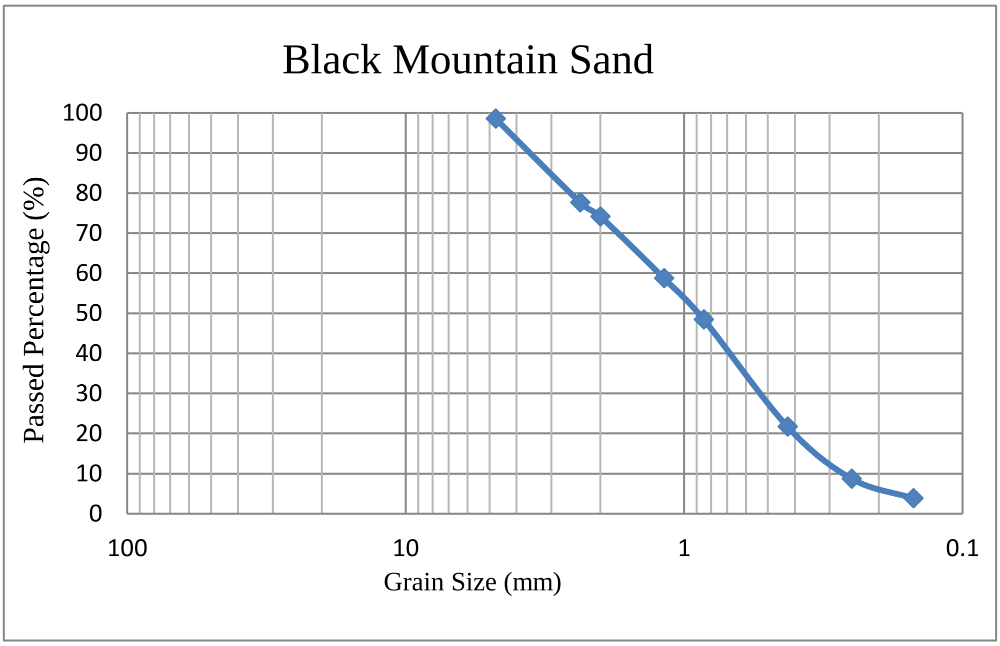


Figure 2 Particle Size Distribution Plot of Black Mountain Sand

Table 1 Sieve Analysis Results of Black Mountain Fine Aggregate

Grain Size (mm)	Passed Mass (g)	Accumulated (g)	Passed Percentage (%)
0.15	66	66	3.80
0.25	85	151	8.71
0.425	225	376	21.68
0.85	464	840	48.44
1.18	178	1018	58.71
2	268	1286	74.16
2.36	60	1346	77.62
4.75	363	1709	98.56
	25	1734	100

2.1.2.2 Cement

Holcim Type GU cement is the most common cement used in local construction, compliant with ASTM standard C 1157 and available in Pennecon. All the information of this cement can be found in the data sheet online [5].

2.1.2.3 Water Reducer

Water reducer plays an important role in casting high strength concrete because it can change the rheology of the concrete mix to provide a good workability.

WRDA® 82 is a water-reducing admixture meeting the specification of ASTM C494 Type A and D. The data sheet of WRDA® 82 can be accessed online [6].

Dosage

Addition Rate: The addition rate range of 3 to 5 fl oz/100 lbs (195 to 326 ml/100 kg) of cement or cementitious is typical for most applications. However, addition rates of 2 to 10 fl oz/100 lbs (130 to 652 ml/100 kg) of cement or cementitious may be used if local testing shows acceptable performance.

WRDA® 82 is compatible with most Grace Company's admixtures as long as they are added separately to the concrete mix.

2.1.2.4 Superplasticizer

Superplasticizer, also known as high range water-reducing admixture, is usually used in concrete mix with low water/cement ratio. This chemical admixture can produce a high slump flowable concrete without lowering the compressive strength.

Daracem® 19 is a high range water reducer meeting the specification of ASTM C494 Type A and F. Data sheet of Daracem® 19 can be accessed online [7].

Dosage

Addition Rate: Addition rates of Daracem® 19 can vary with type of application, but will normally range from 6 to 20 fl oz/100 lbs (390 to 1300 ml/100 kg) of cement. In most instances the addition of 10 to 16 fl oz/100 lbs (650 to 1040 mL/100 kg) of cement will be sufficient.

Daracem® 19 is compatible with most Grace Company's admixtures as long as they are added separately to the concrete mix

2.1.2.5 Silica fume

Silica fume, also referred to microsilica, is introduced as cementitious material to increase compressive strength based on its chemical and physical properties. Silica fume is highly reactive, which can speed up the reaction with the calcium hydroxide produced by the hydration of Portland cement. Furthermore, the very small particles of silica fume can fill the space between large particles to improve packing. Information about Force 10,000® D can be accessed online [8].

Dosage

Based on the working principle of silica fume, Aitcin [9] suggests that the dosage of silica fume should be 25%-30%, and silica fume is needed to neutralize the lime produced by the hydration of Portland cement. In addition, higher dosage of silica fume will provide a higher compressive strength. The relation between the compressive strength and different contents of silica fume is shown in Figure 3 [10]. However, high dosage of silica fume is not usually used in the field because silica fume consumes a large quantity of superplasticizer. In our tests it was found that silica fume at 15% of all cementious materials (by mass) was the appropriate amount to use with water cement ratio of 0.35 with superplasticizer.

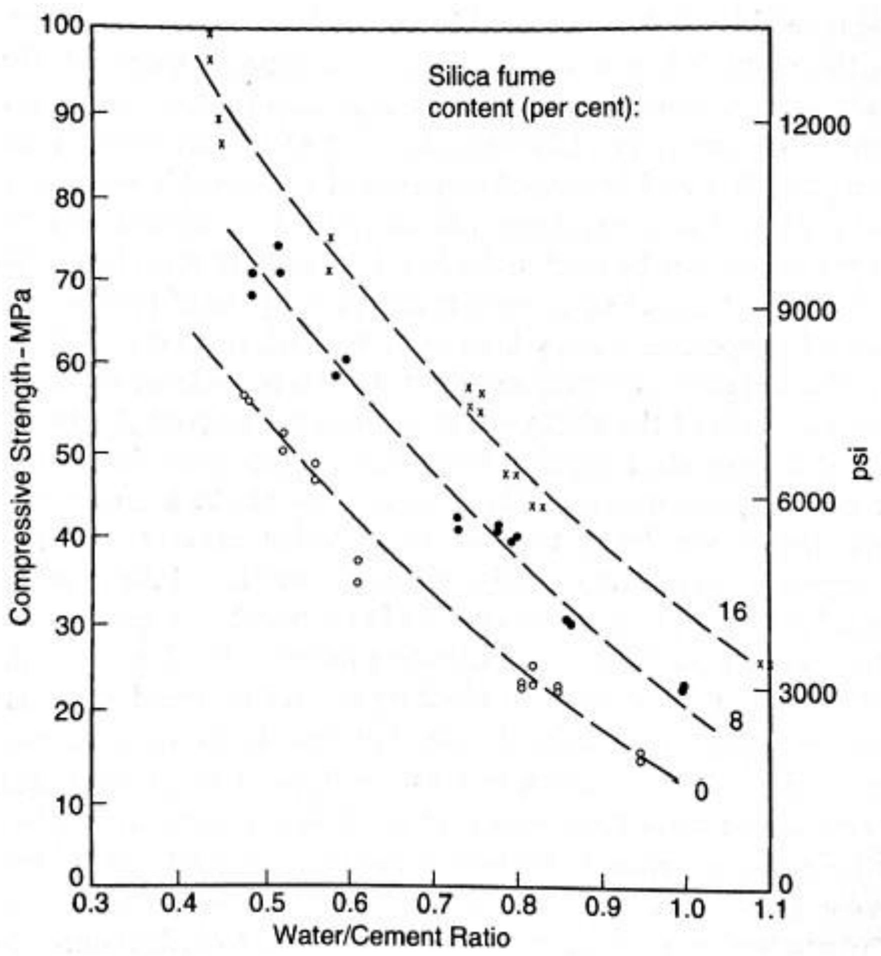


Figure 3 Relationships between the Compressive Strength and the Water/Cement Ratio for Concretes with Different Contents of Silica Fume [10]

The microsilica Force 10000® D is compatible with WRDA® series water reducer and Daracem® series superplasticizer, which is clarified in specification data of Force 10000® D.

2.1.2.6 Information on selected materials

Regarding the investigation above, the selected materials used in the three strengths of concrete are listed in Table 2.

Table 2 Information of selected materials

Materials	Brand
Fine Aggregate	Black Mountain Sand
Cement	Holcim Type GU
Water Reducer	WRDA® 82
Superplasticizer	Daracem® 19
Silica Fume	Force 10000® D

2.2 Drillability

Prasad (2009) has proposed a quantitative methodology to describe rock materials in terms of their “drillability” as a means of predicting rates of penetration and wear, which are closely linked to strength [11] and abrasivity. This methodology includes 8 material properties (density, porosity, compressional and shear wave velocities, unconfined compressive strength, Mohr friction angle, mineralogy, and grain sizes) which can be represented on a Spider Plot as given in Figure 4 as a drillability curve relating all 8 properties. All eight parameters are converted in a scale of 0 to 8; value 0 represents very soft rock and value 8 represents hard rock, ideally.

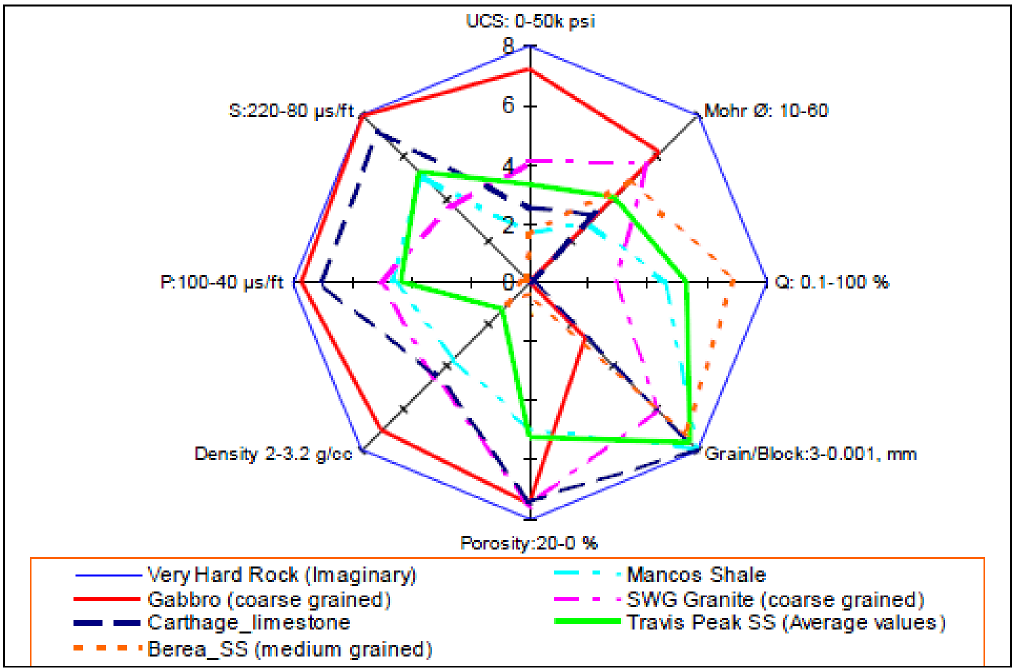


Figure 4 Spider Plot showing drillability curves for several well-known and hypothetical rock types

In eight parameters tests, both non-destructive tests and destructive tests are involved. The series of experimental tests have to be organized with procedures and test materials. In eight parameters tests, density, porosity, P-wave velocity and S-wave velocity are non-destructive tests which should be tested before other destructive tests. UCS and Mohr friction angle tests have crush the samples to obtain the value from tester, which require large amount of specimens. Grain size and mineralogy tests are only involved with fine aggregate because concrete is different from real rock and modifications of tests are necessary. The brief ideas each test are shown as following and more detailed experimental procedures will be described in later chapters.

2.3 Eight Parameters Characterizations

Drillability of rock is a concept usually used to describe the physical properties of rock, which reflect the rate of penetration under certain drilling condition. It can be expressed with a lot of parameters such as hardness, elastic constants, mineral composition, density, permeability, UCS and CCS. Drillability is not a parameter commonly referred in the field because there is no unified standard to describe drillability. However, rock properties like UCS, hardness and density have been frequently used to optimize drilling parameters. In this research, characterization of rock (concrete) will be conducted with eight parameters which proposed by Prasad in 2009. These parameters are density, porosity, compressional and shear wave velocities, unconfined compressive strength, Mohr friction angle, mineralogy, and grain size.

2.3.1 Density

Density is a common physical property which can be determined by mass over volume. Density of same rock can be varied because pore space in rock and crack in matrix is different. Hence, density is often related to the porosity and strength because pore space increase porosity and weaken strength. However, density can be a very valuable parameter to describe the physical property of concrete. As mentioned previously, concrete designed in this research only contains water, fine aggregate and cement. The density of concrete can be an indicator of strength and composition due to the density of individual materials are fixed. Since density of cement (3.15 g/cm^3) is higher than the water (1 g/cm^3) and fine aggregate (2.65 g/cm^3), stronger concrete is consist of more cementious material and less water while density is increasing.

2.3.2 Porosity

Porosity is a useful parameter in drilling field, which is target data to acquire in wireline well log. Porosity is related to the elastic properties because the pore space it where the stress concentrated on during deformation. The vacuum saturation method [12] is commonly used in the lab for porosity measurement of concrete, which involves complicated apparatus which is not available. Therefore porosity is calculated by the percentage of pore area over total thin section area under microscope in this research.

2.3.3 P wave and S wave velocities

The compressional wave, also referred to primary wave (P wave), is the fastest wave can travel in rocks. This feature allows it to be commonly used to characterize rock properties in lab and field for drilling purpose. As the P wave velocity will drastically drop in gas media it can be an indicator to estimate porosity because P waves travel faster in solid than in air. The shear wave (S wave), also referred to secondary wave, travels slower than the P wave, and not like P wave, it only travels in solid. The illustration of P wave and S wave are shown in Figure 5.

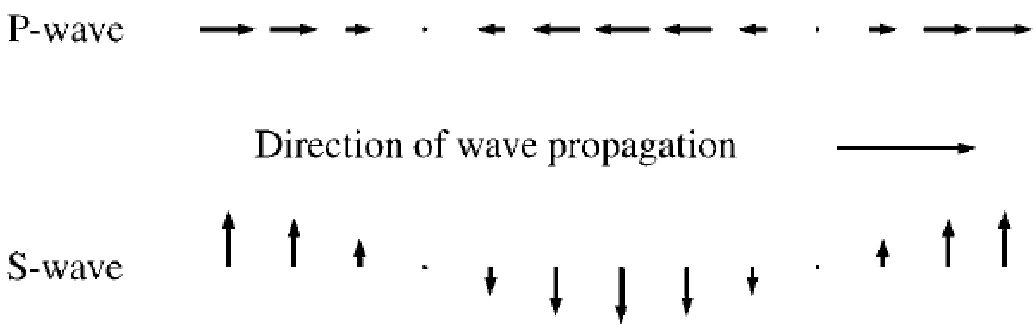


Figure 5 Particle motion and propagation direction of P wave and S wave

Regarding the book “Petroleum Related Rock Mechanics”, wave velocities are closely related to elastic properties of rock. The relationships between wave velocities and elastic modulus are shown in Equation 2.1 and Equation 2.2 [13].

$$v_p = \sqrt{\frac{\lambda + 2G}{\rho}}$$

Equation 2.1 Expression of P wave velocity

Where v_p is P wave velocity,

λ is Lame’s parameter,

G is shear modulus,

ρ is density of rock.

$$v_s = \sqrt{\frac{G}{\rho}}$$

Equation 2.2 Expression of S wave velocity

Where v_s is S wave velocity.

2.3.4 UCS

Unconfined Compressive Strength (UCS) value is the most commonly used parameter in both lab and industry. UCS represents maximum loading capacity of a material under uniaxial loading condition before failure. UCS often referred as strength of rock which closely connected to large amount of properties. In the lab, Young's Modulus can also be calculated using data obtained from UCS tests. Young's modulus equals to the slope of axial curve shown in Figure 6.

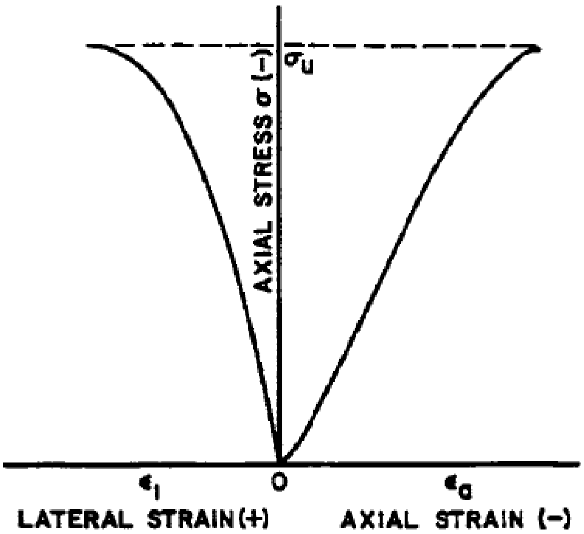


Figure 6 The stress-strain curve [4]

The UCS test usually only gives the axial curve in the stress-strain curve; however, lateral curve can be acquire by installing a pair of strain gauges during the UCS tests. With the data of lateral curve and axial curve, Poisson's ratio can be calculated from Equation 2.3.

$$\nu = -\frac{\text{slope of axial curve}}{\text{slope of lateral curve}} = -\frac{\text{E}}{\text{slope of lateral curve}}$$

Equation 2.3 Poisson's ratio calculation

Where ν is Poisson's ratio,

E is Young's modulus.

More elastic constants such as shear modulus, lame's parameter and bulk modulus can be calculated after Young's modulus and Poisson's ratio are acquired. Equation 2.4, Equation 2.5 and Equation 2.6 can be derived regarding the relationship between elastic moduli shown in Figure 7 [14].

$E = 3K(1-2\nu)$	$K = \lambda\frac{1+\nu}{3\nu}$	$\frac{\lambda}{\lambda+G} = 2\nu$
$E = 2G(1+\nu)$	$K = \frac{2}{3}G\frac{1+\nu}{1-2\nu}$	$\frac{G}{\lambda+G} = 1-2\nu$
$E = \frac{9KG}{3K+G}$	$K = \lambda + \frac{2}{3}G$	$\frac{\lambda+2G}{\lambda+G} = 2(1-\nu)$
$E = G\frac{3\lambda+2G}{\lambda+G}$	$K = \frac{GE}{9G-3E}$	$\frac{3\lambda+2G}{\lambda+G} = 2(1+\nu)$
$E = \frac{\lambda}{\nu}(1+\nu)(1-2\nu)$	$\frac{\lambda}{G} = \frac{2\nu}{1-2\nu}$	$\frac{3\lambda+4G}{\lambda+G} = 2(2-\nu)$
$H = \lambda + 2G$	$H = K + \frac{4}{3}G$	$\nu = \frac{3K-2G}{2(3K+G)}$
$H = E\frac{1-\nu}{(1+\nu)(1-2\nu)}$	$H = 2G\frac{1-\nu}{1-2\nu}$	$H = 3K\frac{1-\nu}{1+\nu}$

Figure 7 Correlations between elastic moduli

$$K = \frac{E}{3(1-2\nu)}$$

Equation 2.4 Bulk modulus expression

Where K is bulk modulus.

$$\lambda = \frac{E\nu}{(1+\nu)(1-2\nu)}$$

Equation 2.5 Lame’s parameter expression

Where λ is lame’s parameter.

$$G = \frac{E}{2(1+\nu)}$$

Equation 2.6 Shear modulus expression

Where G is shear modulus.

UCS has an empirical relationship with tensile strength, which is UCS value equals to ten times of tensile strength. Recently research gave the similar results and provided the numerical correlation between UCS and indirect tensile strength shown in

Equation 2.7 [15] (Correlation between Unconfined Compressive Strength and Indirect Tensile Strength of Limestone Rock Samples).

$$\mathrm{UCS\ (MPa)} = 9.25\ \mathrm{BTS}^{0.947}$$

Equation 2.7 Experimental correlation between UCS and indirect tensile strength

Where BTS is Brazilian Tensile Strength

2.3.5 Mohr friction angle

Mohr friction angle in a parameter in Mohr-Coulomb failure criteria which can be obtained by confined compressive strength (CCS) tests. The mathematic relationship between parameters of Mohr-Coulomb failure envelope is shown in Figure 8 and failure envelope can be expressed in Equation 2.8.

$$\tau = \mathrm{C} + \sigma\tan\varphi$$

Equation 2.8 Mohr-Coulomb failure criteria

Where:

C is cohesion stress,

σ is normal stress on the failure plane,

τ is shear stress on the failure plane,

φ is the friction angle.

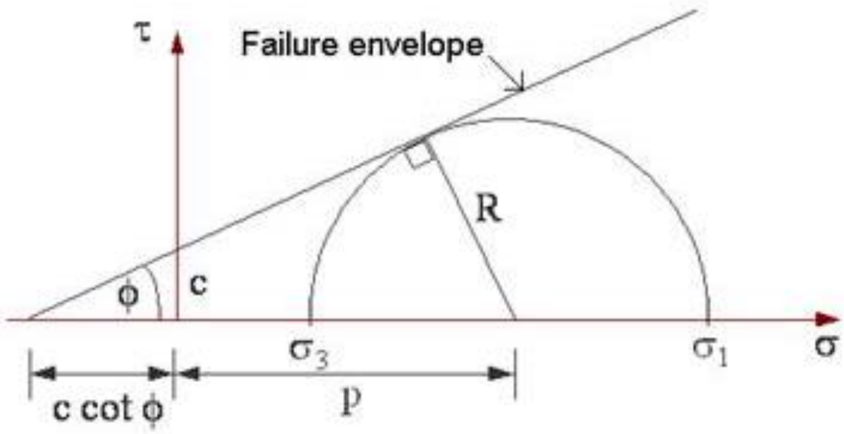


Figure 8 Mohr-Coulomb failure envelope

2.3.6 Grain size

Grain size, the microstructure of rock is characterized using several properties: grain size distribution, shape of grain and degree of grain orientation. A lot of studies have researched the effect of both grain size and block size of rock; however, grain size is the only parameter used in this research. Many tests in field and lab experiments have agreed that grain size is related to strength of Rock-Like Materials such as Figure 9 [16].

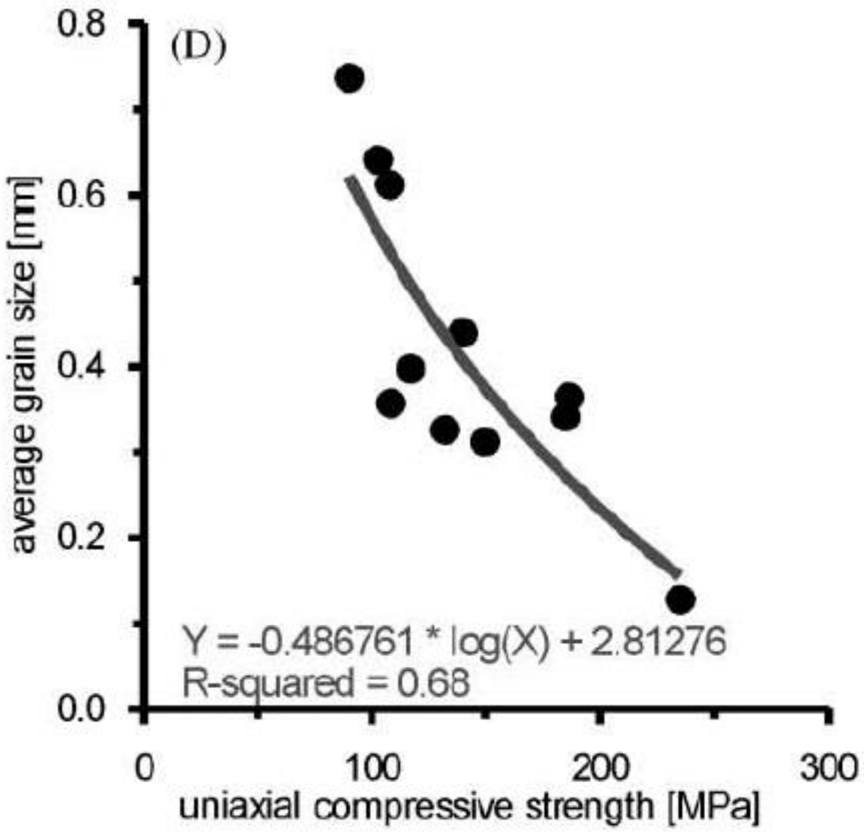


Figure 9 Correlation between UCS and average grain size

2.3.7 Mineralogy

The mineralogy of rocks is a key factor that affects the wear of the bit during drilling process. As in Prasad's paper, mineralogy analysis is concentrated on quartz which is hardest mineral in common rock. Quartz is considered as an abrasive mineral in rock, and a relationship between quartz content and abrasivity is shown in Equation 2.9 [17].

$$F = \frac{EqQtz \times Ø \times BTS}{100} \ (N/mm)$$

Equation 2.9 F-abrasivity factor expression

Where F is the Schimazek's abrasive wear factor (N/mm),

EqQtz is the equivalent quartz volume percentage, Ø is the grain size (mm),

BTS is indirect Brazilian tensile strength.

As indicated by this equation the abrasivity of rock is linked to the quartz content in conjunction with the grain size and strength of the rock.

3. Methodology

3.1 Approach to the Development of Concrete Designs

In order to obtain concrete samples desired, a series of systematic experiments were conducted at Pennecon's lab where materials and tools can be provided. The objectives of this research are to develop three strengths of concrete and characterize samples with eight parameters. The methodology of developing three strengths is critical because the concrete samples will be the basis of later experiments. However, the eight parameters cannot be individually controlled during preparation of the sample though concrete is a synthetic substitute. Unconfined Compressive Strength (UCS) values as an important parameter in both rock property and concrete design should be the controlled parameter of design concrete batches.

3.1.1 Facilities used

The author was awarded a MITACS Internship with Pennecon Limited, which funded collaboration with its subsidiary, Capital Precast Limited, with use of its laboratory facilities and also materials provided by it: Portland Cement and fine aggregate - the latter produced from material from its nearby Black Mountain quarry. Much of the preparation and curing of the initial mixes of concrete and testing for Unconfined Compressive Strength (UCS) were conducted at Capital Precast. All other property tests and later preparation of concrete samples were conducted in the Engineering

Laboratories at Memorial University.

3.1.2 Initialize the design parameters

As mentioned before, water/cement ratio is a key value to determine concrete strength and concrete used in this research will require three ingredients as water, fine aggregate and cement. Aggregate/cement ratio can be calculated with a given water/cement ratio. Therefore, some ideas are borrowed from the method of mixing concrete called Absolute Volume Method of Concrete Mix Design.

The idea of absolute volume method is shown in Equation 3.1.

$$volume\ of\ cement + volume\ of\ aggregate + volume\ of\ water = 1\ m^3\ concrete \quad \text{(Equation 3.1)}$$

Where,

$$volume\ of\ material = \frac{mass\ of\ material}{specific\ gravity\ of\ material \times density\ of\ water}$$

When water/cement ratio is 0.45,

S.G. of cement is 3.15,

S.G. of fine aggregate is 2.65,

Mass of cement is 500 kg,

So the Equation 3.1 can be converted into Equation 3.2

$$\frac{500}{3.15 \times 1000} + \frac{mass\ of\ aggregate}{2.65 \times 1000} + \frac{0.45 \times 500}{1 \times 1000} = 1 \quad \text{(Equation 3.2)}$$

Based on Equation 3.2, the mass of aggregate is 1633 kg, and the aggregate/cement ratio is 3.26. However, aggregate/cement ratio was approximated at 3 to make it easy to calculate. Regarding the calculation above, aggregate: cement: water (A: C: W) is confirmed at 3: 1: 0.45, and this is the first design to start with. More designs will be developed by adjusting water/cement ratio after UCS value of first design confirmed.

3.1.3 Systematic batches by adjusting parameters

After the first batch of concrete were cast based on aggregate: cement: water ratio of 3: 1: 0.45, the UCS tests were conducted at four different test ages which are 7, 14, 21 and 28 days. The UCS results of first batch are shown in Table 3.

Table 3 UCS values of first batch

Test Ages	UCS Values (MPa)
7 days	29.8
14 days	31.9
21 days	34.5
28 days	41.1

Some comments can be made regarding this batch of mix:

1) The 28-days strength seems acceptable as medium strength design.
2) When this batch was cast, the mixture had a low workability which was really too dry to cast it.
3) Higher strengths concrete might be obtained by lower water/cement ratio.

More batches were made by adjusting water/cement ratio and sand content are shown in Table 4.

Table 4 Systematic batches to develop three strength mixes

Trial No.	A: C: W	UCS (MPa)	Additive	Remark
Medium-1	3: 1: 0.45	41.1	No	dry
Medium-2	3: 1: 0.45	48.9	Yes	good workability
High-1	3: 1: 0.40	60.3	Yes	dry
High-2	3: 1: 0.35	62.1	Yes	dry
High-3	3: 1: 0.30	30.1	Yes	high workability
High-4	3: 1: 0.30	94.9	Yes	low workability
Low-1	4: 1: 0.6	37.6	No	good workability
Low-2	6: 1: 1	21.6	No	good workability

Based on the Table 4, all three strengths were developed from the first batch (batch number Medium-1) by altering parameters such as water/cement ratio and sand content. Furthermore, additives to improve workability and to increase strength were introduced in these batches, which include water reducer, superplasticizer and silica fume.

3.1.4 Additives to improve workability and to increase strength

Two main kind of additives were used in this research, one is water reducer which is used to improve the workability of mix; another one is silica fume which is a fine particle to increase the strength of concrete.

There were some water reducers are available at Pennecon, WRDA®82 and Daracem®19 are the best choices after a few trial tests. Adding WRDA®82 was the only thing changed in batch number Medium-2 compared to batch number Medium-1. Improving workability of mixes can significantly increase the strength of concrete samples and provide a better performance of mixes. Daracem®19 is added in high strength concrete design as a high range water reducer (superplasticizer). High strength concrete mix has a really low workability because of low water/cement ratio and silica fume. The recommended dosage didn't work for this kind of application and the right dosage of superplasticizer had to be determined through trial tests. Too much (batch number High-3) or not enough superplasticizer will not obtain desired strength.

Silica fume is usually mixed with Portland cement to improve the concrete properties such as strength. Silica fume is extremely fine particles and can fill the space between fine aggregate and concrete matrix to increase the strength of concrete. Also the pozzolanic reaction between silica fume and calcium hydroxide generated from water and cement provides extra bond strength.

3.1.5 Results of trial tests

After these systematic batch trials, three strengths concrete designs have been completed. The results and observations from trial tests are given as following:

1) Regarding the UCS values obtained from Table 4, low strength, medium strength and high strength concrete designs are batch number Low-2, batch number Medium-2 and batch number High-4, respectively.

2) In design of high strength concrete, silica fume is introduced. It's fairly fine

particle size increases strength. Because of the low water/cement ratio and silica fume in high strength concrete design, proper dosage of superplasticizer is the critical in the mixing high strength concrete. Not enough water reducer and superplasticizer will lower the workability of concrete mix so that the mix cannot be moulded. However, too much superplasticizer will result in an extremely low strength of concrete mix (batch number High-3).

3) The batch number High-4 is acceptable design for high strength concrete, however, it is still a dry mix which is difficult to utilize in larger quantity mixing. A more powerful superplasticizer might be the solution.

3.2 Quality Assurance

The three strength concrete samples will be massively used in future experiments as tests materials. The reproducibility is an important feature in these designs because plenty of tests based on these three strength concrete mixes will be conducted in the future. The purpose of quality control is to cast consistent and reproducible concrete samples with standard procedures.

3.2.1 Sieve analysis

Sieve analysis is a standard procedure to assess particle size distribution in the concrete industry, which can control the quality of sand in this research. The sieve analysis results can be used to identify the fine aggregate (sand) used in every single batch of concrete cast because similar sieve analysis results provide the similar material performance. Therefore, sieve analysis tests for every batch of fine aggregate should be conducted with dry material condition before it is used to mix concrete sample. Furthermore, sieve analysis results and test procedures of fine aggregate should meet the requirement ASTM standard C136 (Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates) [18].

Black Mountain fine aggregate, the material used in this research, is required to conduct the sieve analysis test. The 8 sieves used in sieve analysis are mesh number 4, 8, 10, 16, 20, 40, 60 and 100 which aperture diameter corresponding to 4.75 mm, 2.36 mm, 2 mm, 1.18 mm, 0.85 mm, 0.425 mm, 0.25 mm and 0.15 mm. The results of sieve analysis of Black Mountain sand are shown in Table 1 and Figure 2.

3.2.2 Moisture content calculation

The moisture content calculation is used to calculate the moisture content in fine aggregate. Fine aggregate is usually stored in an open area. Therefore the water content can be affected by the weather. Moisture content is determined by removing the water in fine aggregate through heating process. The difference in weight is the water content. The water/cement ratio used in adjusted accordingly by deducting the calculated water content from the total water content.

Standard Procedures of Moisture Content Calculation:

Weigh around 2000 gram sand and record as mass of wet sand

Put sand into a steel pan and top pan on a hot plate to vaporize the water until the color of sand looks lighter (compare Figure 10 with Figure 11)

Figure 10 Sand samples before dry out

Figure 11 Sand samples after dry out

Weigh the sand and record as mass of dry aggregate then keep drying the sample until the mass of dry aggregate doesn't change

Moisture content can be calculated by the following equation:

$$\text{moisture content} = \frac{\text{mass of wet aggregate} - \text{mass of dry aggregate}}{\text{mass of wet aggregate}} \times 100\%$$

(Equation 3.3)

3.2.3 Internal vibration

Internal vibration was introduced to improve the workability of mix to achieve a better consolidation. Higher workability will provide consistent rock samples compared with the poor consolidation. The internal vibration made a significant improvement in workability when the mix can not be consolidated without vibration. Especially when the high strength concrete was made, both internal vibrator (Figure 12) and vibration table (Figure 13) were used to improve the workability of mix [19]. The significant improvement on flowability of mix was shown in Figure 14 and Figure 15.

Figure 12 Internal vibrator

Figure 13 Vibration table

Figure 14 Flowability of mix before using internal vibration

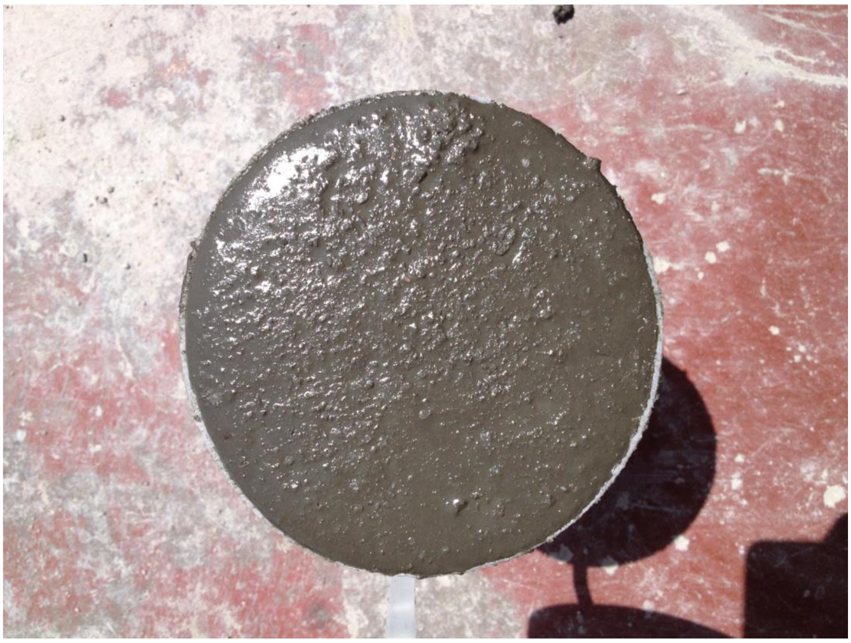


Figure 15 Flowability of mix after using internal vibration

3.2.4 Core specimen preparations

All eight parameters tests were conducted on core specimens obtained from big concrete cylinders. Preparing the concrete cores was followed by ASTM standard D4543-08 (Preparing Rock Core as Cylindrical Test Specimens and Verifying Conformance to Dimensional and Shape Tolerances) [20].

All three strength concrete samples are casted with 6 inches by 12 inches cylinders. Core specimens are cored from them using drilling rig connected with a coring bit, which are shown in Figure 16.

Figure 16 Coring process using drilling setup

Two batches of core specimens were prepared and the amounts of core specimens are shown in Table 5.

Table 5 Detail of specimens prepared

	Low Strength	Medium Strength	High Strength
Batch No. 1	24 specimens	24 specimens	24 specimens
Batch No. 2	24 specimens	24 specimens	24 specimens

All the core specimens were marked to be traced because the dimension of core specimens can be slightly varied. Batch No. 1 and Batch No. 2 are shown in Figure 17 and Figure 18, respectively.

Figure 17 Core specimens of batch No. 1

Figure 18 Core specimens of batch No. 2

After the process as coring, cutting and grinding to meet the requirements of ASTM standards, the core specimens have to be water saturated prior to conduct any tests to eliminate any differences might cause.

Standard Procedures of Water Saturation:

1) Put well prepared core specimens into beakers which are full with water and place beakers on the saturation container as shown in Figure 19. Make sure water is over specimen because vacuum process may rise up temperature leading to water consuming.

Figure 19 Saturation process set-up

2) Put the cover on and turn on the vacuum (Figure 20) until no more air bubble come out.

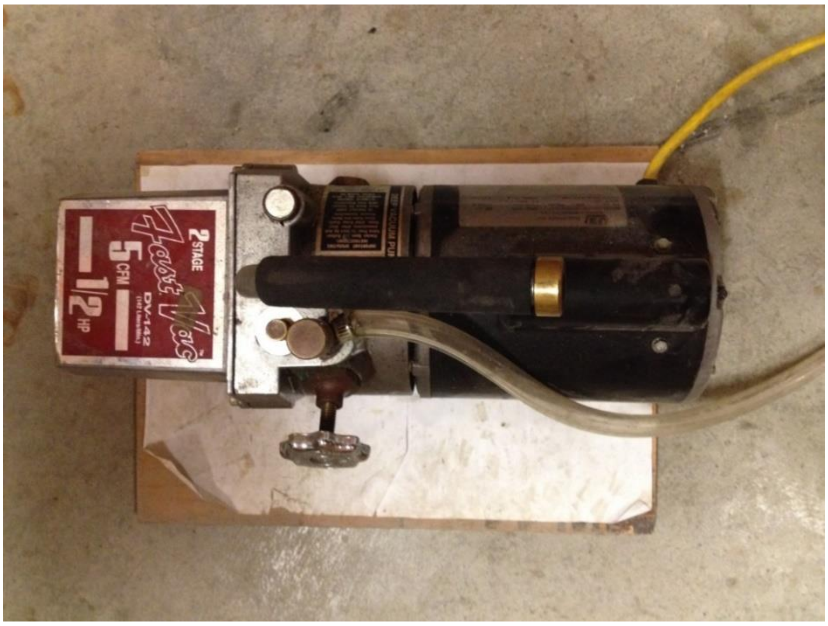


Figure 20 Vacuum for saturation process

3) Bring the specimens back to the curing tanks and make sure this transportation

process happens under water.

3.2.5 Repeatability and reproducibility

Repeatability and reproducibility are important parameters in quality assurance investigations of this research because large amount of tests and massive data were involved. Repeatability conditions are considered as experiments were conducted by the same operator at the same location using the same equipment with the same procedures over a short time; on the other hand, reproducibility conditions are usually referring to the same experiments were conducted by different operator at different location using different equipment followed by same principle of measurement procedures [21]. Table 6 was obtained from ASTM standard E177-14 (Standard Practice for Use of the Terms Precision and Bias in ASTM Test Methods) [22] which gives a better idea of the differences between repeatability and reproducibility.

Table 6 Differences between repeatability condition and reproducibility condition

Variables	Repeatability	Reproducibility
Laboratory	Same	Different
Operator	Same	Different
Apparatus	Same	Different
Time between tests	Short	Not specified

Repeatability analysis is more important than reproducibility analysis in this research because reproducibility analysis requires a lot more batches of test data which can not be easily obtained with the given manpower and time. However, future work will provide more data needed to carry out a reproducibility analysis. Regarding the differences between repeatability and reproducibility in Table 6, repeatability analysis in each batch can be evidence of quality assurance studies.

A lot of ASTM standards contain repeatability and reproducibility analysis using different equations and methods because of different test objectives [23]. In this research, standard deviation calculation and standard error calculation can provide consistency of measurements in eight parameters tests. Both Non-destructive tests (density and wave velocities) and Destructive tests (UCS and CCS) will be conducted with repeatability analysis.

Standard deviation [24] is a value to show how spread out a set of data values is. Lower standard deviation values indicate all the data are close to the mean value of the set of data, while high standard deviation values indicate the data are spread out. The standard deviation can be calculated by Equation 3.4 shown as following:

$$\sigma = \sqrt{\frac{1}{n}\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

Equation 3.4 Standard deviation calculation

Where σ is standard deviation,

n is number of samples,

x_i is value of sample number i,

$\bar{x}$ is mean value of all samples.

Standard error [25] usually refers to calculating the standard deviation of the sampling distribution of the mean, which is standard error of mean. In statistics, the mean value of the whole population is normally estimated by mean value of samples. The standard error of mean will provide how precisely the true mean of the whole population. The standard error can be calculated by Equation 3.5 shown as following:

$$\mathrm{SE} = \frac{\sigma}{\sqrt{\mathrm{n}}}$$

Equation 3.5 Standard error calculation

Where SE is standard error,

σ is standard deviation,

n is number of samples.

Standard error, mean value and quantiles of the normal distribution can be used to calculate approximate confidence intervals for the mean when data was assumed to be normally distributed. The confidence interval shows a range of estimated values and expressed by lower and upper confidence limits. Equation 3.6 can be used to calculate the upper and lower 95% confidence limits.

$$\text{Upper 95\% limit} = \bar{\mathrm{x}} + (\mathrm{SE} \times 1.96)$$

$$\text{Lower 95\% limit} = \bar{\mathrm{x}} - (\mathrm{SE} \times 1.96)$$

Equation 3.6 Upper and lower 95% confidence limit calculation

Where $\bar{\mathrm{x}}$ is equal to the sample mean,

SE is equal to the standard error for the sample mean,

1.96 is the corresponding value of 0.975 quantile of the normal distribution.

4. Three Strengths of Concrete Designs

We were given access to Pennecon's lab and Memorial University of Newfoundland's lab with material and apparatus which are related to concrete casting and strength tests. In order to obtain a strength gain curve of concrete samples, the Unconfined Compressive Strength (UCS) test was conducted at four different test ages which are 7, 14, 21 and 28 days. The following describes the apparatus used and procedures followed for each mix.

4.1 Low Strength Concrete Design

As described previously, water/cement ratio is the key factor to determine the compressive strength of concrete. In low strength concrete design, water/cement ratio should be increased to obtain a lower strength. All the tools and equipment used in all three strengths mixes are same so that the detailed information about apparatus are only shown in first design (Low strength concrete design).

4.1.1 Apparatus

Cylinder Molds: 4 by 8 inches cylindrical plastic mold for casting specimens vertically conforming to the requirements of ASTM standard C470/C470M.

Tamping Rods: 3⁄8 in. [10 mm] in diameter and approximately 12 in. [300 mm] long.

Internal Vibrator: "WYCO" internal vibrator (Figure 12), internal vibrator conforming to the requirements of ASTM standard C192/C192M-15.

Vibration Table: Designed and made by our own group (Figure 13).

Graduated Cylinder: Maximum scale is 2000 ml and minimum scale is 20 ml.

Hot Plate: Diameter of the element is 20 cm and max temperature is 400℉.

Small Tools: Tools and items such as shovels, pails, trowels, scoop, pan and rubber gloves were provided in Pennecon Precast Limited's lab.

Rotating Mixer: Electronic mixer with capacity of 1 cubic feet and Workman II 350 mixer (Figure 21).

UCS Tester: calibrated by CSA every year

Scales: Scales for determining the mass of batches of materials and concrete shall be accurate within 0.3 % of the test load at any point within the range of use.

Figure 21 Workman II 350 mixer used in MUN's lab

4.1.2 Procedure

Much of the following was according to the ASTM Standard C192/C192M-15 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory [26].

1) Storage: The cementious materials were stored in a dry place at room temperature (20 to 30℃).
2) Design: The design of low strength concrete is A: C: W= 6: 1: 1, and the designed amounts of materials in the batch are shown in Table 7.

Table 7 Designed quantities and used quantities of materials of low strength concrete design

Materials	Designed Quantities	Used Quantities
A: C: W	6: 1: 1	6: 1: 1
Aggregate	30 kg	31.397 kg
Cement	5 kg	5 kg
Water	5 kg = 5000ml	3.603 kg = 3603 ml
Water Reducer	N/A	N/A
Superplasticizer	N/A	N/A
Silica Fume	N/A	N/A

3) As the sand used will usually contain some moisture, the amount of water already in the sand should be taken into account, by determining the moisture content as in following example.

   Moisture Content Calculation: Two scoops of sand sample was weighed and found to weigh 1775.7 g and then placed on the hot plate which temperature was set at 400 ℉. After 20 minutes, the water was evaporated until mass of sand sample didn't change any more which the mass of sand sample was 1696.7 g. Moisture content can be calculated as following:

$$\text{moisture content} = \frac{\text{mass of wet aggregate} - \text{mass of dry aggregate}}{\text{mass of wet aggregate}} \times 100\%$$
$$= \frac{1775.7\text{g} - 1696.7\text{g}}{1775.7\text{g}} \times 100\% = 4.449\%$$

Therefore, total sand needed $= \frac{30\text{kg}}{1-4.449\%} = 31.397\text{kg}$

Water content in sand $= 31.397\ \text{kg} - 30\ \text{kg} = 1.397\ \text{kg} = 1397\ \text{ml}$

Therefore, water needed $= 5\ \text{kg} - 1.397\ \text{kg} = 3.603\ \text{kg} = 3603\ \text{ml}$

4) Machine Mixing: 5 kg cement and 31.397 kg sand were put together into the mixer and start rotation of mixer. After 60 seconds of rotating, 3603 ml water was added into the rotating mixer. Mixing continued for 3 minutes followed by a 3 minutes rest, followed by a 2 minutes final mixing. During the rest period, the mixer was covered up to prevent evaporation. The slump numbers of the batch for all three strengths were determined.

5) Making Specimens: Concrete was filled into each plastic mold using a scoop in 3 equal layers, vibrating each layer before filling next layer. When placing the final layer, overfilling by more than 1⁄4 in. [6 mm] was avoided. When the finish was applied after vibration, add only enough concrete with a trowel to overfill the mold about 1⁄8 in. [3 mm], work it into the surface and then strike it off. Mold caps were placed on the mold to prevent water evaporation and kept on when the specimens were stored at 23±2℃ while setting. In this step, internal vibration

was introduced to improve the workability of mix to achieve a better consolidation. Filling the mold and vibrating by internal vibrator was done in 3 equal layers. Each layer was placed in the mold before starting vibration of that layer.

6) Curing: Curing was performed according to ASTM standard C192/C192M-15, which the curing temperature is 23±2℃. After 24 hours the specimens were removed from the mold and specimens were kept under water until tested. For each batch two samples were tested for UCS at 7, 14, 21, 28 days from the day of mixing.

4.1.3 Results

Table 8 gives the slump value and UCS values (mean of two tests) of the low strength concrete.

Table 8 Results of UCS values of low strength concrete

Design	A: C: W	Slump Value (mm)	UCS Value (MPa)			
			7 days	14 days	21 days	28 days
Low Strength	6: 1: 1	221	16.95	19.55	20.12	21.59

4.1.4 Conclusions

1) In this low strength concrete design, mix had a good workability and can be applied in large quantity of concrete mixing with slump value of 221 mm.
2) The UCS value after 28 days was 21.59 MPa, which match with the desired low

strength.

4.2 Medium Strength Concrete Design

Compared with low strength concrete, medium strength concrete require higher UCS value by lowering the water content and increasing the percentage of cement in solid content. However, the dosage of additives and internal vibration are critical because of low workability caused by low water/cement ratio.

4.2.1 Procedure

Much of the following was according to the ASTM Standard C192/C192M-15 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.

1) Storage: The cementious materials were stored in a dry place at room temperature (20 to 30℃).
2) Design: The design of high strength concrete is A: C: W= 3: 1: 0.45 the designed amounts of materials in the batch are shown in Table 9.

Table 9 Designed quantities and used quantities of materials of medium strength concrete design

Materials	Designed Quantities	Used Quantities
A: C: W	3: 1: 0.45	3: 1: 0.45
Aggregate	30 kg	31.436 kg
Cement	10 kg	10 kg
Water	4.5 kg = 4500ml	3.064 kg = 3064 ml
Water Reducer	N/A	N/A
Superplasticizer	Daracem 19= 60 ml	Daracem 19= 60 ml
Silica Fume	N/A	N/A

3) Moisture Content Calculation: 1764.1 g sand sample was reduced to 1683.5 g after dry out process. Moisture content can be calculated as following:

$$\text{moisture content} = \frac{\text{mass of wet aggregat} - \text{mass of dry aggregate}}{\text{mass of wet aggregate}} \times 100\%$$

$$= \frac{1764.1\ \text{g} - 1683.5\ \text{g}}{1764.1\ \text{g}} \times 100\% = 4.568\%$$

Therefore, total sand needed $= \frac{30\ \text{kg}}{1-4.568\%} = 31.436\ \text{kg}$

Water content in sand $= 31.436\ \text{kg} - 30\ \text{kg} = 1.436\ \text{kg} = 1436\ \text{ml}$

Therefore, water needed $= 4.5\ \text{kg} - 1.436\ \text{kg} = 3.064\ \text{kg} = 3064\ \text{ml}$

4) Machine Mixing: 10 kg cement and 31.436 kg sand were put together into the mixer and start rotation of mixer. After 60 seconds of rotating, 3064 ml water was added into the rotating mixer. Mixing continued for 3 minutes followed by a 3 minutes rest, followed by a 2 minutes mixing. During the rest period, the mixer was covered up to prevent evaporation. Because of low water/cement ratio, the

slump value of 77 mm resulted in a low workability. After the 5 mins mixing, superplasticizer was added in individually. In this case, 60 ml of Daracem 19 was added into the mixer. Keep rotating for 5 more mins and a slump value of 198 mm was obtained.

5) Making Specimens: Concrete was filled into plastic mold using a scoop in 3 equal layers, vibrating each layer before filling next lay. When placing the final layer, overfilling by more than 1⁄4 in. [6 mm] was avoided. When the finish is applied after vibration, add only enough concrete with a trowel to overfill the mold about 1⁄8 in. [3 mm], work it into the surface and then strike it off. Mold caps were placed on the mold to prevent water evaporation and kept on when the specimens were stored at 23±2℃ while setting. In this step, internal vibration was introduced to improve the workability of mixture to achieve a better consolidation. Filling the mold and vibrating by internal vibrator was done in 3 equal layers. Each layer was placed in the mold before starting vibration of that layer.

6) Curing: Curing was performed according to ASTM C192/C192M-15, which the curing temperature is 23±2℃. After 24 hours the specimens were removed from the mold and specimens were kept under water until tested. For each batch two samples were tested for UCS at 7, 14, 21, 28 days from the day of mixing.

4.2.2 Results

Table 10 gives the slump value and UCS values (mean of two tests) of the medium strength concrete.

Table 10 Results of UCS values of medium strength concrete

Design	A: C: W	Slump Value (mm)	UCS Value (MPa)			
			7 days	14 days	21 days	28 days
Medium Strength	3: 1: 0.45	198	41.00	43.31	44.51	48.90

4.2.3 Conclusions

1) In medium strength concrete design, mix had a poor workability without add in superplasticizer. The slump value of mix is only 77 mm without any water reducer or superplasticizer; however, the slump value of 198 mm was obtained by adding superplasticizer.
2) The UCS value after 28 days was 48.90 MPa, which match with the medium strength expected.

4.3 High Strength Concrete Design

High strength concrete requires low water/cement ratio and cementitious materials to fill the small space between the larger particles. Water/cement ratio is usually lower than 0.35, which brings the workability issue. In this design, a large quantity of superplasticizer was required to improve the workability by decreasing flocculation as silica fume was added in as an additional cementitious material. The highly reactive fine particles of silica fume are considered to improve the strength of concrete with low water/cement ratio by improving the bonding of cement to aggregate and reducing porosity at the interface. However, longer mixing time and extra vibration processes were required to improve the workability of high strength mix caused by low water/cement ratio and silica fume.

4.3.1 Procedure

Much of the following was according to the ASTM Standard C192/C192M-15 Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.

1) Storage: The cementious materials were stored in a dry place at room temperature (20 to 30℃).
2) Design: The design of high strength concrete is A: C: W= 3: 1: 0.3 with silica fume is 15% of total cementious material. The designed amounts of materials in the batch are shown in Table 11. The designed quantities assume completely dry sand and the cement includes 15% silica fume. The actual weights of sand used include the moisture in it as calculated below, and the weight of cement and silica fume used listed separately.

Table 11 Designed quantities and used quantities of materials of high strength concrete design

Materials	Designed Quantities	Used Quantities
A: C: W	3: 1: 0.3	3: 1: 0.3
Aggregate	30 kg	31.122 kg
Cement	10 kg	8.5 kg
Water	3 kg = 3000ml	1.878 kg = 1878 ml
Water Reducer	WRDA 82, 32.6 ml	WRDA 82, 32.6 ml
Superplasticizer	Daracem 19, 390 ml	Daracem 19, 390 ml
Silica Fume	15% of cementious materials	1.5 kg

3) Moisture Content Calculation: 1924.5g sand sample was reduced to 1855.1 g after dry out process and moisture content can be calculated as following:

$$\text{moisture content} = \frac{\text{mass of wet aggregate} - \text{mass of dry aggregate}}{\text{mass of wet aggregate}} \times 100\%$$
$$= \frac{1924.5\text{g} - 1855.1\text{g}}{1924.5\text{g}} \times 100\% = 3.606\%$$

Therefore, total sand needed $= \frac{30\text{ kg}}{1-3.606\%} = 31.122\text{ kg}$

Water content in sand $= 31.122\text{ kg} - 30\text{ kg} = 1.122\text{ kg} = 1122\text{ ml}$

Therefore, water needed $= 3\text{ kg} - 1.122\text{ kg} = 1.878\text{ kg} = 1878\text{ ml}$

4) Machine Mixing: In this trial test, silica fume was added into the cement to the weight of 15% of total cementious material. 1.5 kg silica fume and 8.5 kg cement were mixed together prior to mixing concrete. Then 31.122 kg sand was put into mixer and rotation of mixer started. After 60 seconds of rotating, a pre-mixed water-water reducer solution was added into the rotating mixer. Mixing of the

concrete for 3 minutes was followed by a 3 minutes rest, followed by a 2 minutes mixing. During the rest period, the mixer was covered up to prevent evaporation. The slump value of this mix was lower than 50 mm due to the low water/cement ratio. This workability is too low for solid compacted concrete. In order to improve the workability of this concrete, 390 ml superplasticizer was added at this stage followed by rotation for 20 minutes. Because the water/cement ratio is low and silica fume consume more water, it takes longer time to mix.

5) Making Specimens: Concrete was filled into plastic mold using a scoop in 3 equal layers, vibrating each layer before filling next lay. When placing the final layer, overfilling by more than 1⁄4 in. [6 mm] was avoided. When the finish is applied after vibration, add only enough concrete with a trowel to overfill the mold about 1⁄8 in. [3 mm], work it into the surface and then strike it off. Mold caps were placed on the mold to prevent water evaporation and kept on when the specimens were stored at 23±2 ℃ while setting. In this step, internal vibration was introduced to improve the workability of mixture to achieve a better consolidation. Filling the mold and vibrating by internal vibrator was done in 3 equal layers. Each layer was placed in the mold before starting vibration of that layer. Because of low workability of high strength mix, vibration process is important to the concrete sample and reproducibility. Vibration table is introduced in our later experiments as supplementary consolidation procedures. Filled cylinder molds were placed on the vibration table for 1 minute right after the internal vibration was completed. In this high strength design, a set of compared pictures show the

difference between without vibration (Figure 22) and with vibration (Figure 23).

Figure 22 Flowability of mix before using internal vibration

Figure 23 Flowability of mix after using internal vibration

6) Curing: Curing was performed according to ASTM C192/C192M-15, which the

curing temperature is 23±2℃. After 24 hours the specimens were removed from the mold and specimens were kept under water until tested. For each batch two samples were tested for UCS at 7, 14, 21, 28 days from the day of mixing.

Note:

1) The recommended dosage of superplasticizer (Daracem 19) is 390-1300 ml/100 kg cement. Trial test number 6 shows that the maximum recommended dosage (130 ml) can not achieve desired workability. Therefore, triple maximum dosage (390 ml) was tested in this batch and it worked out.
2) Both water reducer and superplasticizer are liquid solution, however, the water content in both of these admixtures was not counted in total water used.

4.3.2 Results

Table 12 gives the slump value and UCS values of the high strength concrete.

Table 12 Results of UCS values of high strength concrete

Design	A: C: W	Slump Value (mm)	UCS Value (MPa)			
			7 days	14 days	21 days	28 days
High Strength	3: 1: 0.3	85	69.91	83.56	87.55	94.94

4.3.3 Conclusions

1) In high strength concrete design, the mix has an extremely low workability because of low water/cement ratio and silica fume blended in [27]. Even though a large quantity of superplasticizer was added in, the mix might not be able to be

applied in large quantity of concrete mixing due to the low workability. More efficient superplasticizer should be introduced to improve workability of the high strength concrete in future work.

2) The UCS value after 28 days is 94.94 MPa, which match with the high strength expected.

5. Eight Parameters Tests

One main objective of this research is to characterize the physical properties of concrete samples used to simulate real rocks. A quantitative methodology to describe rock materials in terms of their "drillability" was proposed by Prasad (2009) in paper "Drillability of a Rock in Terms of its Physico-Mechanical and Micro-Structural Properties". This paper provides the guidance of the eight parameters tests conducted in this research. However, the characterization of properties was targeted on the real rock, not concrete, the modification of experiments have to be done to suit the concrete samples.

As mentioned previously, a large number of core specimens were prepared for the eight parameters tests, which is a good opportunity to analyze the consistency of samples. Repeatability analyses were carried out during both Non-destructive tests (density, wave velocities) and destructive test (UCS).

5.1 Density

Density usually determined by weighing mass of samples and dividing by total volume, which can be an indicator of porosity for a rock with given mineral composition because density of mineral matrix is fixed. However, fracture and fluids in the rock can be a game changer. Therefore, parameters like dry density, bulk density and water saturated density are generally involved and discussed individually to give a better picture of rock porosity in field tests.

5.1.1 Apparatus, equipment and materials

1) The apparatus used in this test are digital scale (sensitive to 0.1 gram) and caliper which are shown in Figure 24

Figure 24 Caliper and digital scale for density test

2) Well prepared core specimen samples shown in Figure 16 in previous chapter.

5.1.2 Experimental procedures of density measurement

1) Weigh core concrete specimen on the digital scale and accurate to 0.1 gram to get the mass data.
2) Measure the length and diameter of core specimen each for three times and take the mean value.
3) Using mass data divide by the volume calculated from length and diameter data to

get the density values.

5.1.3 Repeatability study

In this test, 20 core specimens per strength in one batch were tested; therefore, 60 core specimens in total were tested for three strengths in each batch. With this large amount of specimens involved, standard deviation and standard error are two important values in statistics to provide confidence in repeatability. Regarding the repeatability study discussed in previous chapter, standard deviation, standard error and confidence interval are three values need to be calculated for density measurement.

Repeatability study of density is done using the method mentioned in previous chapter. Standard deviation, standard error and confidence interval are calculated regarding Equation 3.4, Equation 3.5 and Equation 3.6, respectively. The results are shown along with the density measurement in later.

5.1.4 Results of density measurement

Density measurement of all three strength concrete samples for batch 1 and batch 2 were completed and were shown in Table 13 and Table 14 along with standard deviation and standard error.

Table 13 Density values and repeatability of batch No. 1

Batch 1	Density	Mean	Standard Deviation	Standard Error	95%
Low Strength	ρ_L (kg/m^3)	2255.8	20.2	4.1	2255.8±8.1
Medium Strength	ρ_M (kg/m^3)	2313.1	24.2	4.9	2313.1±9.7
High Strength	ρ_H (kg/m^3)	2341.9	23.3	4.8	2341.9±9.3

Table 14 Density values and repeatability of batch No. 2

Batch 2	Density	Mean	Standard Deviation	Standard Error	95%
Low Strength	ρ_L (kg/m^3)	2222.0	24.8	5.1	2222.0±9.9
Medium Strength	ρ_M (kg/m^3)	2314.3	17.4	3.5	2314.3±7.0
High Strength	ρ_H (kg/m^3)	2364.2	12.2	2.5	2364.2±4.9

5.1.5 Conclusions of density measurement

Regarding the test results and repeatability study results shown in Table 13 and Table 14, the conclusions of density tests are shown as following:

1) Density results of all three strength concrete samples are consistent based on standard deviation calculation and standard error calculation.
2) Differences between batch No. 1 and batch No. 2 are not significant, which means these 2 batches samples are consistent. However, consistency between batches need more batches of tests to prove.

5.2 Unconfined Compressive Strength (UCS) Value

UCS value is the most important property to describe the quality of concrete in field, which is also considered to be a fundamental property of rock. UCS value represents the uniaxial loading capacity of a material. UCS value test is specified by ASTM C39/C39M-12 (Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens) [28].

5.2.1Apparatus, equipment and materials

1) Loading frame (INSTRON 5585H) is shown in Figure 25.

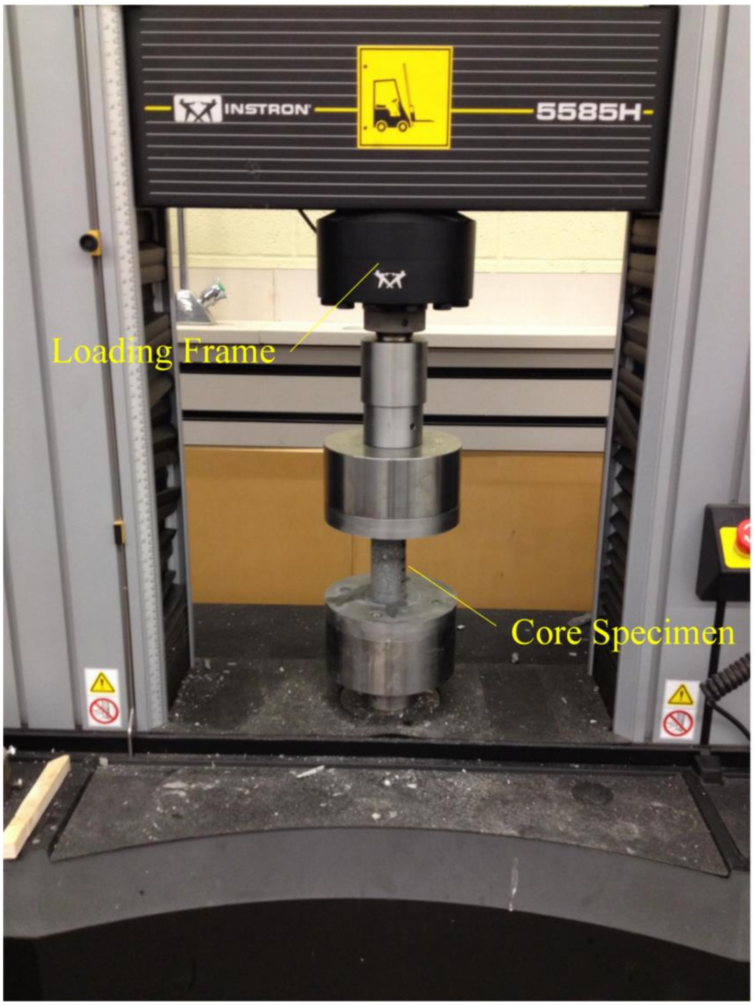


Figure 25 Loading frame and core specimen

2) Well prepared core specimen samples shown in Figure 16 and in previous chapter.

3) Data acquisition system (computer with Instron Bluehill software) shown in Figure 26

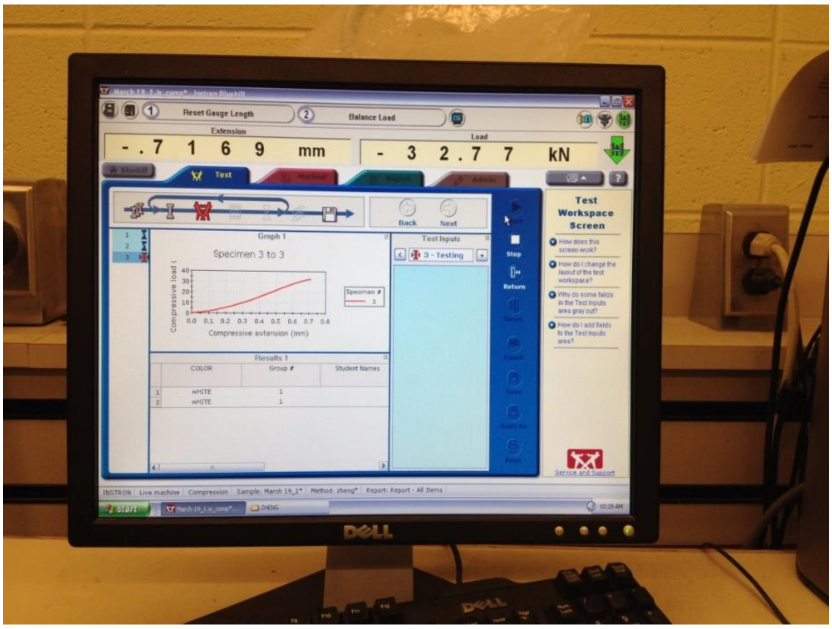


Figure 26 Computer with Instron Bluehill software can save and output data

5.2.2 Experimental procedures of UCS tests

All procedures are followed by ASTM standards C39/C39M-12 (Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens). Neither the failure angle nor the UCS value are normal for the first a few specimens. After the investigation on the equipment and procedures the problem is identified that the swivel platen (Figure 27) is faulty. Because all the specimens are well prepared followed by the ASTM standard D4543-08 (Preparing Rock Core as Cylindrical Test Specimens and Verifying Conformance to Dimensional and Shape Tolerances), swivel platen is removed from the loading system (Figure 28).

Figure 27 Swivel platen in the loading system is faulty

Figure 28 Swivel platen is removed from loading system

5.2.3 Repeatability study of UCS tests

Repeatability study of UCS values is done using the method mentioned in previous chapter. Standard deviation, standard error and confidence interval are calculated and shown along with the UCS values in later.

5.2.4 Results of UCS tests

UCS values and repeatability results for batch No.1 and batch No.2 shown in Table 15 and Table 16 respectively.

Table 15 UCS values and repeatability of batch No. 1

Batch No. 1	UCS Values (MPa) at 28 days			
	Mean	Standard Deviation	Standard Error	95%
Low Strength	19.28	0.66	0.21	19.28±0.41
Medium Strength	55.52	0.71	0.32	55.52±0.63
High Strength	80.88	3.42	1.08	80.88±2.12

Table 16 UCS values and repeatability of batch No. 2

Batch No. 2	UCS Values (MPa) at 28 days			
	Mean	Standard Deviation	Standard Error	95%
Low Strength	19.38	0.84	0.27	19.38±0.52
Medium Strength	55.89	0.65	0.20	55.89±0.40
High Strength	87.08	3.14	0.99	87.08±1.95

5.2.5 Conclusions of UCS tests

Regarding the UCS value results and repeatability analysis results shown above, the conclusions of UCS tests are:

1) UCS values shown the specimens achieved desired strength as low strength, medium strength and high strength concrete.
2) Repeatability analysis shown that UCS values are consistent for all three strengths. The differences between batch No. 2 and batch No. 1 are not significant gave us confidence about the reproducibility. However, more batches of tests need to be done in the future to prove that.
3) High strength UCS values in batch No. 2 gave a higher value compared with batch No. 1 which showed the difference of high strength concrete UCS values between batches. However, medium strength and low strength values didn't give the same results as high strength concrete showed better consistency between batches was obtained in medium strength and low strength.
4) Comparing the failure modes when UCS tests were done with swivel platen (Figure 29) and without swivel platen (Figure 30), the ones without swivel platen gave acceptable failure angles while the ones with swivel platen only gave a straight line instead. The UCS values shown in Table 17 and Table 18 indicate that the ones tested with swivel platen gave an abnormally low UCS values than designed. All these observations shown that this swivel platen is not suitable for the test and the test data using swivel platen is invalid.

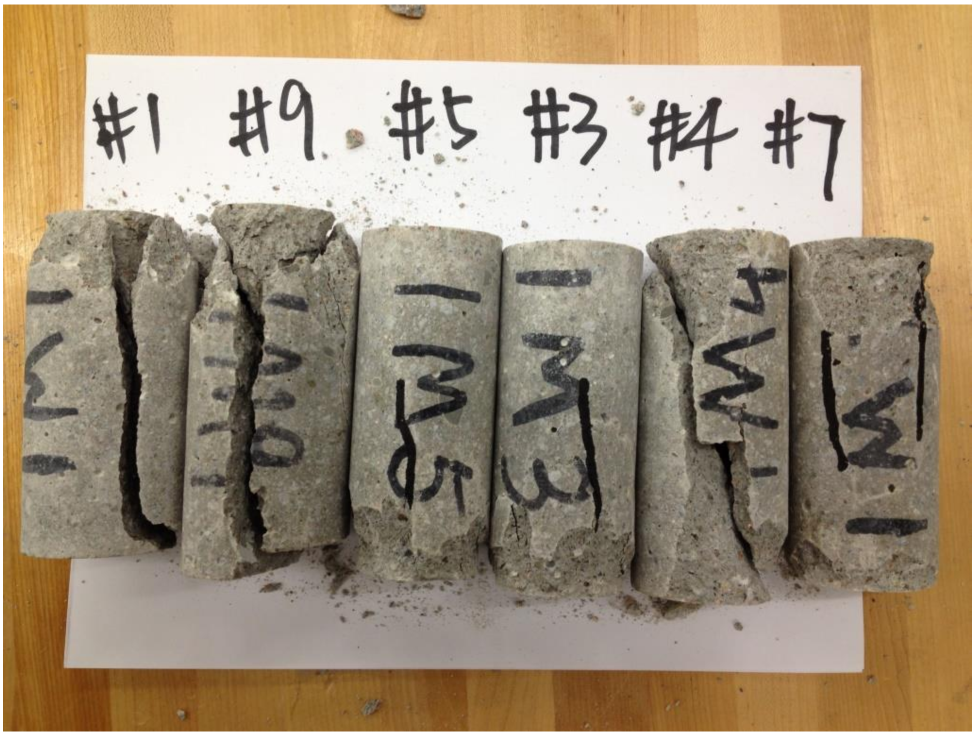


Figure 29 Failure modes with swivel platen

Figure 30 Failure modes without swivel platen

Table 17 UCS tests values with swivel platen (medium strength)

Specimen Number	UCS Values (MPa)
1M1	45.27
1M2	42.47
1M3	43.35
1M4	48.04
1M5	42.02
1M6	30.99
1M7	30.81
1M8	43.09
1M9	42.96

Table 18 UCS tests values without swivel platen (medium strength)

Specimen Number	UCS Values (MPa)
1M10	55.89
1M11	55.10
1M12	55.94
1M13	56.32
1M14	54.33

5.3 Mohr Friction Angle

Mohr Friction angle can be calculated by Mohr failure envelope for which all involved parameters can be obtained by Confined Compressive Strength (CCS) test. CCS test, also knowns as tri-axial test, is a compressive test after providing the confining pressure by confining pressure cell. The confining pressure cell is usually performed by increasing the axial and confining loads simultaneously, until a prescribed hydrostatic stress level is reached, and then the confining pressure is kept constant while the axial load is increased until failure occurs. CCS tests results will provide failure loads corresponding different confine pressure to compose a failure envelope shown in Figure 31.

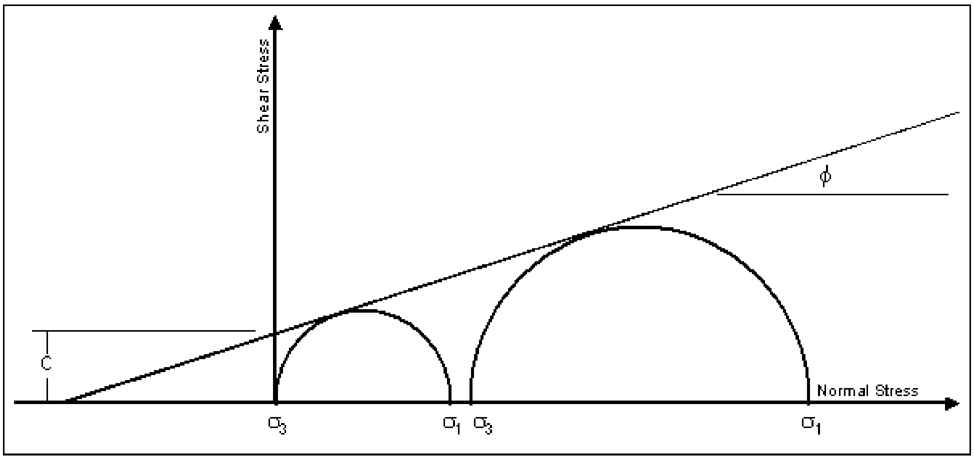


Figure 31 A graphical representation of the Mohr-Coulomb failure criteria

Where σ_1 is confine pressure,

σ_3 is failure loads under confine pressure,

The failure envelope (Mohr-Coulomb failure criteria) is tangent with circles created by σ_1 and σ_3, and it can be written as Equation 5.1

$$\tau = C + \sigma \tan\varphi \qquad \text{Equation 5.1}$$

Where:

C is cohesion stress,

σ is normal stress on the failure plane,

τ is shear stress on the failure plane,

φ is the friction angle.

5.3.1 Apparatus, equipment and materials

1) Loading frame (INSTRON 5585H) with confine pressure cell shown in Figure 32.

Figure 32 Loading frame and core specimen in confine pressure cell

2) Hydraulic pressure pump and confine pressure cell shown in Figure 33.

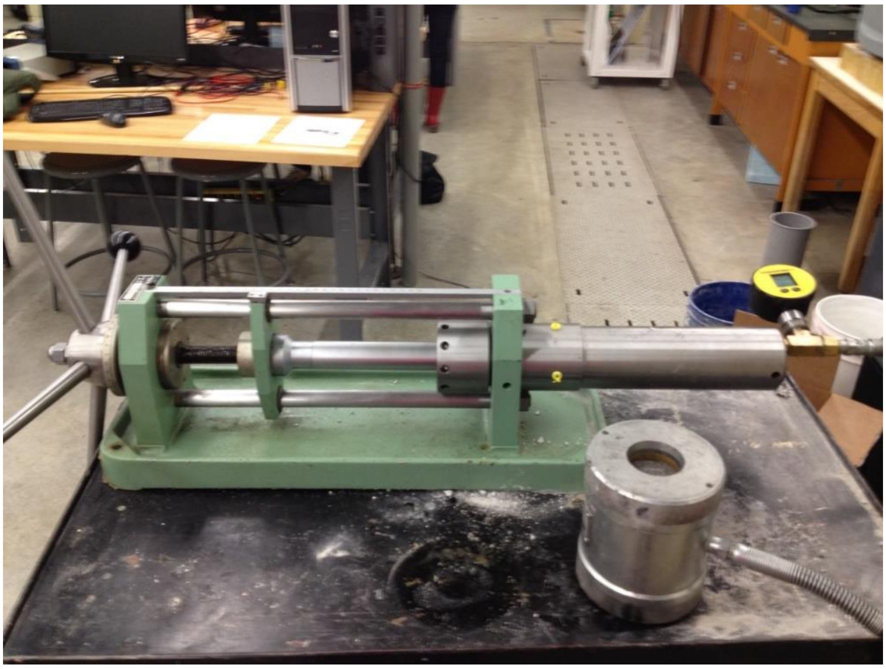


Figure 33 Pressure pump and confine pressure cell

3) Data acquisition system (computer with Instron Bluehill software) shown in Figure 26

5.3.2 Experimental procedures of CCS tests

All CCS tests are performed followed by ASTM D7012-10 (Standard Test Method for Compressive Strength and Elastic Moduli of Intact Rock Core Specimens under Varying States of Stress and Temperatures) [29].

5.3.3 Results of CCS tests

All the CCS tests results for three strengths in two batches are shown from Table 19 to Table 24 as following.

The corresponding failure envelopes are plotted from Figure 34 to Figure 39. In Figure 34 to Figure 39, each circle has two connections with X-axis which represent confining pressure and failure axial load, respectively. Three circles provided 3 different sets of confining pressures and failure axial loads. Then a failure envelope can be draw as a straight line which is tangent with all three circles. The intercept on Y-axis is cohesion stress (C) and the angle is Mohr friction angle (φ).

Low strength concrete samples of batch No. 1

Table 19 CCS tests results of low strength concrete in batch No. 1

Batch No. 1	Low Strength (MPa)
UCS (MPa)	19.28
Confine P=5MPa	41.02
Confine P=10MPa	60.76
Friction Angle	37.70°
C	4.81

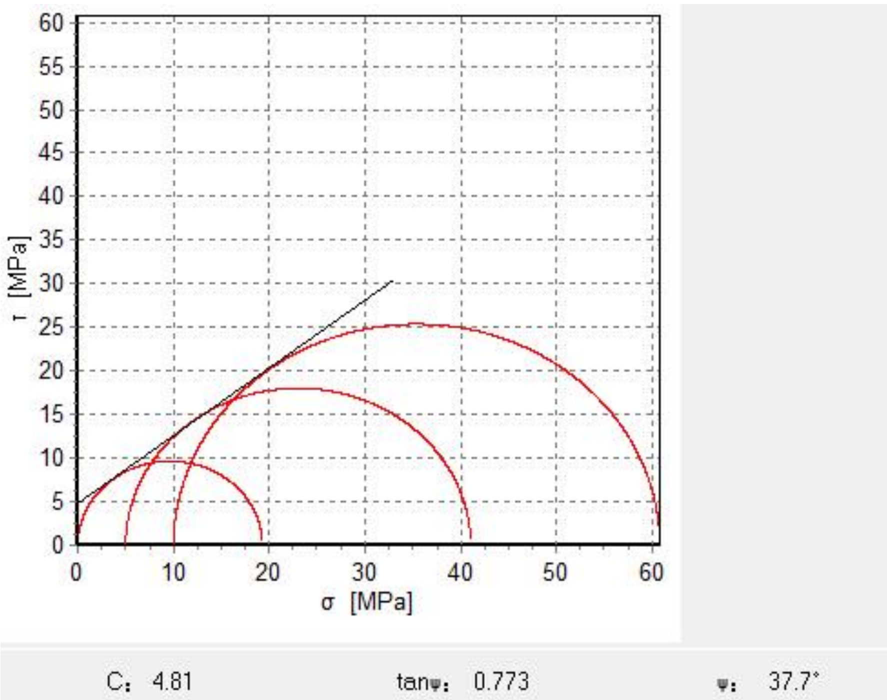


Figure 34 Failure envelope plot of low strength concrete in batch No. 1

Medium strength concrete samples of batch No. 1

Table 20 CCS tests results of medium strength concrete in batch No. 1

Batch No. 1	Medium Strength (MPa)
UCS (MPa)	55.52
Confine P=3MPa	74.77
Confine P=6MPa	85.26
Friction Angle	42.11°
C	12.57

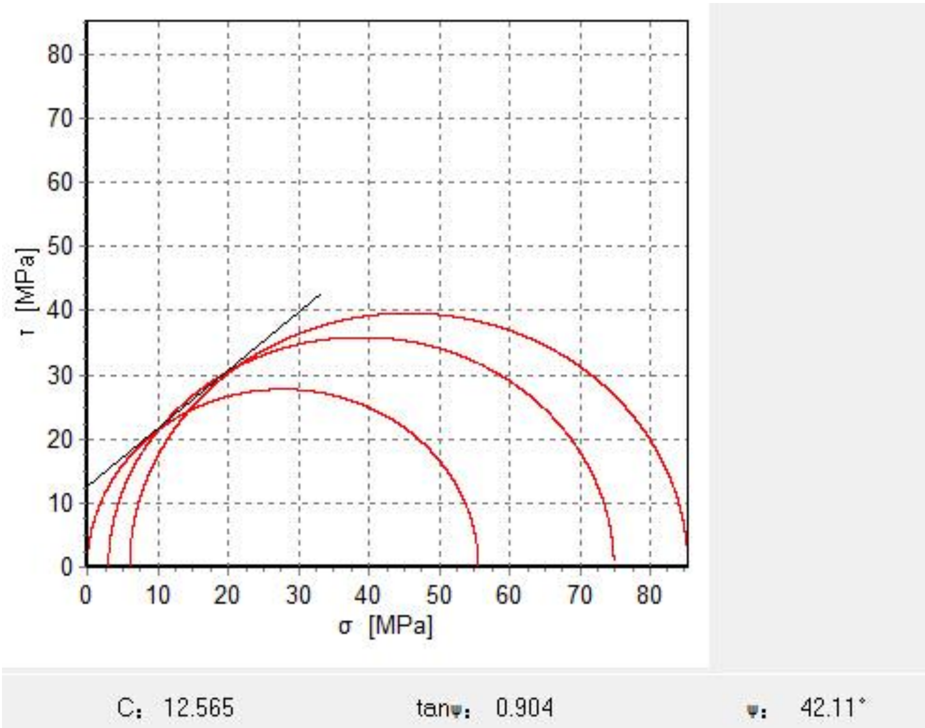


Figure 35 Failure envelope plot of medium strength concrete in batch No. 1

High strength concrete samples of batch No. 1

Table 21 CCS tests results of high strength concrete in batch No. 1

Batch No. 1	High Strength (MPa)
UCS (MPa)	80.88
Confine P=3MPa	95.29
Confine P=6MPa	111.72
Friction Angle	42.39°
C	17.75

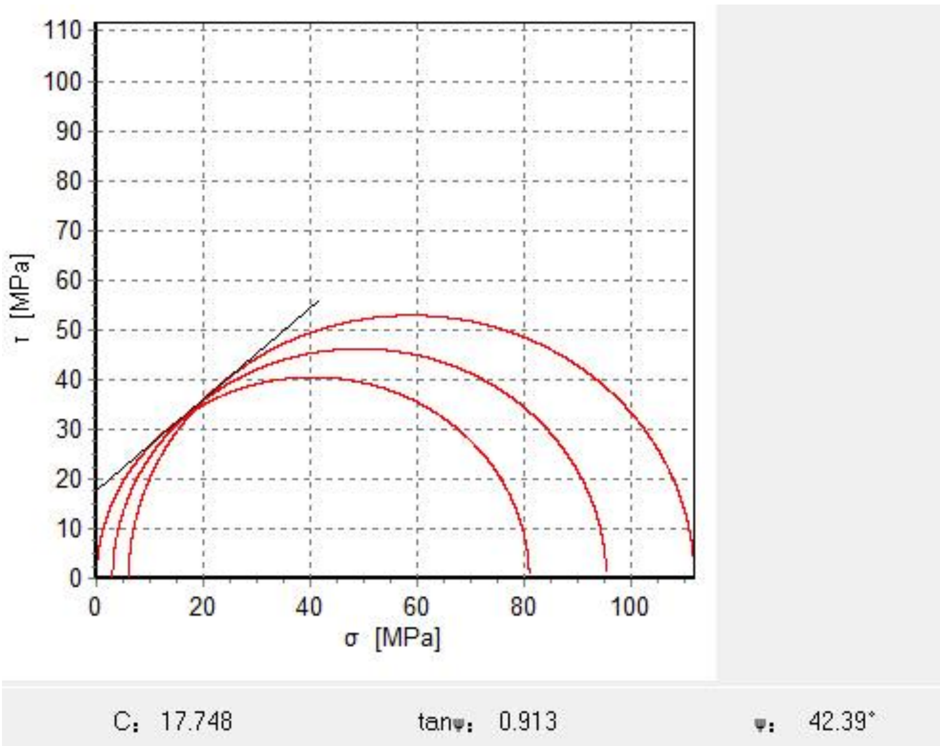


Figure 36 Failure envelope plot of high strength concrete in batch No. 1

Low strength concrete samples of batch No. 2

Table 22 CCS tests results of low strength concrete in batch No. 2

Batch No. 2	Low Strength (MPa)
UCS (MPa)	19.38
Confine P=3MPa	32.13
Confine P=6MPa	43.92
Friction Angle	37.37°
C	4.83

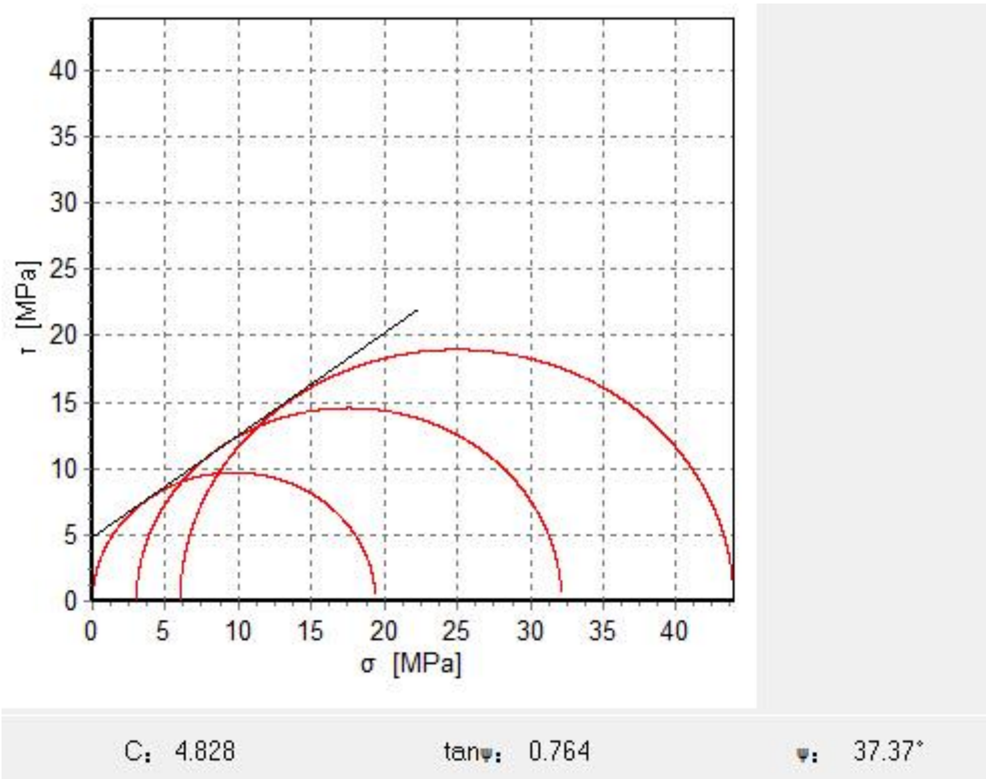


Figure 37 Failure envelope plot of low strength concrete in batch No. 2

Medium strength concrete samples of batch No. 2

Table 23 CCS tests results of medium strength concrete in batch No. 2

Batch No. 2	Medium Strength (MPa)
UCS (MPa)	55.89
Confine P=3MPa	74.51
Confine P=6MPa	83.42
Friction Angle	40.66°
C	13.09

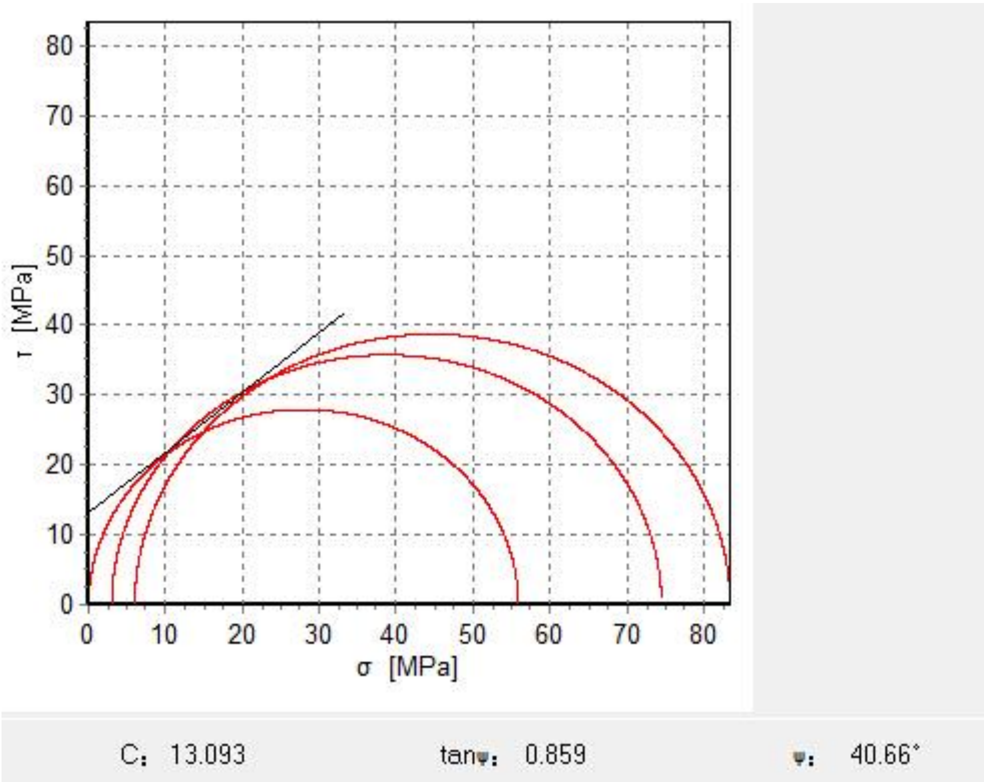


Figure 38 Failure envelope plot of medium strength concrete in batch No. 2

High strength concrete samples of batch No. 2

Table 24 CCS tests results of high strength concrete in batch No. 2

Batch No. 2	High Strength (MPa)
UCS (MPa)	87.08
Confine P=3MPa	99.25
Confine P=6MPa	115.99
Friction Angle	41.15°
C	19.57

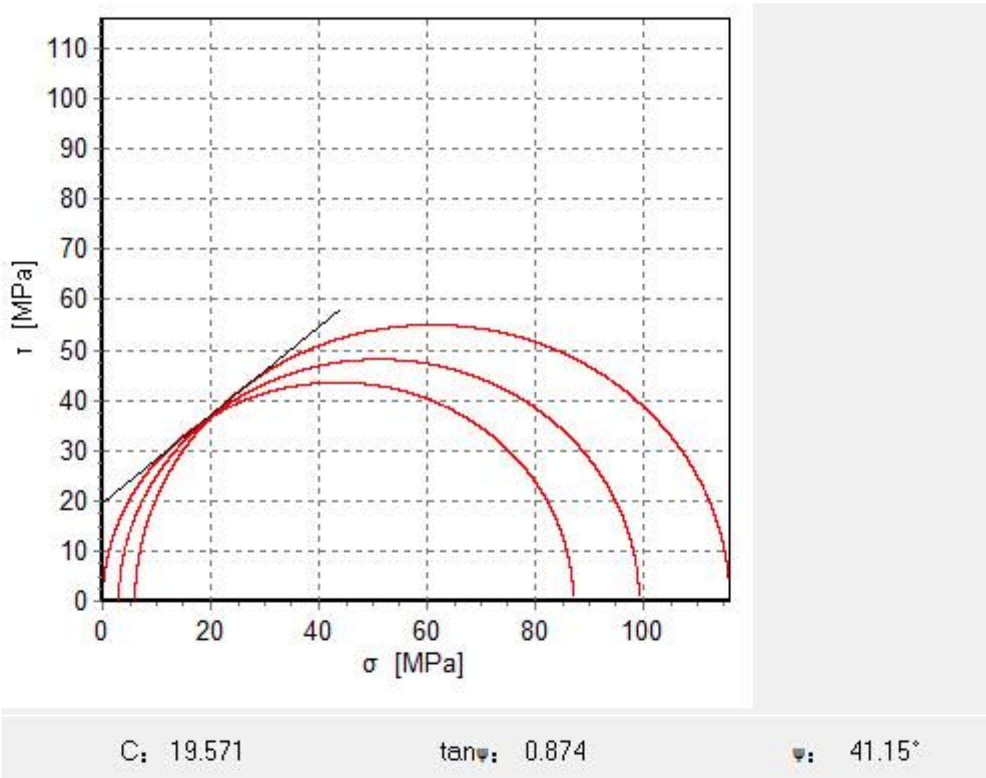


Figure 39 Failure envelope plot of high strength concrete in batch No. 2

5.3.4 Conclusions of CCS tests

1) Both UCS data and CCS data collected in each batch shown consistent results,

which provided confidence in repeatability tests.

2) Similar Mohr friction angle results obtained in medium strength concrete and high strength concrete of both 2 batches proved the consistency of the designs because the two designs were sharing same materials even though the strengths were different.
3) Lower Mohr friction angle value in low strength concrete may indicate that low strength concrete design is too weak to work with. Because all three concrete design share the same fine aggregate, Mohr friction angles of three concretes should be close to each other. Low strength concrete samples is too weak to be called as “rock” and they were all crushed to powder after CCS tests rather than an intact rock sample.
4) Usually two batches of samples aren’t sufficient to provide confidence in reproducibility. But data obtained in these batches were a good start to improve consistency. More test batches of can be carried on in future works to complete the reproducibility study.

5.4 Compressional and Shear Wave Velocity

The compressional wave (P wave), also referred to primary wave or pressure wave, is the fastest wave can travel in rocks. This feature allows it to be commonly used to characterize rock properties in lab and field for drilling purpose. Also, the P wave velocity will drastically drop in gas media can be an indicator to estimate porosity.

This measurement is performed in accordance with the procedures recommended by ASTM D2845-08 (Standard Test Method for Laboratory Determination of Pulse Velocities and Ultrasonic Elastic Constants of Rock) [30], usually on samples prepared for UCS testing.

5.4.1 Apparatus, equipment and materials

1) Olympus Pulser/Receiver 5077R shown in Figure 41 to provide excitation pulse and to receive pulse through the transducers.
2) Oscilloscope Tektronix TDS1002B shown in Figure 41 to display the data captured and output it.
3) P-Wave/S-Wave Transducers: Panametrics-NDT transducer
4) Core specimens with all three strengths
5) Shear Wave Couplant shown in Figure 41

5.4.2 Experimental procedures of wave velocities tests

1) Connect the equipment regarding the schematic shown in Figure 40, the full set up is shown in Figure 41.

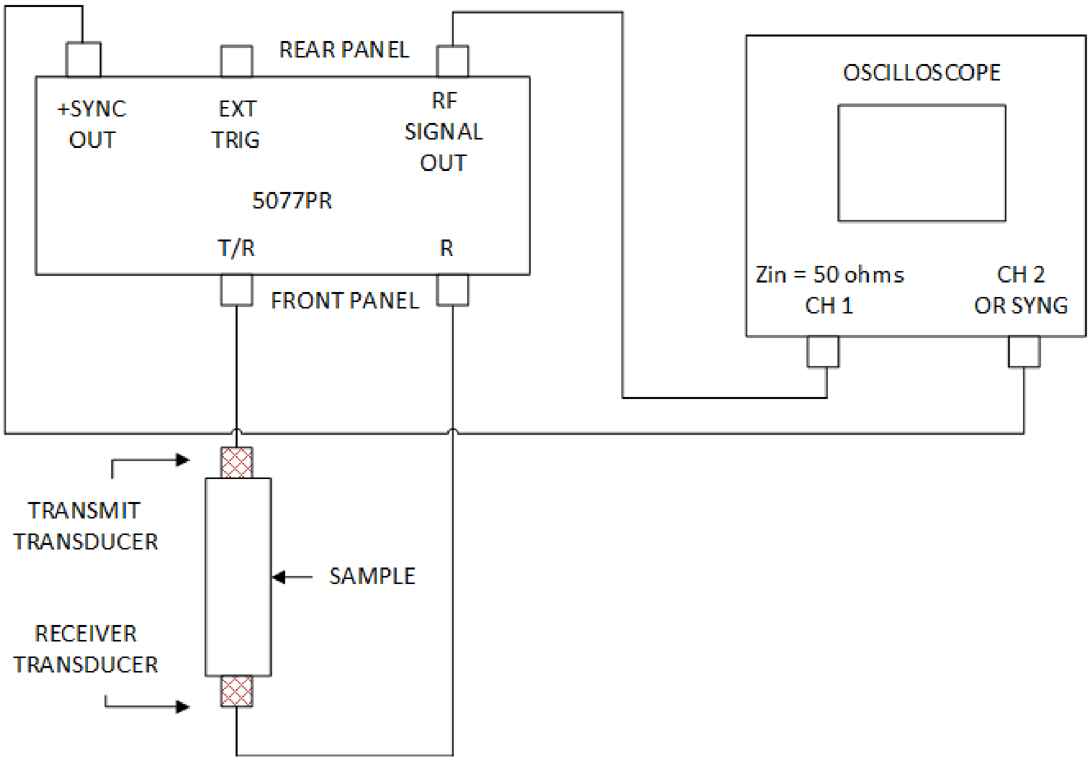


Figure 40 Schematic of the setup of wave velocity tests

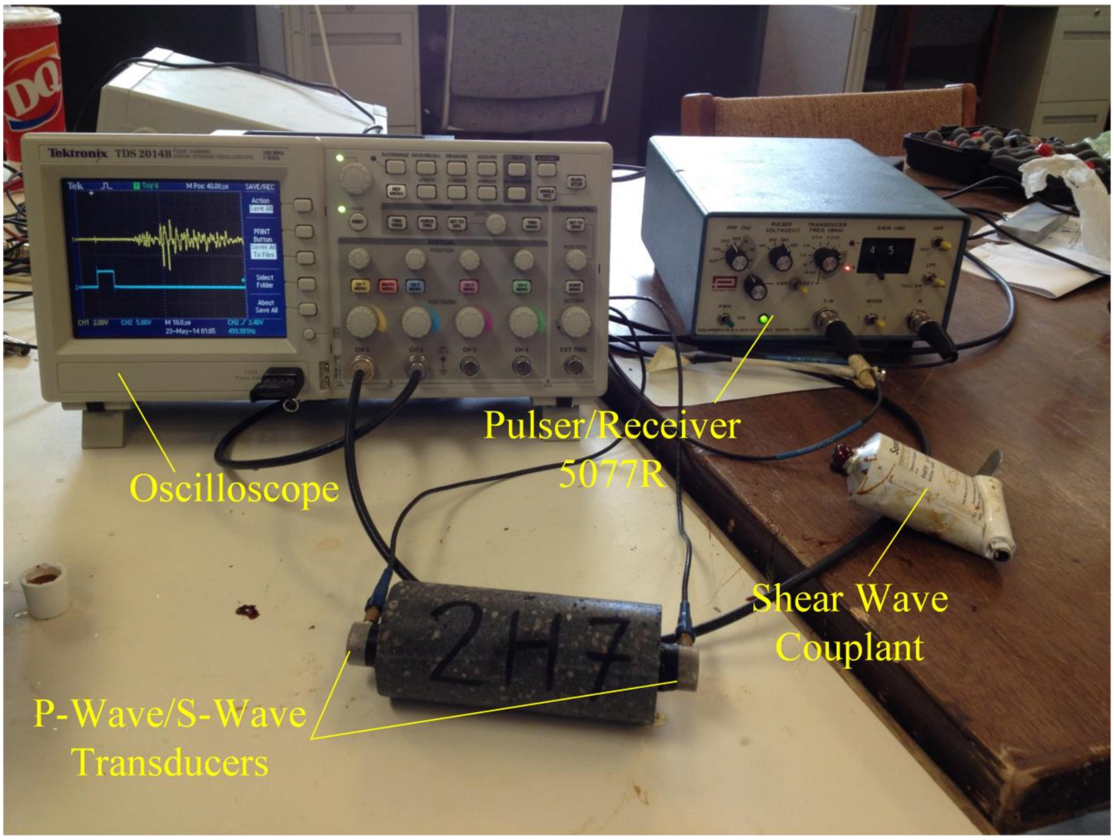


Figure 41 Experimental setup of wave velocity tests

2) Coat the couplant on the contact area of transducers, then put the two transducers together to find the zero position using oscilloscope

3) Place the two transducers at the ends of core specimen, then read the time difference which is the time of penetrating the specimen with acoustic wave, as shown in Figure 42.

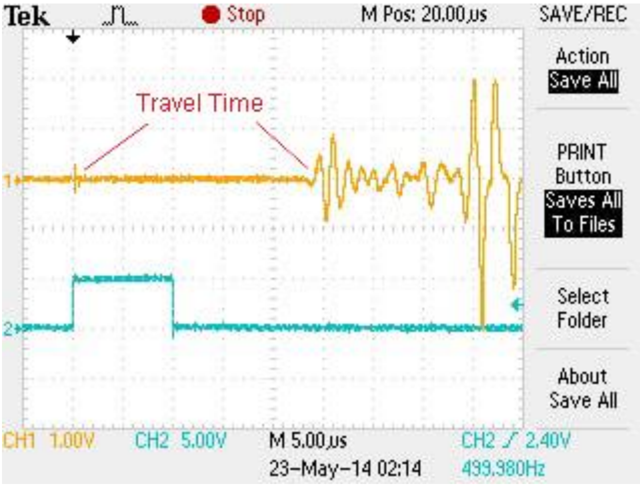


Figure 42 Example picture of wave velocity tests

4) Measure each core specimen with caliper for three times and take the mean value as travel distance, which to be divided by travel time to obtain wave velocity

5.4.3 Results of wave velocity tests

All the wave velocity tests results are shown from Table 25 to Table 28 along with repeatability studies.

P wave velocity tests results of batch No. 1

Table 25 P wave velocity tests results and repeatability study of batch No. 1

Batch No. 1	P wave	Mean	Standard Deviation	Standard Error	95% Limit
Low Strength	V_{pL} (m/s)	4304.0	70.0	14.3	4304.0±28.0
Medium Strength	V_{pM} (m/s)	4785.4	51.1	10.4	4785.4±20.5
High Strength	V_{pH} (m/s)	4710.4	60.3	12.3	4710.4±24.1

S wave velocity tests results of batch No. 1

Table 26 S wave velocity tests results and repeatability study of batch No. 1

Batch No. 1	S wave	Mean	Standard Deviation	Standard Error	95% Limit
Low Strength	V_{sL} (m/s)	2449.5	45.1	9.2	2449.5±18.1
Medium Strength	V_{sM} (m/s)	2730.8	44.1	9.0	2730.8±17.7
High Strength	V_{sH} (m/s)	2736.9	30.9	6.3	2736.9±12.3

P wave velocity tests results of batch No. 2

Table 27 P wave velocity tests results and repeatability study of batch No. 2

Batch No. 2	P wave	Mean	Standard Deviation	Standard Error	95% Limit
Low Strength	V_{pL} (m/s)	4330.5	89.3	18.2	4330.5±35.7
Medium Strength	V_{pM} (m/s)	4719.8	79.3	16.2	4719.8±31.7
High Strength	V_{pH} (m/s)	4774.6	69.5	14.5	4774.6±28.4

S wave velocity tests results of batch No. 2

Table 28 S wave velocity tests results and repeatability study of batch No. 2

Batch No. 2	S wave	Mean	Standard Deviation	Standard Error	95% Limit
Low Strength	V_{sL} (m/s)	2447.2	48.1	9.8	2447.2±19.2
Medium Strength	V_{sM} (m/s)	2795.9	29.8	6.1	2795.9±11.9
High Strength	V_{sH} (m/s)	2845.7	34.3	7.1	2845.7±14.0

5.5 Grain Size

The grain size is a parameter to give more detailed information about texture information and microstructure of rock. The shape and size of grain and block affect the abrasivity and hardness of rock sample. Specifically, in this research, grain size is focus around 2 mm, which would not be too big for the 2 inch drill bit.

5.5.1 Apparatus, equipment and materials

1) Sieves used to conduct sieve analysis test shown in Figure 43.

Figure 43 Sieves used to identify the size of fine aggregate

2) Digital scale to weigh the mass of fine aggregate.

3) Shovel, scoop, pan and other small tools.

5.5.2 Experimental procedures of grain size calculation

1) Conduct sieve analysis test on fine aggregate (Black Mountain) recording the ASTM standard C136 (Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates) [18]. The 8 sieves are mesh number 4, 8, 10, 16, 20, 40, 60 and 100 which aperture diameter corresponding to 4.75 mm, 2.36 mm, 2 mm, 1.18 mm, 0.85 mm, 0.425 mm, 0.25 mm and 0.15 mm.

2) Weigh the retained fine aggregate mass on each sieve to calculate the percentage of each size (sieve size).

3) After the grain size distribution of fine aggregate, the total grain size percentage can be calculated based on the A: C: W ratio of each design. The calculation is

shown in Equation 5.2.

$$grain\ size\ of\ concrete$$
$$= grain\ size\ of\ aggregate$$
$$\times \frac{mass\ of\ aggregate}{mass\ of\ cementious\ materials + mass\ of\ water + mass\ of\ aggregate}$$

Equation 5.2 Calculation of grain size distribution of concrete

5.5.3 Results of grain size calculation

Cementious materials will be in form of concrete matrix exist in concrete, which is considered to be not in count of grain size. All three concrete designs share the same fine aggregate so that the grain size distribution of fine aggregate is the key part of the grain size calculation of concrete.

Sieve analysis of fine aggregate has been done in previous works which is shown in Table 1 Passed percentage of fine aggregate. Regarding the results in Table 1, the percentage of fine aggregate retained on each sieve can be calculated and shown in Table 29.

Table 29 Retained percentage of fine aggregate on each sieve

Grain Size (mm)	Passed Percentage (%)	Retained Percentage (%)
	100.00	0
4.75	98.56	1.44
2.36	77.62	20.94
2.	74.16	3.46
1.18	58.71	15.45
0.85	48.44	10.27
0.425	21.68	26.76
0.25	8.71	12.97
0.15	3.81	4.9

The average grain size of fine aggregate can be calculated as Equation 5.3.

$$grain\ size\ of\ fine\ aggregate = \sum grain\ size \times retained\ percentage$$

Equation 5.3 Grain size calculation of fine aggregate

$$\begin{aligned}\sum grain\ size\ of\ fine\ aggregate\\ &= 4.75 \times 1.44\% + 2.36 \times 20.94\% + 2 \times 3.46\% + 1.18 \times 15.45\%\\ &+ 0.85 \times 10.27\% + 0.425 \times 26.76\% + 0.25 \times 12.97\% + 0.15\\ &\times 4.9\% = 1.05\ \ (mm)\end{aligned}$$

Since grain size of aggregate is calculated to be 1.05 mm, grain size of concrete can be calculated regarding Equation 5.2, and the results are shown in Table 30.

Table 30 Grain size results of all three concretes

	A: C: W	Aggregate Percentage	Grain size (mm)
Low Strength	6: 1: 1	75%	0.79
Medium Strength	3: 1: 0.45	67.42%	0.71
High Strength	3: 1: 0.3	69.77%	0.73

5.6 Mineralogy

Mineralogy is an important factor which can affect the physical properties of rock in drilling. Although concrete is not real rock, it made by rock particles where fine aggregate comes from. Mineralogy analysis is focus on the minerals in the fine aggregate. Furthermore, the basis of eight parameters tests is Prasad's paper (2009) "Drillability of a Rock in Terms of its Physico-Mechanical and Micro-Structural Properties", which consider the quartz content is the key mineral in mineralogy analysis. Therefore, the first step of mineralogy analysis is to determine the quartz content in aggregate. After the quartz content of aggregate is determined, the quartz content of concrete can be estimated by the weight percentage of composition.

The sampling of fine aggregate is important because the selected fine aggregate should properly represent all the fine particles. In this case, fine aggregate was selected by different size through sieve analysis. The aperture size were 4.75mm, 2.36mm, 1.18mm, 425µm, 300µm, 150µm and 80µm, which are based on CSA fine aggregate distribution.

5.6.1 Apparatus, equipment and materials

1) Laboratory disc mill (Siebtechnik) (Figure 44) to crush fine aggregate into finer powder so that it can be fit in XRD machine.

Figure 44 Siebtechnic disc mill

2) Rigaku Ultima IV x-ray diffractometer (XRD) to analyze the crystal structure of fine aggregate shown in Figure 45.

Figure 45 Rigaku Ultima IV x-ray diffractometer

3) Black Mountain sand (fine aggregate).

5.6.2 Experimental procedures of mineralogy analysis

1) Dry the Black Mountain sand sample out to make sure water content was vaporized
2) Separate the sand sample into 7 portions using sieves which aperture size are 4.75mm, 2.36mm, 1.18mm, 425µm, 300µm, 150µm and 80µm. Then calculate the weight percentage for each portion. (Table 32)
3) Take sand samples from each portion then crush them separately, the ideal size for XRD tests is around 100µm
4) Put sample powder into XRD machine to obtain spectrum
5) Identify the minerals and calculate the quantity using commercial software "Jade 9"

5.6.3 Results of mineralogy analysis

As mentioned previously, fine aggregate sample were sieved into 7 portions and weighed each portion so that the sampled fine aggregate can represent the real percentage of aggregate sample. The percentages results of mineralogy contents are shown in Table 31 and are plotted in Figure 46.

Table 31 Detailed information of mineralogy analysis of all portions

No.	Particle Size	Quartz	Albite	Anorthite
1	Larger than 4.75mm	59.90%	40.10%	0
2	2.36mm-4.75mm	33.90%	0	66.10%
3	1.18mm-2.36mm	42.00%	17.40%	40.60%
4	425µm-1.18mm	47.70%	14.90%	37.40%
5	300µm-425µm	56.00%	18.50%	25.50%
6	150µm-300µm	50.00%	17.80%	32.20%
7	80µm-150µm	50.30%	18.60%	31.20%

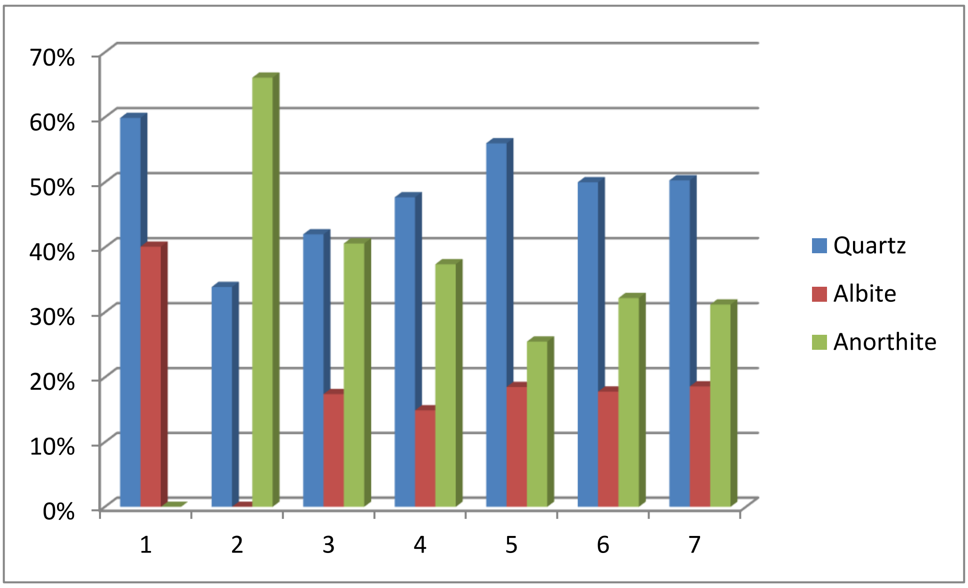


Figure 46 Mineralogy analysis results of all portions

Quartz contents of each portion and weight percentages of each portion are shown in Table 32.

Table 32 Weight percentage and quartz content of each portion

Grain Size	Weight Percentage (%)	Quartz Content (%)
4.75 mm	1.44	59.90
2.36 mm	20.94	33.90
1.18 mm	18.91	42.00
425 μm	37.03	47.70
300 μm	13.00	56.00
150 μm	4.90	50.00
80 μm	3.80	50.30

Regarding the information in Table 32, the average quartz content of fine aggregate can be calculated as Equation 5.4.

$$quartz\ content\ of\ fine\ aggregate = \sum quartz\ content\ of\ each\ portion \times weight\ \ percentage$$

Equation 5.4 Quartz content calculation of fine aggregate

$$\sum quartz\ content\ of\ fine\ aggregate = 59.90\% \times 1.44\% + 33.90\% \times 20.94\% + 42.00\% \times 18.91\% + 47.70\% \times 37.03\% + 56.00\% \times 13.00\% + 50.00\% \times 4.90\% + 50.30\% \times 3.80\% = 45.21\%$$

In this designed synthetic rock (concrete), fine aggregate is considered as main material that provides abrasivity and hardness. Quartz content of concrete can be calculated regarding Equation 5.5 since quartz content of aggregate is determined to be 45.21%.

$$quartz\ content\ of\ concrete = quartz\ content\ of\ aggregate \times \frac{mass\ of\ aggregate}{mass\ of\ cementious\ materials + mass\ of\ water + mass\ of\ aggregate}$$

Equation 5.5 Calculation of quartz content of concrete

The results of quartz content of concrete for all three strengths are shown in Table 33.

Table 33 Quartz content results of all three concretes

	A: C: W	Aggregate Percentage	Quartz content (%)
Low Strength	6: 1: 1	75%	33.91
Medium Strength	3: 1: 0.45	67.42%	30.48
High Strength	3: 1: 0.3	69.77%	31.54

5.6.4 Conclusions of mineralogy analysis

1) Quartz contents of all three concrete designs are determined based on quartz content of aggregate and weight percentage of composition.
2) Quartz content of low strength concrete is higher than medium and high strength because only quartz content of aggregate is considered and higher aggregate percentage in low concrete design.
3) Quartz content is only required mineral in spider plot, but Anorthite and Albite are feldspars with Mohs scale hardness range from 6 to 6.5 which can also been an indicator of hardness. This fine aggregate consists of quartz and feldspar which is hard mineral can provide strength in concrete.

5.7 Porosity

Porosity is a key parameter in petroleum industry because it’s important to drilling engineering, reservoir engineering and production engineering. Porosity represent the pore space or void volume in the bulk and expressed by percentage. Void space is where the stress concentrated and the failure occurred, which is critical in drilling process because high porosity leads to a decreasing in strength. Furthermore, void space not only provide the storage room for fluid and gas but also can be the flowing channel of oil and gas, which are closely related to both reservoir engineering and production engineering.

5.7.1 Apparatus, equipment and materials

1) Wild Makroskop M420 microscope and Nikon digital camera shown in Figure 47 to magnify and capture texture images

Figure 47 Microscope and digital camera

2) Software ImageJ to analyze the digital pictures and calculate the porosity of rock sample

3) Thin section samples (Figure 48) of three strength made by Core Research Equipment & Instrument Training Network (CREAIT)'s laboratory.

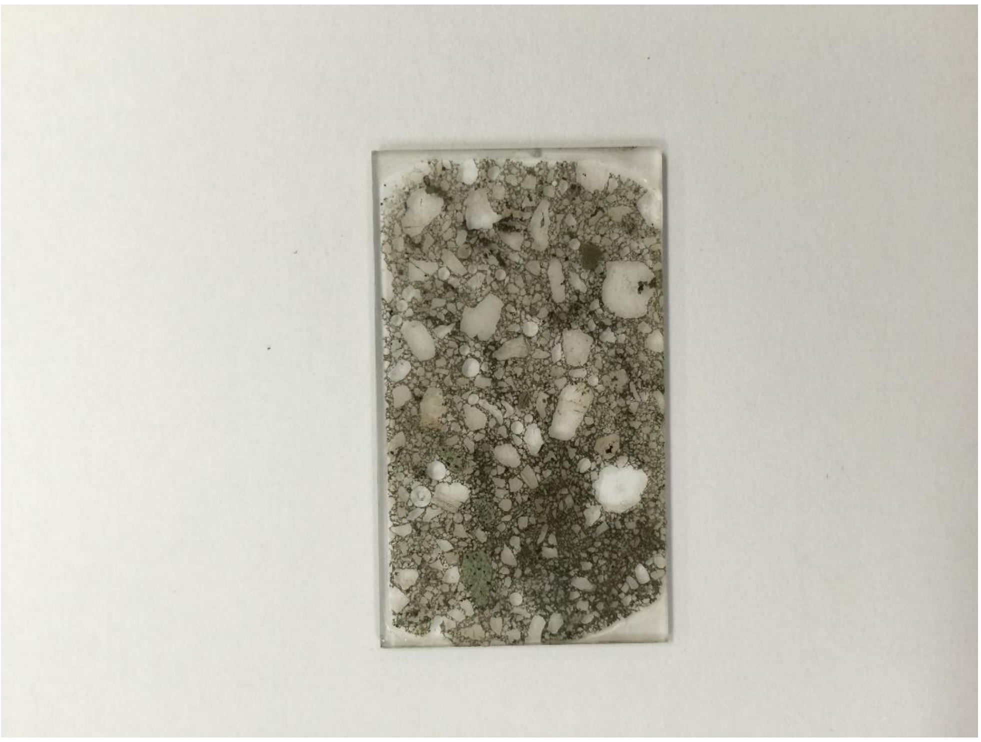


Figure 48 Thin section sample

5.7.2 Experimental procedures of porosity calculation

1) Put thin section sample under the microscope and adjust the focus knob to get the clear image
2) Hook up the digital camera with eyepiece and take a clear photo (Figure 49)

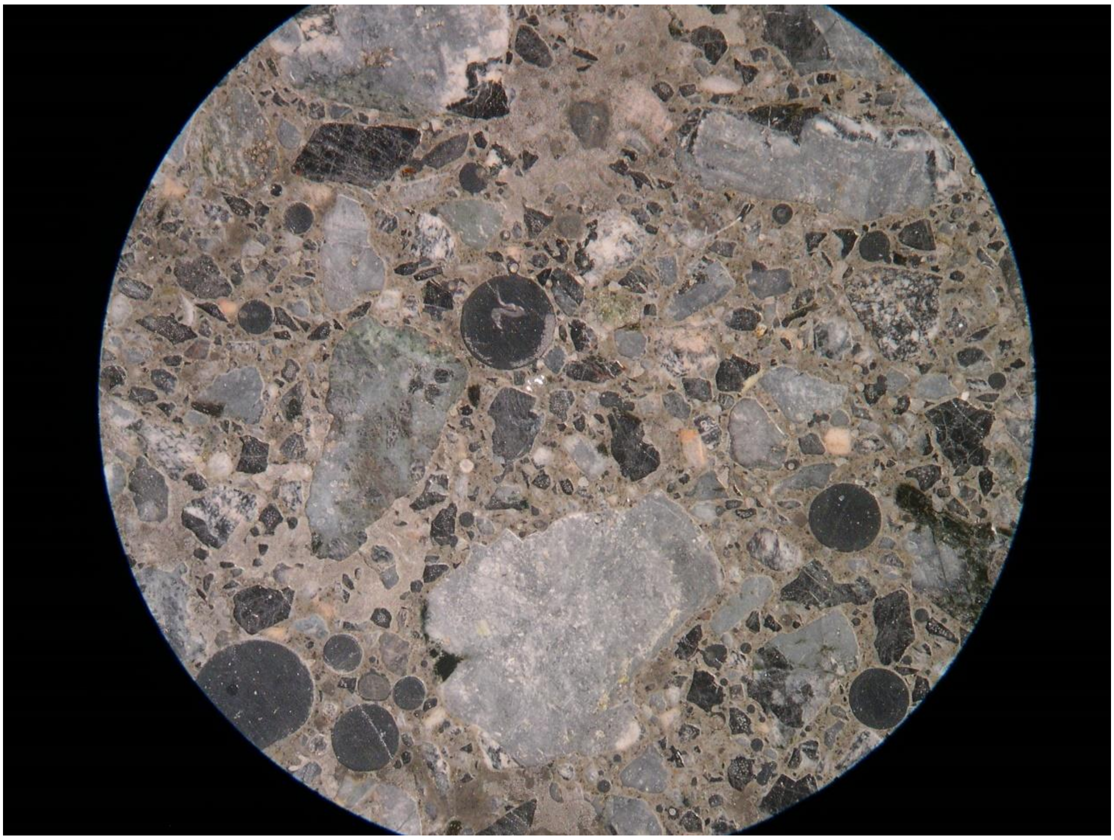


Figure 49 A clear picture of thin section slide

3) Mark every pore space in the picture and calculate the area of pore space using software ImageJ (Figure 50). Sum up all the area of pore space then divided by the total area in the shot

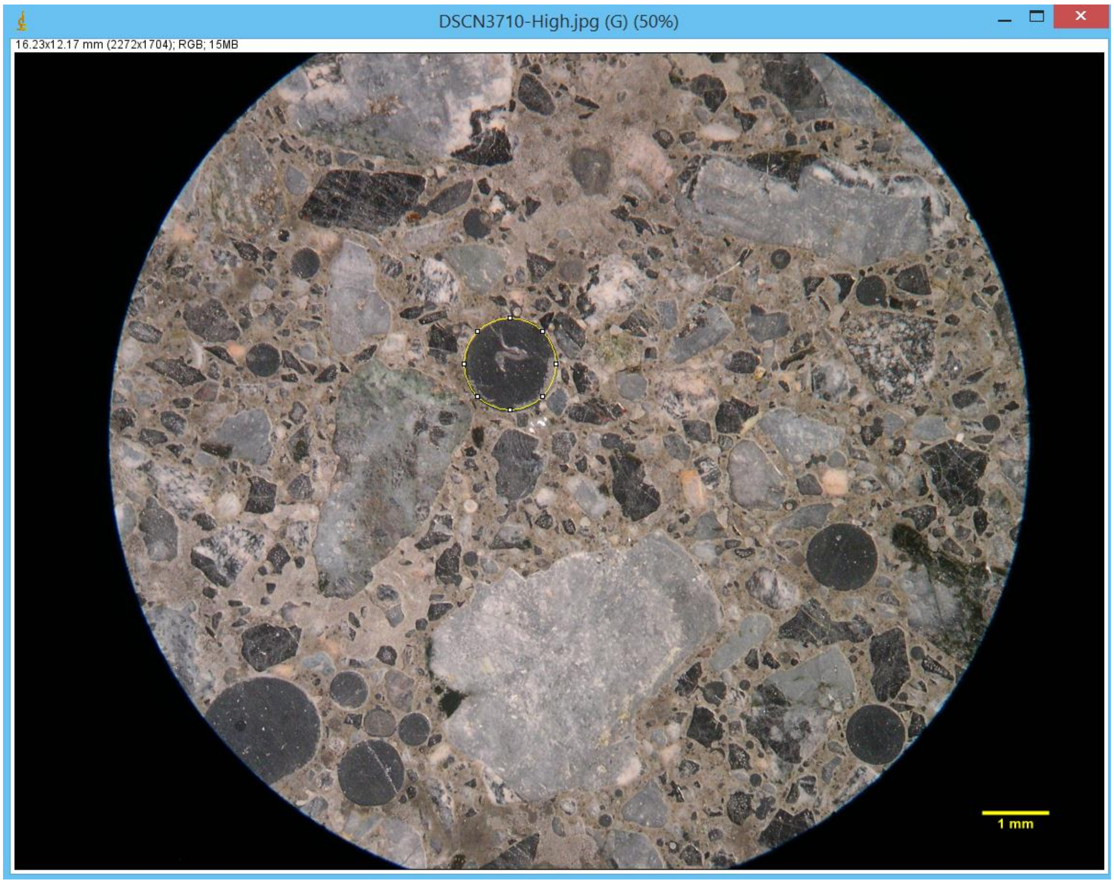


Figure 50 Porosity calculation using thin section image

5.7.3 Results of porosity calculation

Areas of void space in the thin sections are calculated through ImageJ software for all three strength concrete and the results are shown in Table 34, Table 35 and Table 36.

Table 34 Porosity calculated of low strength concrete

Voids Breakdown	Area Measured (mm^2)
1	1.118
2	0.943
3	1.132
4	0.401
5	3.764
6	0.591
7	0.627
8	5.565
Total Voids	14.141
Total Cross-section	138.06
Porosity of Low Strength	10.24%

Table 35 Porosity calculated of medium strength concrete

Voids Breakdown	Area Measured (mm^2)
1	0.266
2	0.158
3	0.101
4	1.698
5	0.42
6	0.625
7	0.444
8	0.648
9	2.534
10	0.538
11	0.158
12	0.187
13	0.462
14	0.379
15	0.281
16	0.184
19	0.283
20	0.16
21	0.154
22	0.16
23	0.124
Total Voids	9.964
Total Cross-section	137.365
Porosity of Medium Strength	7.25%

Table 36 Porosity calculated of high strength concrete

Voids Breakdown	Area Measured (mm^2)
1	0.236
2	0.19
3	1.232
4	0.13
5	0.164
6	0.193
7	1.416
8	0.595
9	0.039
10	0.26
11	0.179
12	0.169
13	0.769
14	1.755
15	0.07
16	0.147
17	0.054
18	0.755
19	0.089
20	0.597
21	0.157
Total Voids	9.196
Total Cross-section	138.842
Porosity of High Strength	6.62%

5.7.4 Conclusions of porosity calculation

1) Porosities of all three strength are 10.24% (low strength), 7.25% (medium strength) and 6.62% (high strength). Large amount of cement material in the concrete designs result in low porosity values of synthetic rocks.
2) Regarding the breakdown values in three concrete design, voids in low strength concrete are fewer and bigger while voids in medium and high strength concrete are more and smaller.

5.8 Spider plot of eight parameters tests

The large amount of data generated by the testing is most conveniently presented in terms of the eight drillability parameters. All the data will be processed and converted into number in scale of 0 to 8 to present in a more intuitive and convenient approach.

5.8.1 Results of eight parameters tests

All eight parameters tests for three concretes designs are completed and results of characterization are shown in Table 37, Table 38 and Table 39.

Table 37 Eight parameters tests results of low strength concrete

8 Parameters	Batch No. 1	Batch No. 2	Average Value
Density	2255.8 kg/m^3	2222.0 kg/m^3	2238.9 kg/m^3
UCS	19.28 MPa	19.38 MPa	19.33 MPa
Mohr Friction Angle	37.70°	37.37°	37.54°
P-wave Velocity	4304.0 m/s	4330.5 m/s	4317.3 m/s
S-wave Velocity	2449.5 m/s	2447.2 m/s	2448.4 m/s
Grain Size	0.79 mm	0.79 mm	0.79 mm
Mineralogy	33.91%	33.91%	33.91%
Porosity	10.24%	10.24%	10.24%

Table 38 Eight parameters tests results of medium strength concrete

8 Parameters	Batch No. 1	Batch No. 2	Average Value
Density	2313.1 kg/m^3	2314.3 kg/m^3	2313.7 kg/m^3
UCS	55.52 MPa	55.89 MPa	55.71 MPa
Mohr Friction Angle	42.11°	40.66°	41.39°
P-wave Velocity	4785.4 m/s	4719.8 m/s	4752.6 m/s
S-wave Velocity	2730.8 m/s	2795.9 m/s	2763.4 m/s
Grain Size	0.71 mm	0.71 mm	0.71 mm
Mineralogy	30.48%	30.48%	30.48%
Porosity	7.25%	7.25%	7.25%

Table 39 Eight parameters tests results of high strength concrete

8 Parameters	Batch No. 1	Batch No. 2	Average Value
Density	2341.9 kg/m^3	2364.2 kg/m^3	2353.1 kg/m^3
UCS	80.88 MPa	87.08 MPa	83.98 MPa
Mohr Friction Angle	42.39°	41.15°	41.77°
P-wave Velocity	4710.4 m/s	4774.6 m/s	4742.5 m/s
S-wave Velocity	2736.9 m/s	2845.7m/s	2791.3 m/s
Grain Size	0.73 mm	0.73 mm	0.73 mm
Mineralogy	31.54%	31.54%	31.54%
Porosity	6.62%	6.62%	6.62%

5.8.2 Spider plot of eight parameters tests

Spider plot of eight parameters provide the direct pictures for the characterization of synthetic rocks. Figure 51 is an example of spider plot in Prasad’s paper (2009).

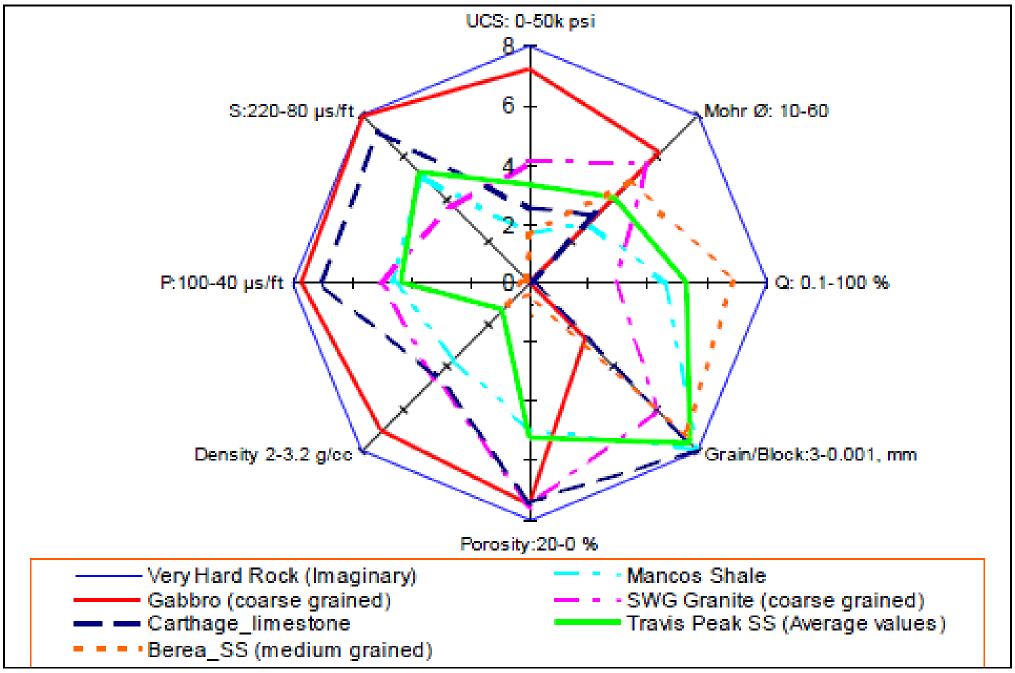


Figure 51 Spider plot as a model from Prasad's paper

All eight parameters tests results (average value of 2 batches) shown in previous tables should be converted into number in the scale 0 to 8 since Figure 51 have provided the basis of the range and scale for the parameters. The converted Results of all three strength concretes are shown in Table 40, Table 41 and Table 42.

Table 40 Converted results of low strength concrete

Low Strength	Converted Results	Number in scale of 0-8
UCS: 0-15k psi	2.80 k psi	1.49
Mohr Ø: 30-50°	37.54°	3.02
Q: 20-40%	33.91%	5.56
Grain/Block: 2-0.1mm	0.79 mm	4.72
Porosity: 15-5%	10.24%	3.81
Density: 2-2.5 g/cc	2.24 g/cc	3.84
P: 80-50 µs/ft	70.60 µs/ft	2.51
S: 160-80 µs/ft	124.49 µs/ft	3.55

Table 41 Converted results of medium strength concrete

Medium Strength	Converted Results	Number in scale of 0-8
UCS: 0-15k psi	8.08 k psi	4.31
Mohr Ø: 30-50°	41.39°	4.56
Q: 20-40%	30.48%	4.19
Grain/Block: 2-0.1mm	0.71 mm	5.05
Porosity: 15-5%	7.25%	6.20
Density: 2-2.5 g/cc	2.31 g/cc	4.96
P: 80-50 µs/ft	64.13 µs/ft	4.23
S: 160-80 µs/ft	110.30 µs/ft	4.97

Table 42 Converted results of high strength concrete

High Strength	Converted Results	Number in scale of 0-8
UCS: 0-15k psi	12.18 k psi	6.50
Mohr Ø: 30-50°	41.77°	4.71
Q: 20-40%	31.54%	4.62
Grain/Block: 2-0.1mm	0.73 mm	4.97
Porosity: 15-5%	6.62%	6.70
Density: 2-2.5 g/cc	2.35 g/cc	5.60
P: 80-50 µs/ft	64.27 µs/ft	4.19
S: 160-80 µs/ft	109.20 µs/ft	5.08

Regarding all the data processed above, the spider plot of all three strengths concrete is shown in Figure 52.

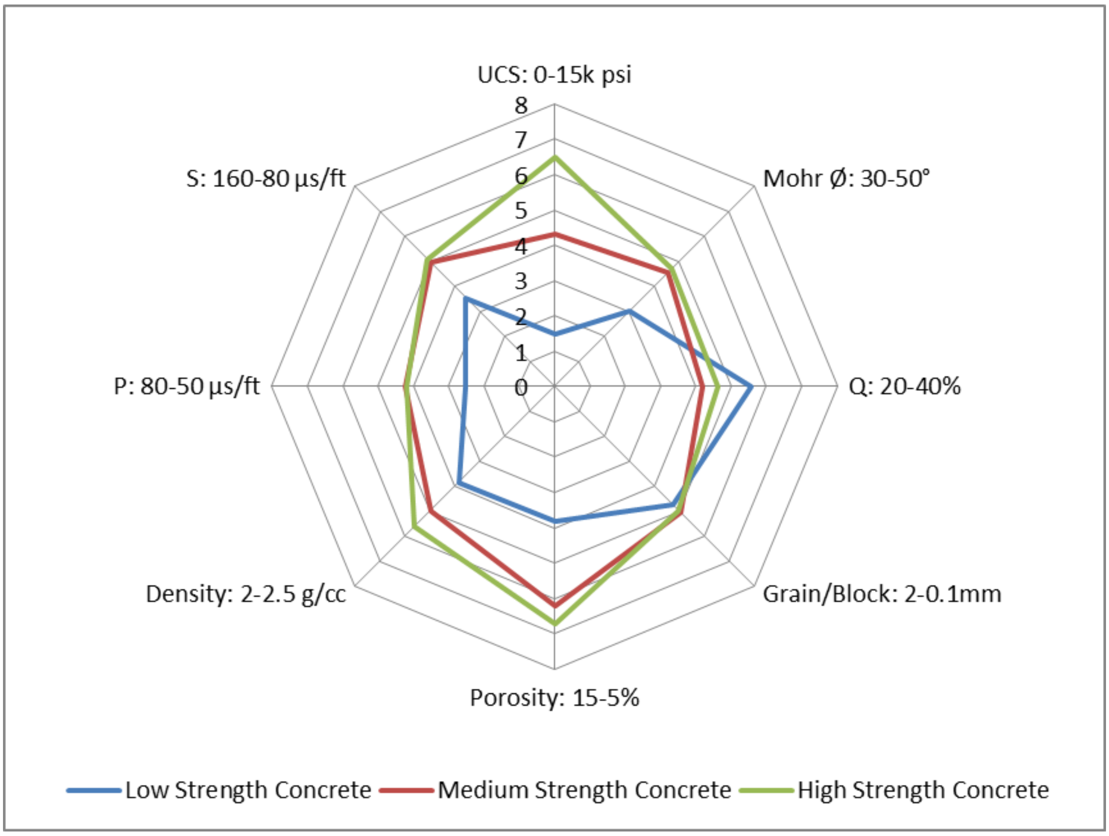


Figure 52 Spider plot of all three strength concrete

5.8.3 Conclusions of eight parameters tests

1) The results of eight parameter tests can quantify the physical properties of synthetic rocks and provide a standard for the synthetic rock designed.
2) Regarding Figure 52, all properties of three concrete designs have pattern comparing with the properties of real rock in Figure 51. The curves in Figure 51 are interlaced and curves in Figure 52 are not. This phenomenon is because the synthetic rocks are designed using the same materials for all three strengths and only the percentage of materials are changed. Real rocks consist of different minerals and mineraloids which can present different physical properties. Therefore, synthetic rock didn't show significant difference in properties without

the diverse minerals and mineraloids.

3) Grain size values and quartz content values in synthetic rocks (Figure 52) are close for all three strengths because the minerals are only come from fine aggregate and same fine aggregate is used in all three strengths.

6. Studies involving the developed Rock-Like Materials

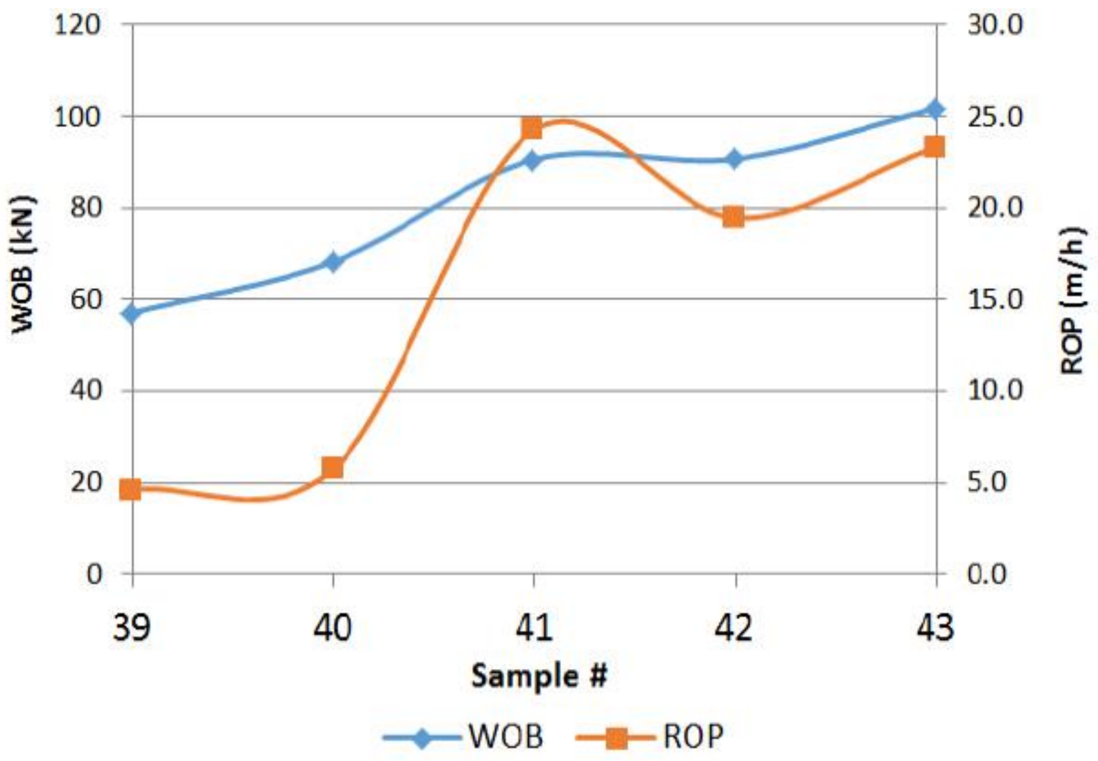
The Rock-Like Materials (RLM) developed in this work were designed to be a possible substitute for real rock in laboratory studies. This RLM development took place in the Drilling Technology Laboratory (DTL) research program. Field work by the DTL group, involving drilling tests in natural rock as well as further laboratory work using both RLM and rock taking from the field drilling site, provided an opportunity to compare the performance of RLM and natural rock. This chapter presents some results of those tests. This includes cuttings analysis, bit hydraulics and isotropy studies. Similarity was found in some respects between RLM and shale rocks.

6.1 Cuttings analysis

Cuttings analysis can provide geological information during drilling process. Petro-physical properties such as porosity and permeability can be determined by cuttings analysis [31]. Furthermore, recently study shows that cuttings analysis can evaluate the cutting mechanism and drilling performance using a passive Vibration Assisted Rotary Drilling (pVARD) tool [32]. In this research, the cuttings samples of shale rocks were obtained from the rotary drilling process performed at field site which located at Conception Bay South, Newfoundland, Canada. Therefore, detailed cuttings analysis of shale rock was conducted and ready to be used as field tests data. On the other hand, cuttings analysis using RLM was conducted at Memorial University of Newfoundland's laboratory by my colleagues [33].

6.1.1 Cuttings analysis results of shale rock

Rate of Penetration (ROP) is the measurement of drilling speed that drill bit penetrate through rock formation. Weight on Bit (WOB) is the downward loads applied on the drill bit while drilling process. Regarding Figure 53 shown in paper "ARMA 15-764 Cuttings Analysis for Rotary Drilling Penetration Mechanisms and Performance Evaluation" [32], ROP and cuttings size (CI, d) have a mainly positive relationship. Reyes, R., Kyzym, I. and Rana P.S. have collected and analyzed data including ROP, WOB and cutting size on Red Shale obtained from field test. Increased WOB and ROP result in the cutting sizes increasing can be concluded from Red Shale samples number 39 to 43 shown in Figure 53 [32].



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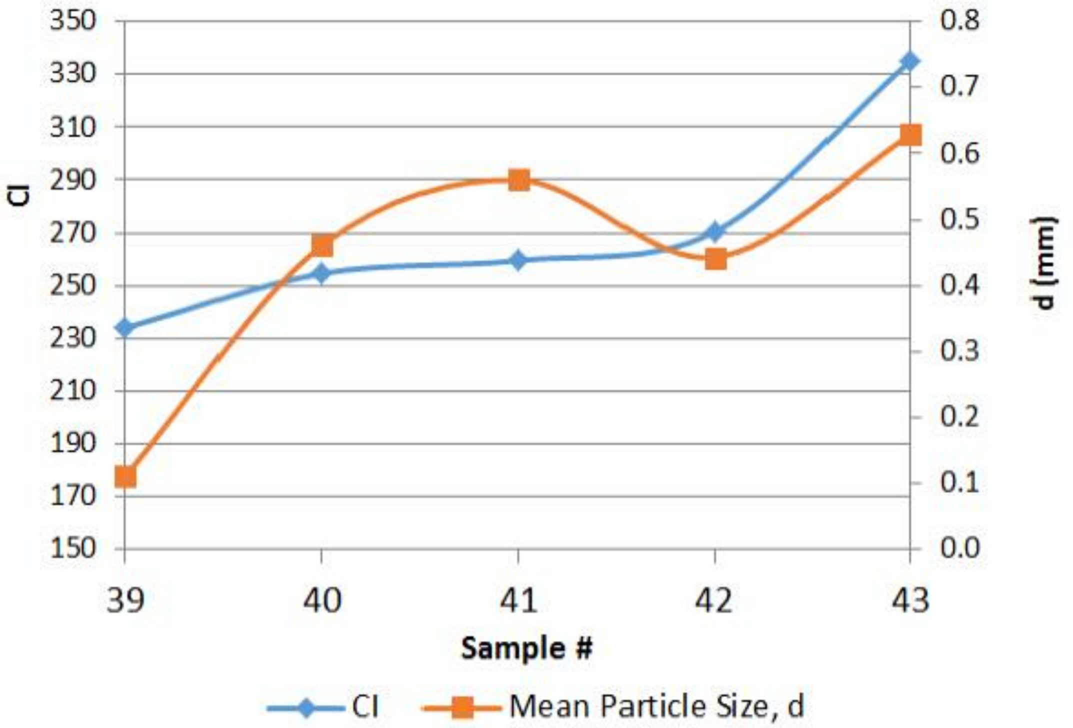

Figure 53 Correlations between cutting size and ROP of Red Shale rock [32] (Reyes et al., 2015, ARMA 15-764)

6.1.2 Cuttings analysis of RLM

According to Figure 54 shown in paper "ARMA 15-474 Micro-Seismic Accounts for Improved PDC Bit Drilling Performance during Vibration Assisted Rotational Drilling" [33], higher ROP with increase of WOB will result in bigger cutting size.

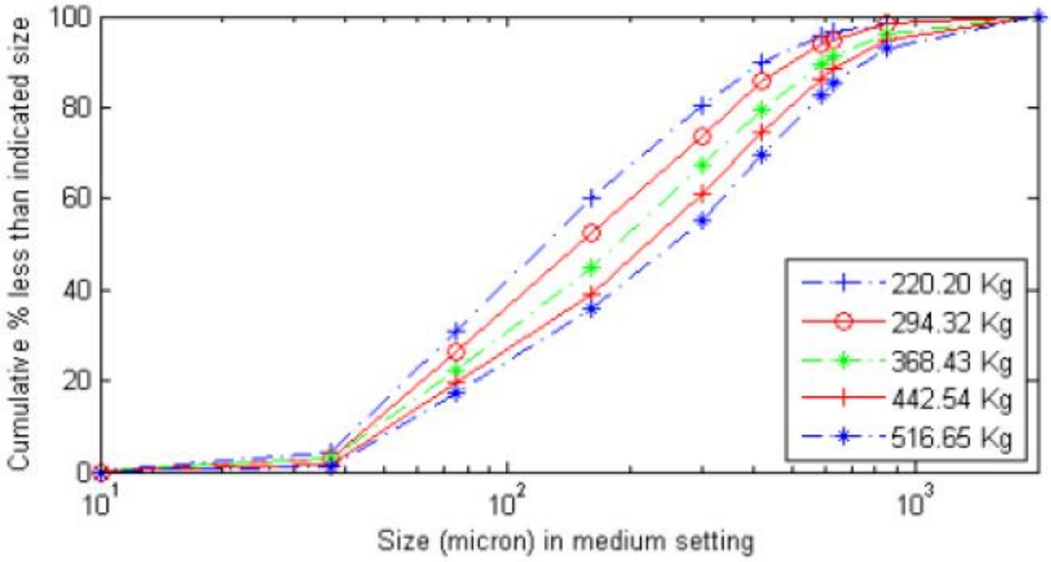

Figure 54 Correlations between cutting size distribution and WOB with medium compliance [33] (Xiao et al., 2015, ARMA 15-474)

The general trend indicates that higher WOB tends to generate bigger cutting size as Xiao, Y. mentioned in his paper regarding tests conducted on RLM [33]. Therefore, both cuttings analyses of Red Shale rock and RLM give same trend which is higher ROP generate bigger cutting size. Cuttings analyses of Red Shale rock and RLM provide the proof of validation of synthetic rocks.

6.2 Bit hydraulics

Bit hydraulics has always been studied in drilling industry and optimization of hydraulics is a key topic in several researches. With optimized hydraulic parameters, reduced bit balling, more efficient energy transmission and cuttings transportation will significantly increase rate of penetration (ROP) [34]. Khorshidian, H studied how the bottom hole pressure (BHP) and flow rate influence the ROP using medium strength RLM described in this research [35]. Regarding Figure 55 in his paper, bottom hole pressure have a negative effect on ROP because of rock strengthening and flow rate however mainly have a positive relationship with ROP because of cleaning efficiency.

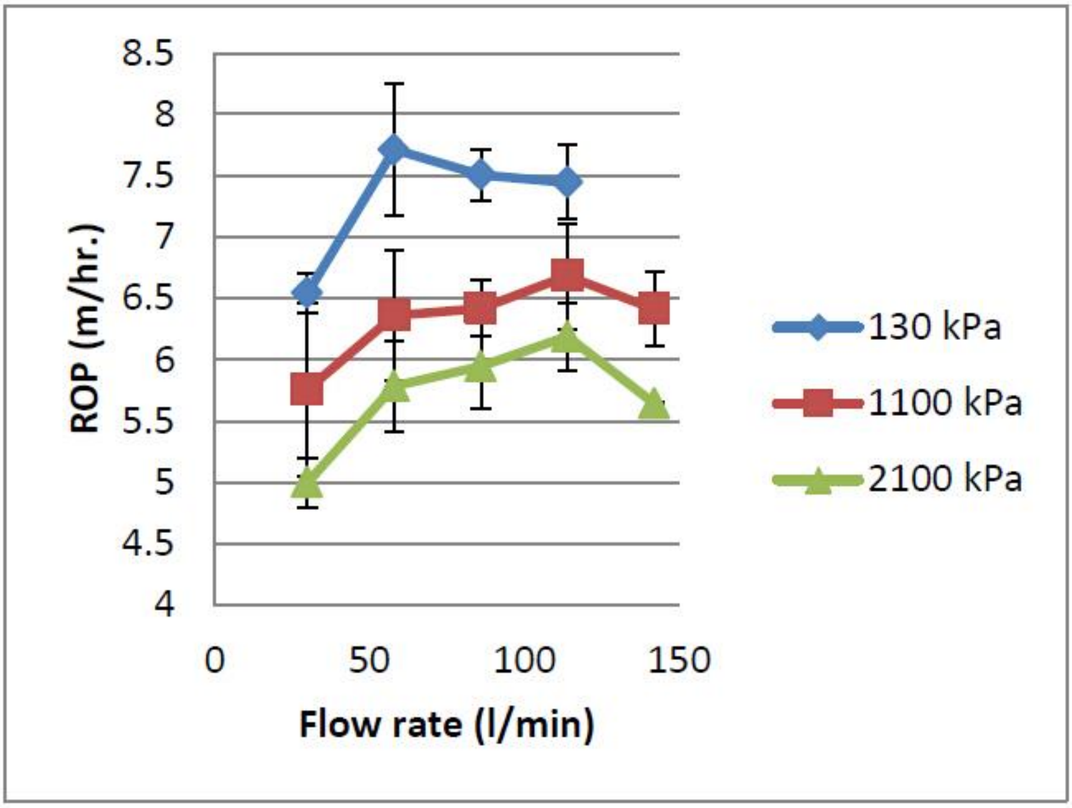

Figure 55 Correlations between ROP and hydraulic parameters of medium RLM [35]

(Khorshidian et al., 2014, ARMA 14-7465)

Khorshidian, H. explained the borehole pressure contributes to the reduction of ROP in two mechanisms: the borehole pressure causes the rock strengthening and intensifies the accumulation of cutting material beneath the cutter; the exit of crushed material from the zone of penetration yields additional confinement on the rock surface [35]. Appropriate bottom hole cleaning condition can improve the drill bit performance through cleaning the generated cuttings material by increasing flow rate. Therefore, increased bottom hole pressure decreased ROP because pressurized bottom hole pressed and hardened the bottom hole rocks; and increased flow rate improved ROP due to higher cleaning efficiency.

6.3 Isotropy Studies

Abugharara, A.N have conducted a few sets of experiments to determine the isotropy of RLM and Red Shale rock [36, 37]. Physical properties, mechanical properties and drilling performance were measured with three orientations (0º, 45º and 90º) in his research. Demonstrations of three orientations of RLM and Red Shale rock are shown in Figure 56 and Figure 57, respectively.

Figure 56 Orientations of RLM in measurements and tests [36] (Abugharara et al., 2016, OMAE2016)

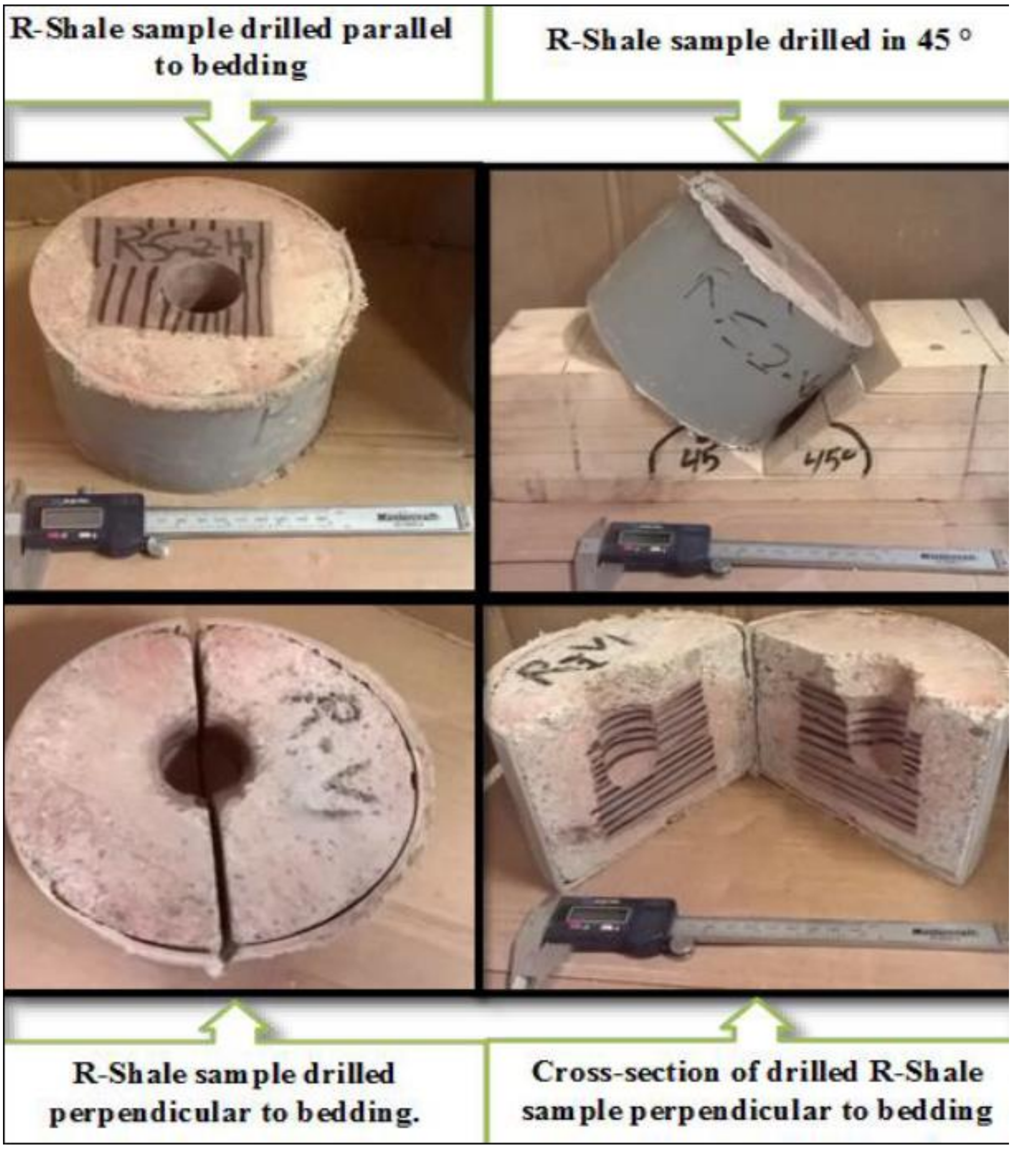

Figure 57 Orientations of Red Shale rock in measurements and tests [37] (Abugharara et al., 2016, ARMA 16-868)

6.3.1 Isotropy of RLM

Measurements and tests of RLM were conducted with three orientations, which including compressional and shear wave velocities, density, dynamic elastic moduli, Point Load strength index, Indirect Tensile strength and UCS values. The results of these tests are shown from Figure 58 to Figure 64.

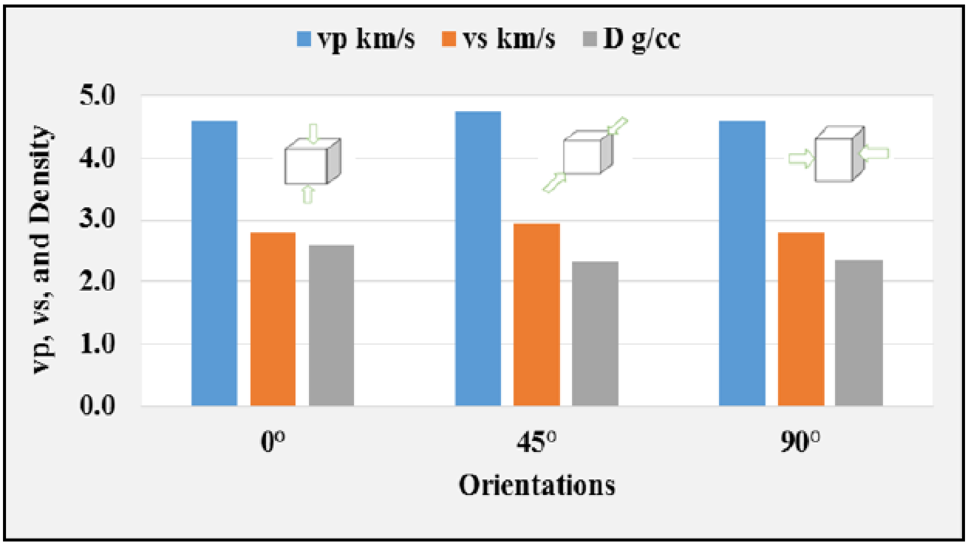

Figure 58 Measurements of wave velocities and density of RLM in orientations [37]

(Abugharara et al., 2016, ARMA 16-868)

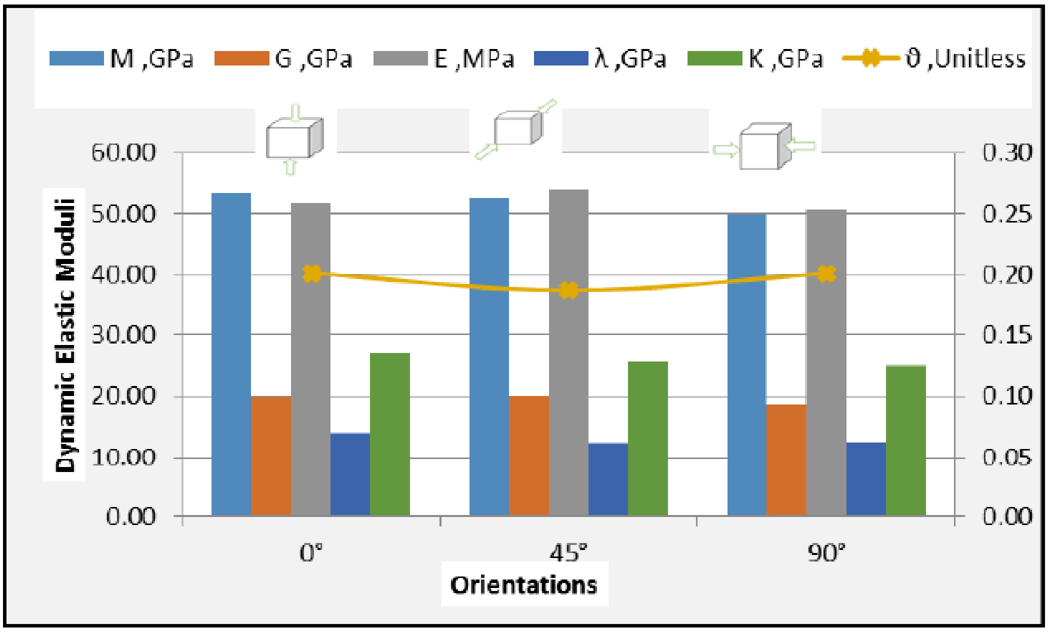

Figure 59 Dynamic elastic moduli of RLM with orientations [37] (Abugharara et al., 2016, ARMA 16-868)

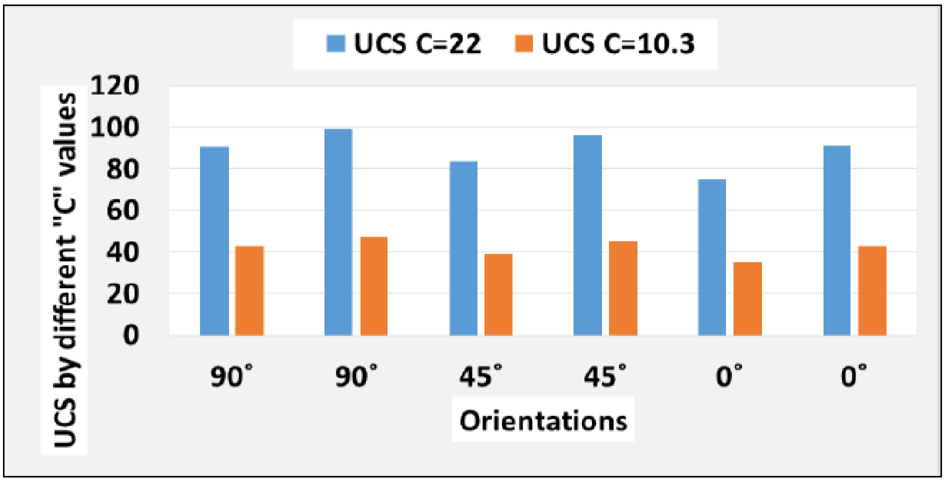

Figure 60 Point load strength of RLM with different C factor values [36] (Abugharara et al., 2016, OMAE2016)

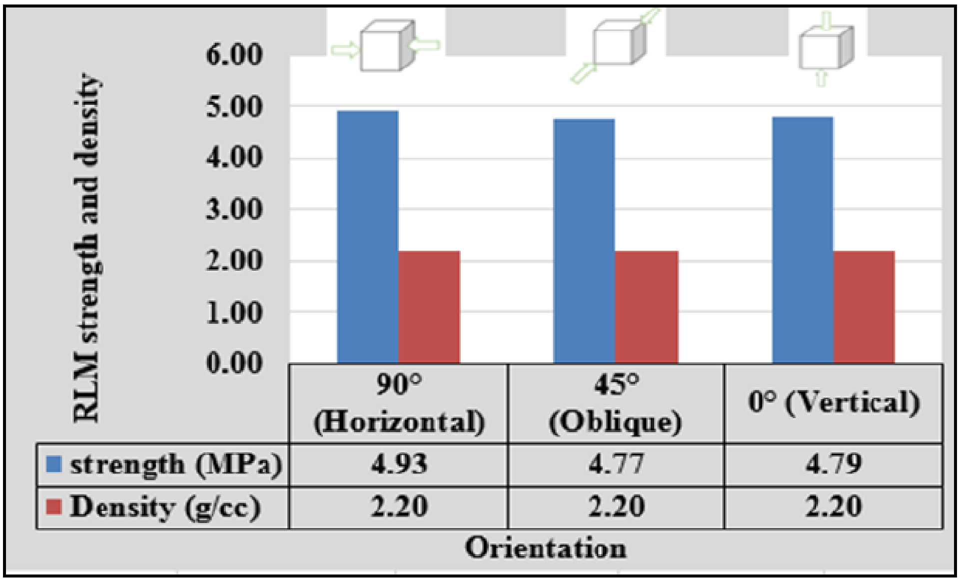

Figure 61 Indirect tensile strength of RLM using splitting tests [37] (Abugharara et al., 2016, ARMA 16-868)

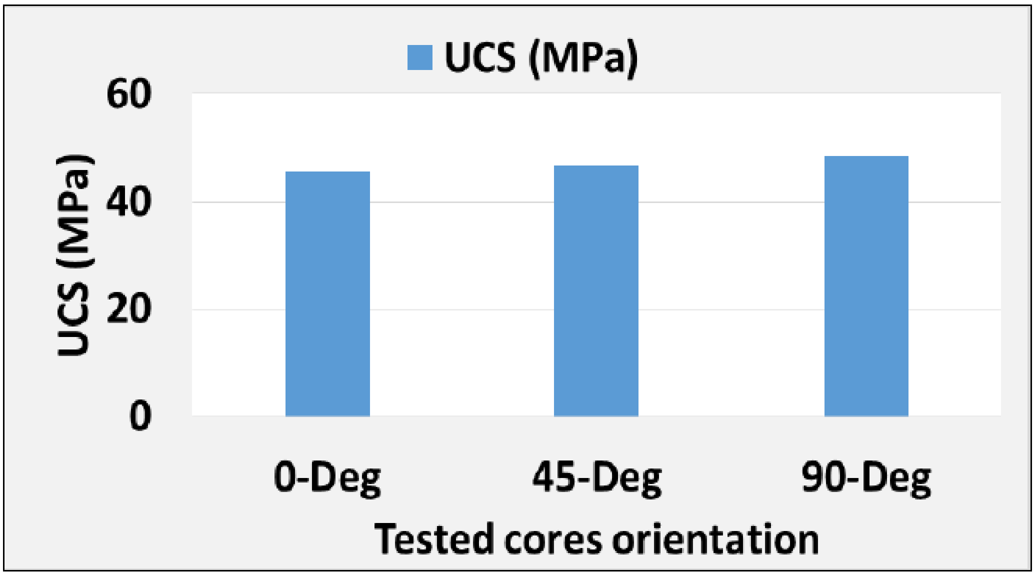

Figure 62 UCS values of RLM in orientations [36] (Abugharara et al., 2016, OMAE2016)

Regarding previous measurements and tests results of RLM with three orientations, RLM is confirmed as an isotropic material. Both drilling performance data and cutting analysis results of RLM shown in Figure 63 and Figure 64 proved that.

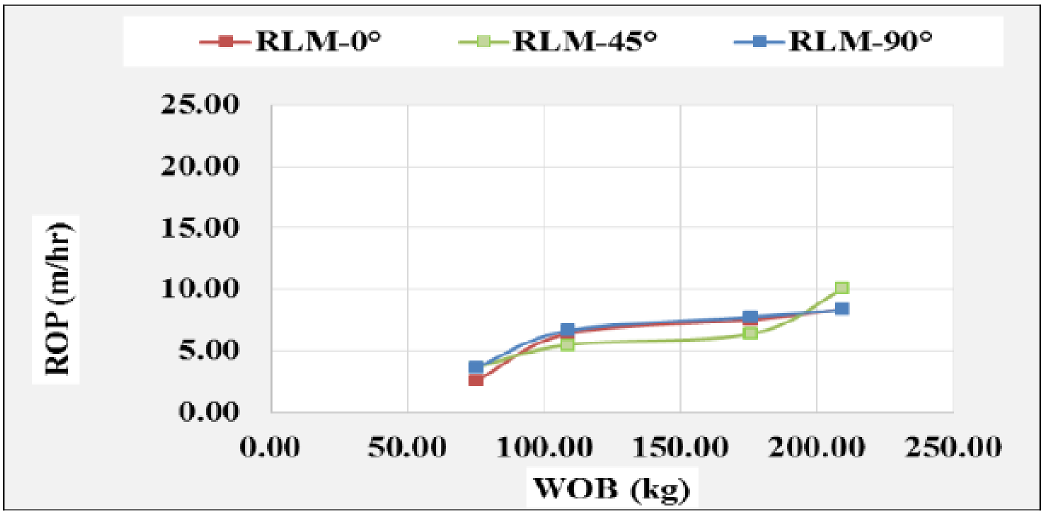

Figure 63 Matching ROP results of RLM with three orientations [36] (Abugharara et al., 2016, OMAE2016)

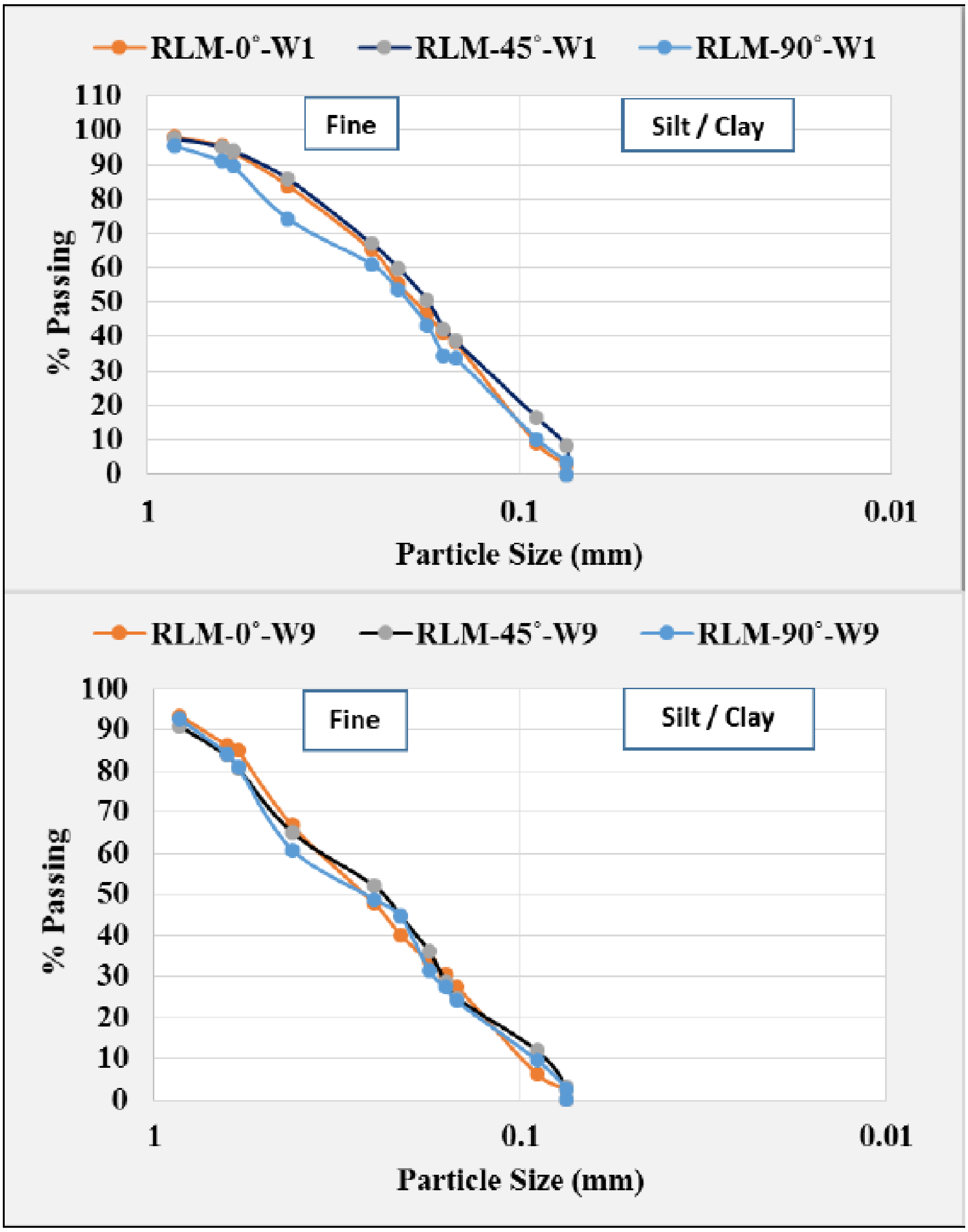

Figure 64 Matching cutting distributions of RLM with two different WOBs in three orientations [37] (Abugharara et al., 2016, ARMA 16-868)

Physical properties using acoustic wave measurement and mechanical properties using destructive tests determined RLM is an isotropic material while drilling tests results provide same conclusion.

6.3.2 Anisotropy of Red Shale

On the contrary, real rock like Red Shale acquired at field tests has shown the material anisotropy, which can be observed from Figure 65 to Figure 67.

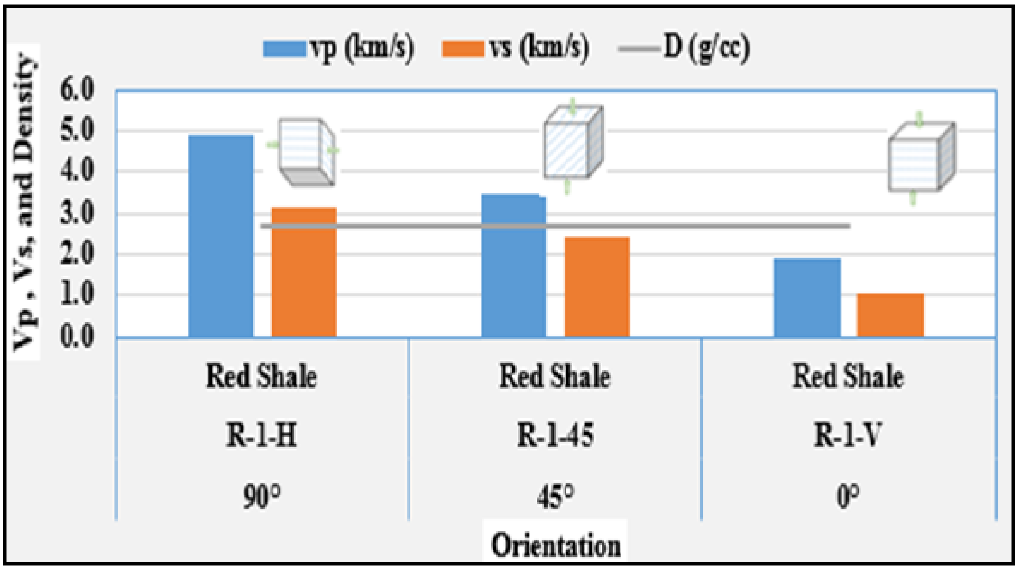

Figure 65 Wave velocities and density of Red Shale in three orientations [37] (Abugharara et al., 2016, ARMA 16-868)

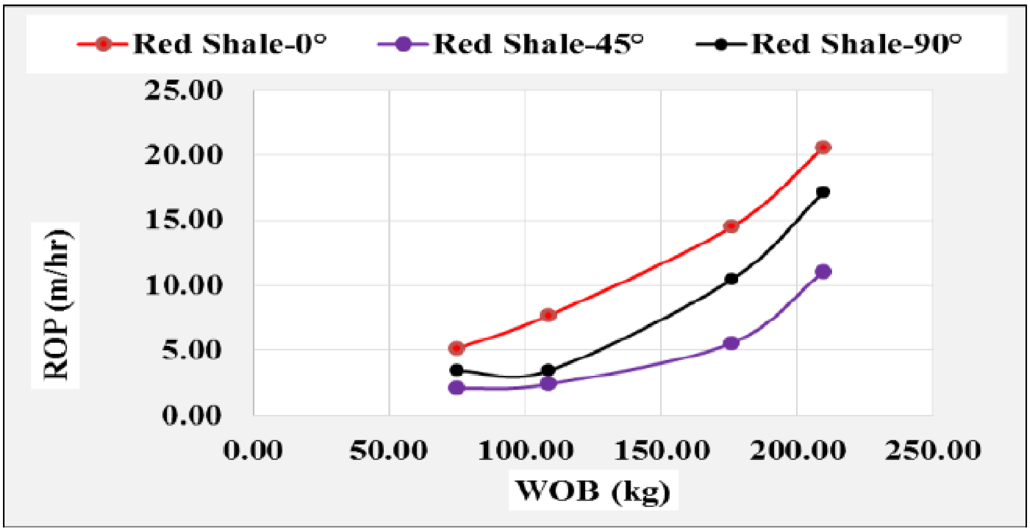

Figure 66 Drilling performance of Red Shale in three orientations [36] (Abugharara et al., 2016, OMAE2016)

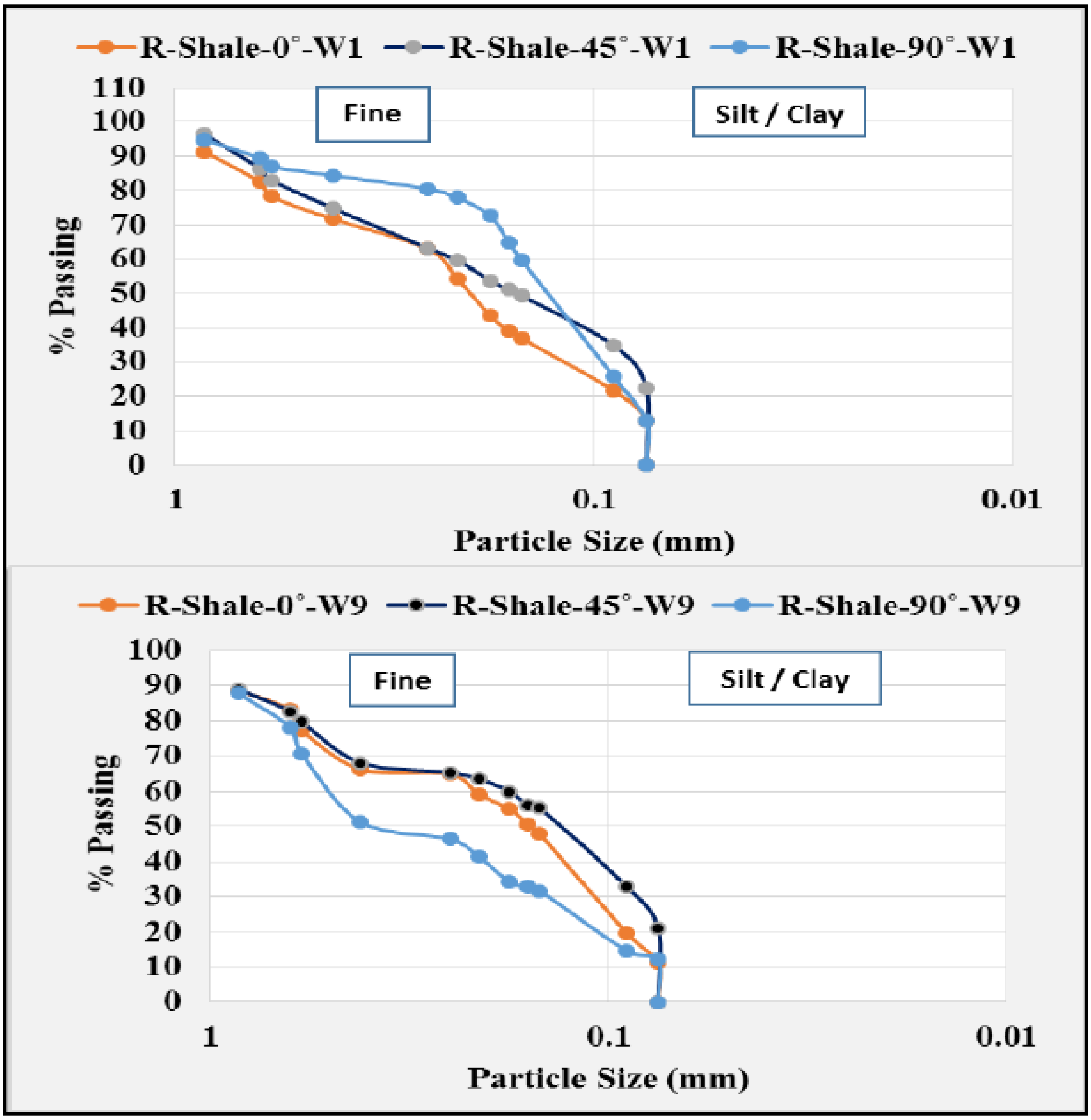

Figure 67 Mismatching cutting distributions of Red Shale with two different WOBs in three orientations [37] (Abugharara et al., 2016, ARMA 16-868)

In conclusion, RLM has shown material isotropy during the physical properties measurements, mechanical properties tests and drilling tests while Red Shale has shown material anisotropy. Hence, RLM is only an appropriate substitute of real rock when the isotropy issue is not involved. Future experiments and simulations might affected by isotropy feature should be conducted with isotropy studies as well.

7. Conclusions and Future Work

In this research, concrete mixes using only fine aggregate (plus Portland Cement, water and appropriate additives) have been developed. These mixes are aimed at a low strength (19 MPa), medium strength (56 MPa) and high strength (84 MPa), to be used as synthetic rock - also referred to as Rock Like Materials (RLM) have been successfully designed to meet the requirements for lab tests. Repeatability and reproducibility studies have demonstrated the consistency of three types of RLM. In this respect, as well as in cost, these RLM are expected to be a useful stand-in for real rock of similar strengths, particularly sedimentary rocks such as limestone and shale. Standard procedures of making these samples have been established. If any other strength value of concrete for this purpose needs to be designed in the future, it should be possible derive that from these mixes.

Eight parameters tests have been conducted on samples of these RLM at all three strengths. The three concretes are expected to be useful simulations in laboratory studies. However, as the same ingredients, fine aggregate and cement, were used in making the concrete RLM, properties such as grain size and quartz content are the same in all three forms of RLM developed. This is one limitation of this synthetic rock. Nevertheless, research in this thesis provided standard procedures for testing the eight parameters recommended by Prasad [1] for evaluating drillability, including determination of fundamental physical properties of all three strengths of the RLM.

Quality assurance, standard procedures of making concrete samples and the eight parameter tests used will provide guidelines for future experimental work using designed concrete samples. In a word, future experimental tests in the lab will benefit from the data acquired in this research.

The drilling performance of the RLM was compared with that of shale. Both cuttings analysis and bit hydraulics have shown promising results, indicating that RLM can be a successful substitute of shale rock, though, as determined by Abugharara et.al. [36, 37] RLM is an isotropic material and Red Shale rock is an anisotropic material, and this difference must be taken into account.

In this research, physico-mechanical and micro-structural properties of designed synthetic rocks were investigated based on the methods proposed by Prasad [1]. All the test methods are designed for the real rocks and were modified to be suitable for concrete. Therefore, the same tests should be conducted in the future on real sedimentary rocks, as well as on RLM, and compared. This could facilitate further progress in investigating rock properties related to the drilling process.

Reference

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