SCALE DEPENDENT OPTIMIZATION OF PETROPHYSICAL PROPERTIES

A DISSERTATION

Submitted in partial fulfilment of the

Requirements for the award of the degree

Of

INTEGRATED MASTER OF TECHNOLOGY

In

GEOLOGICAL TECHNOLOGY

By

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MAY 2019

INDIAN INSTITUTE OF TECHNOLOGY ROORKEE

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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the dissertation entitled "**Scale Dependent Optimization of Petrophysical Properties**" in partial fulfilment of the requirements for the award of the Degree of Integrated Master of Technology in Geological Technology and submitted in the Department of Earth Sciences of the Indian Institute of Technology Roorkee, Roorkee is an authentic record of my own work carried out during a period from May, 2018 to May, 2019 under the supervision of **Dr. Ravi Sharma**, Assistant Professor, Department of Earth Sciences, IIT Roorkee, **Dr. Nachiketa Rai**, Assistant Professor, Department of Earth Sciences, IIT Roorkee and **Dr. Debdutta Ghosh**, Scientist CSIR-CBRI Roorkee.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other Institution.

Date: 15 May, 2019 **(Jeeshan Ahmad)**

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

Dr. Debdutta Ghosh	**Dr. Nachiketa Rai**	**Dr. Ravi Sharma**
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Acknowledgement

At the very outset , I would like to express my soulful gratitude, sincere appreciations and a big thanks to my guide **Dr. Ravi Sharma**, Assistant Professor and **Dr. Nachiketa Rai** Assistant Professor, Department of Earth Sciences, IIT Roorkee for his invaluable guidance, expert suggestions, immense knowledge, constant engagement and encouragement throughout the entire course of my dissertation work with his personal attention and interest, without which it would have become very difficult and cumbersome for me to complete this dissertation work in an efficient manner.

I would also like to thanks **Dr. Debdutta Ghosh**, Scientist at CSIR-CBRI, Roorkee for taking ultrasonic measurement in his lab and his support in providing infromation and software needed to interpret the data. Without his help this project would not go at that level.

I would also like to express my sincere and profound gratitude to **Prof. Sunil Bajpai**, the Head of the Department, Department of Earth Sciences, IIT Roorkee, for providing all the necessary facilities and fast administrative help necessary to complete this work. I would also like to thank professor-in- charge **Dr. Govind J. Chakrapani** under whom I got this golden opportunity.

I am heartily thankful to Swati Dahiya, Rajat Goel, and Abhishek for his immense support.

I feel short of words to express my sentiments towards my loving family for their unduly love and fondle, for their everlasting moral support, for the sacrifices they have made on my behalf. Their prayers for me were what sustained me thus far and you have been a constant source of inspiration for me.

Abstract

Porosity plays a major role in oil and gas prospect determination. Hydrocarbons are stored in pore space so quantification of pores is of uttermost important. Water saturation and immersion techniques and other conventional methods have various limitation as all pores are not connected. Development of imaging techniques and computational capacity has taken the characterization of pores on different scales. In this work five different rocks Sandstone, Granite Gneiss

, Dolomite, Limestone and Meta-Basalt are taken for detailed analysis on porosity determination and its manifestation on physical properties such as velocity. Porosity was distinctly different for different rocks. It was found that porosity measurement could be method and scale dependent within the same rock.

Three measurements techniques were used for measuring porosity (i) Porosity calculation technique using a water immersion saturation technique (ii) Surface (Areal) Porosity calculation by using SEM images different resolution. (iii) Total porosity calculation from ultrasonic measurement. There is total of 5 order of magnitude difference in resolution across different techniques used for porosity measurements. The quantitative measurement of pores in reservoirs rocks like sandstone, limestone, dolomite at different scale have implication in developing exploitation strategies. As all rocks have range of porosity so, field porosity is assumed to be equal to that calculated from water immersion saturation technique.

Elastic modulus of all rocks is calculated by ultrasonic method which is non-destructive method which depends on mineralogy, grain size, porosity and density. From the obtained results it was almost found same correlation between porosity and different elastic modulus in all rock types. Modulus of elasticity is decreasing with increase in porosity. The results show that scale of observation should be different for different rock types and generally can take 200μm scale of observation for porosity calculation. Dolomite showing exceptionally low porosity at scale of 1 mm and Granite showing exceptionally high porosity by ultrasonic methods (10 cm scale).

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Chapter 1: Introduction

Porous rocks are very important rocks for hydrocarbons. A pore is the void or empty space embedded within the solid matrix of porous rocks. Presence of majority of small pores within natural occurring rocks cannot be easily perceived by naked eye and if perceived it quantification manually can't be possible. Almost each and every mechanism occurring in porous rocks is indirectly or directly related to pores. This void of the rock must be understood in order to understand the behaviour of porous rock. Accurate and precise quantitative understanding of pores is the fundamental requirement for all engineering fields dealing in porous rocks. For hydrocarbon industries quantification of pores have economic implication. The hydrocarbons are stored within the pores and flows though the connected pores. Proper quantification of total pore volume help in estimation of total hydrocarbon present in the reservoirs. Some minerals which deformed/swell after saturated with water or any other fluid damage the rock.

The lack of standardized scale of measurements and uncertainty related to size of rock sample size can lead to significant error in the quantification of pore volume by different methods and also in the economic assessment of hydrocarbon industries. Different industries used different methods for calculating porosity for different rock types. With advancement in the field of image acquiring and processing industries are moving towards digital method. Despite the advantages and disadvantages of conventional methods which are mostly destructive methods. In this project I have adopted two simple, non-conventional and constructive approaches to predict the porosity of different rock types. The first method is based on the SEM images taken at different resolutions using Fiji software. The second method used is ultrasonic wave propagation method to predict the porosity. The SEM images approach is directly unable to determine three-dimensional porosity of the porous rocks due to 2D image. To calculate the bulk porosity of the rock, surface porosity of the rock from the two ends are extrapolated by doing average. **Five different types of rocks are taken like Dolomite, Granitic gneiss, Meta-Basalt, Sandstone, Limestone.**

Comparisons of porosity measurements on mudrocks from immersion methods, helium pycnometry, and mercury porosimetry demonstrate large inconsistencies between the methods (Dorsch et al., 1996). Significant discrepancies exist in porosity and grain density values of comparable samples measured by different commercial laboratories using the GRI method (Karastathis, 2007; Passey et al., 2010; Sondergeld et al., 2010; Spears et al., 2011)

1.1. Objective

The importance of this research is in understanding the dependence of scale of measurement in porosity. All type of measurement are not available at the same time due to economic, location constrain.

- The objective of project to introduce a simple, non-conventional and non-destructive approach to find the porosity at different scale of measurements for different types of rocks and its correlation with field porosity of that rock.
- Using another non-destructive method such ultrasonic wave propagation method (seismic rock properties, for example Vp, Vs, shear modulus, bulk modulus, bulk density) to conform the field values obtained in step one.

Chapter 2: Literature

2.1. Terminology

Pore

A discrete void within rock which can contain water, air, hydrocarbons or any other fluids (Schlumberger oil field Glossary).

Pore Structure/Size

Porosity does not represent the quality of pore structure or heterogeneity that occur in nature. To understand the qualitative nature of porosity, study of pore size distribution is necessary.

The International Union of Pure and Applied Chemistry [IUPAC] (Sing et al., 1985) recommend the following classification of pores according to their size:

1. Micropores: pores with pore-size less than 2 nm

2. Mesopores: Pores with pore-size between 2 nm to 50 nm

3. Macropores: Pores with pore-size greater than 50 nm

Porosity

Porosity is defined as the fraction of bulk volume that is occupied by void or pore space (Dullien,1991). The portion of rock that is occupied by solid material is the grain and that is occupied by fluid is the pore. Porosity is single value quantification (Nimmo, J. R. 2004)

$$Porosity = \frac{Volume\ of\ void}{Total\ volume}$$

$$Porosity = 1 - \frac{Bulk\ Density}{Particle\ Density}$$

Bulk Density

It is defined as the ratio of total weight of rock and total volume of rock. For uniform sample it is easier to calculate the bulk density by measuring dimension and weight of sample. Cylindrical cores are taken to calculate the bulk density. Dimensions of the core are measured using Vernier calliper with a least count of 0.01 mm and weight is measured using weighing machine having least count of .01 gm.

Particle density

It is also called grain density. It is the ratio of mass of the solid rock matrix and volume of the solid rock matrix. It is always greater than bulk density because some of the volume is occupied by the fluid which has negligible mass compared to solid rock matrix.

Dry Density

It is the density of the rock when the rock is fully dried. It is free from all types of fluid.

Wet Density

It is the density of the rock when the pores are fully or partially saturated with the fluid. Wet density is always greater than dry density.

This table of grain density is acquired from the literature

Table 1 Grain density of common minerals (P.V Sharma 1997)

Mineral Name	Grain density
Calcite	2.71
Dolomite	2.87
Olivine	3.22
Plagioclase feldspar	2.65
Quartz	2.65

Table 2 Bulk density of rocks (P.V Sharma 1997)

Rocks	Bulk Density(g/cc)
Limestone	2.5-2.75
Sandstone	2.35-2.65
Gneiss	2.6-2.9
Basalt	2.7-3.3
Granite	2.52-2.81

Shale	2.65-2.75

Table 3 Calculated Bulk Density of rocks

Rock	Bulk density (calculated)
Meta-Basalt	2.99
Limestone	2.81
Sandstone	2.81
Granite Gneiss	2.60
Dolomite	2.50

2.2. Elastic Modulus

Elastic properties such as Bulk modulus(K), shear modulus (modulus of rigidity) (G) and Young's modulus(E) are very important parameters for the well bore stability. These parameters can be calculated indirectly in the lab from ultrasonic wave propagation method. Conventional method of calculating the modulus is time consuming, destructive method. Ultrasonic method uses transition time to travel elastic pulse between the two end points of rock. Both compressional wave velocity (Vp) and shear wave velocities (Vs) are used for calculating elastic properties. The velocity of elastic waves in rocks depends on density, grain size, porosity, stress level, mineralogy, stress level, water absorption and temperature.

2.2.1. Young's Modulus

It is the mechanical or elastic property that measure the stiffness of solid body. It is also defined as ratio of stress and strain for uniaxial deformation. Youngs modulus is named after the scientist Thomas Young.

Table 4 Modulus of elasticity of common rocks (jsg.utexas.edu)

Rock	Bulk modulus (GPa)	Shear modulus (GPa)	Young's modulus (GPa)
Granite	50	24	10-70
Limestone	65	24	15-55

sandstone	0.7	0.4	1-20

$$E = \frac{\rho Vs2(3Vp2 - 4Vs2)}{Vp2 - Vs2} = \frac{stress}{strain}$$

2.2.2. Shear Modulus

It is also the mechanical properties that resist transverse deformation, mathematically it is defined as the ratio of shear stress and shear strain. It is zero for fluids

$$Vs = \sqrt{\frac{G}{\rho}} = \frac{shear\ stress}{shear\ strain}$$

2.2.3. Bulk Modulus

It is the measure of ability of substance to resist changes in volume when pressure is applied from all sides, mathematically it is defined as the ratio of applied pressure and relative deformation in volume.

$$Vp = \sqrt{\frac{K + (\frac{4}{3})G}{\rho}} = -\frac{P}{\frac{\Delta V}{V}}$$

Where, V_P= Compressional wave velocity

Vs= Shear wave velocity

G= shear modulus

K= Bulk modulus

E= Youngs Modulus

p= Density of rock

P= pressure

V=volume

Chapter 3: Sample Description and Preparation

Five different type of rocks are collected from different locations. They are Meta-Basalt, Limestone, Sandstone, Granitic Gneiss and Dolomite.

3.1. Rock Description

3.1.1. Granite Gneiss

The rock belongs to Greater Himalayan crystalline belt near Munsiari, Uttarakhand. This rock is low grade metamorphized with granitic composition. Evidence of intrusion is the presence of xenolith of amphibolite xenolith. The rock is white in colour with black patches and does not contain bands. The rock is coarse grained having quartz, alkali and plagioclase feldspar and amphibolite. The measured bulk density of the rock is 2.60 g/cc.

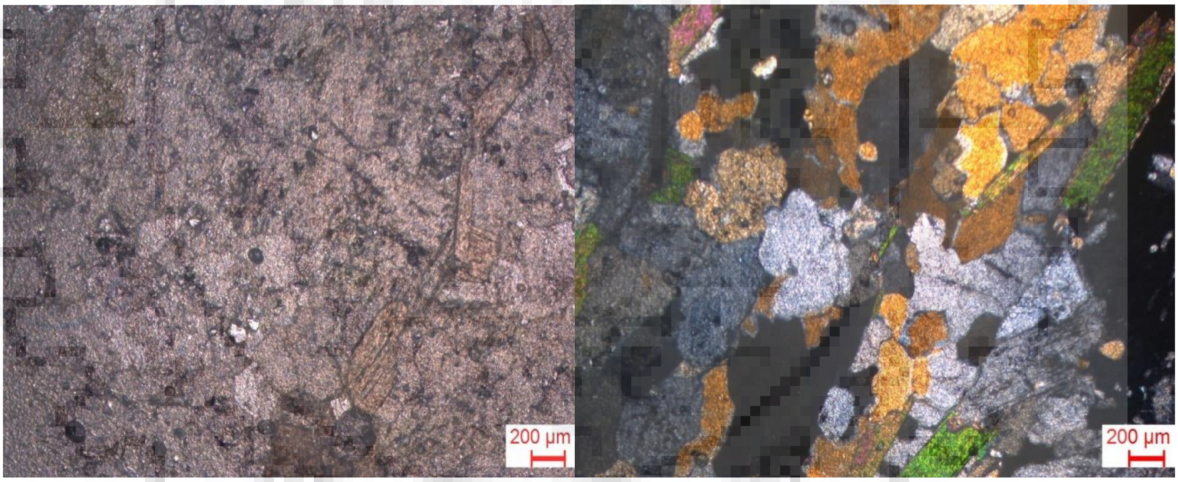

Figure 1 PPL and XPL thin section image of Granitic Gniess

3.1.2. Meta- Basalt

The rock is amygdaloidal Meta-Basalt of Dhanjori lava, Singhbhum shear zone near Masubari, Jharkhand. This is volcanic rocks that are present within the sedimentary rocks. The rock is fine grained and light green and measured bulk density is 2.99 g/cc. Biotite and opaque minerals occur as a accessory minerals. The Meta- basalt are typically actinolite-albite -epidote schist.

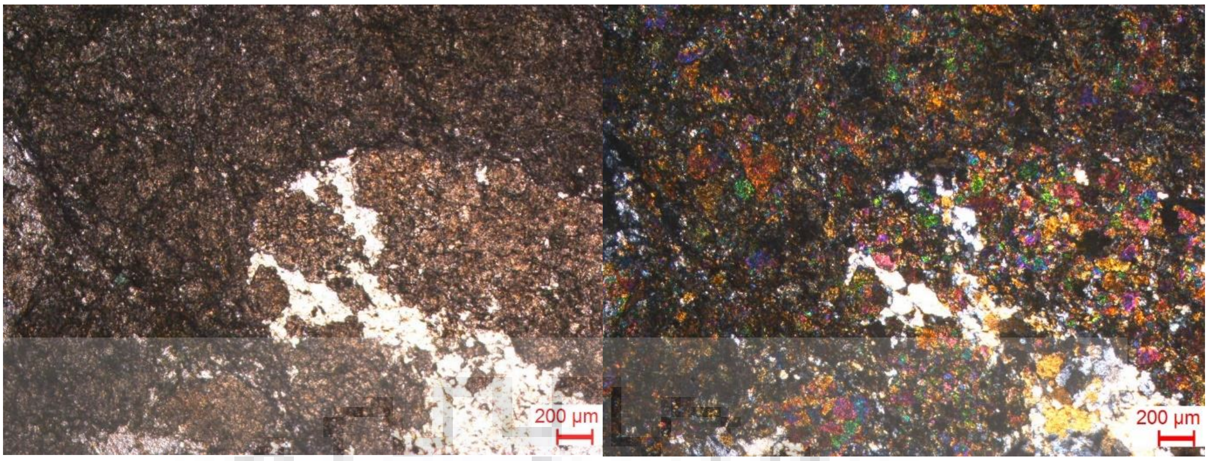

Figure 2 PPL and XPL thin section image of Meta- Basalt

3.1.3. Limestone

This rock belongs to Kutch basin, Gujarat. The rock consists of Calcite, dolomite and some heavy minerals like hematite or magnetite. The rock is grey in colour, fine grain size and density of the rock is 2.88 g/cc.

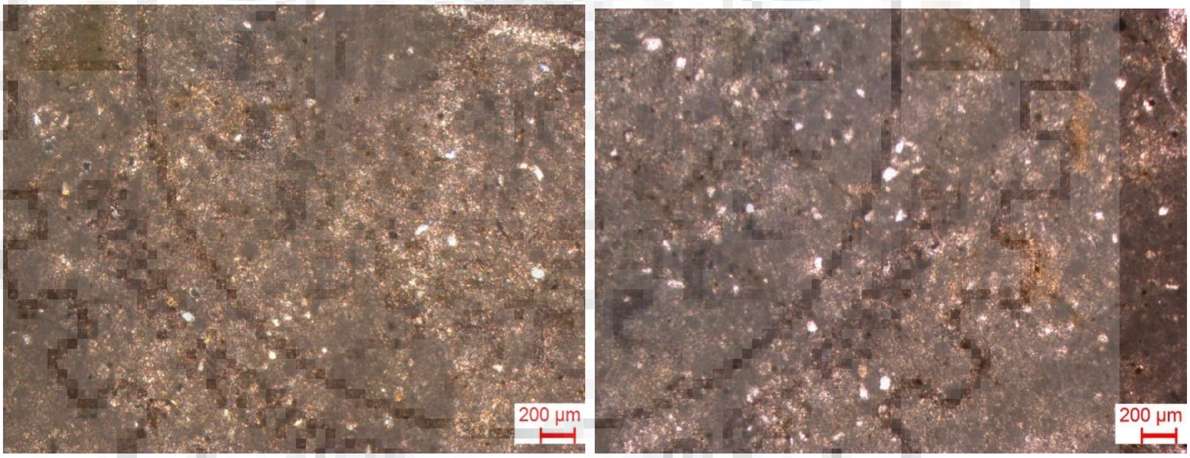

Figure 3 PPL and XPL thin sections image of Limestone

3.1.4. Sandstone

This rock muddy sandstone and is collected from near Ramjhula in Rishikesh, Uttarakhand The rock is reddish in colour due to presence of hematite and finer in grain size. The measured density of the rock is 2.81g/cc which is high than quartz.

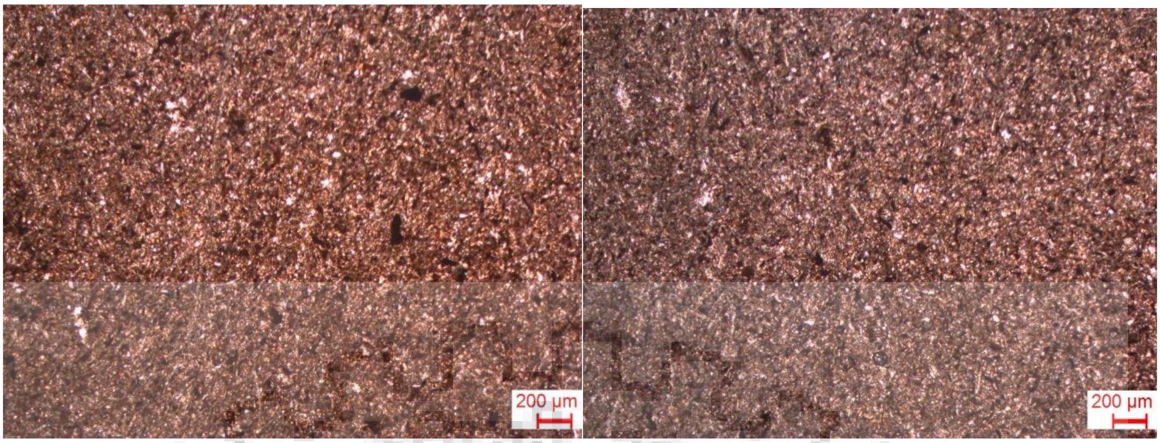

Figure 4 PPL and XPL thin sections image of sandstone

3.1.5. Dolomite

This rock is collected from Kutch, Gujarat. It is oil bearing rock and mainly consist of dolomite, calcite. The rock is yellow in colour and have presence of small vugs. The measured density of the rock is 2.50 g/cc.

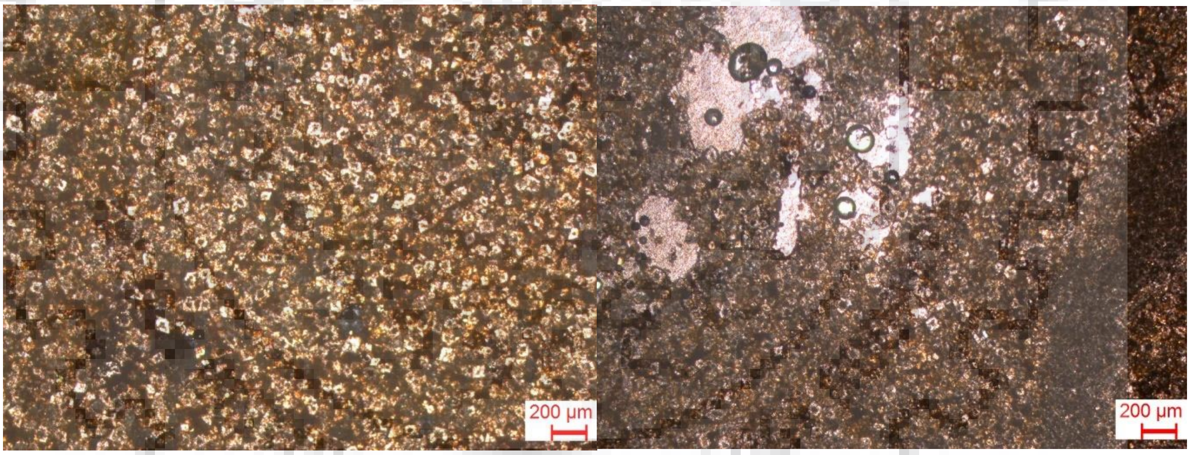

Figure 5 PPL and XPL thin sections image of Dolomite

3.2. Sample Preparation

3.2.1. Coring

Core plug samples are obtained from the irregular shape rocks by doing coring. Coring was done by 35.5 mm of thin walled diamond rotary bits. It contains hollow iron tube in which core is sucked during rotation. Water is used during coring to maintain temperature and to remove the dust. Core plug of approximately 35.5 mm diameter and 10 cm length is obtained for each rock.

3.2.2. Cutting

Cutting is required to make the two ends flat and to cut the chunks from the two opposite ends of rocks for SEM experiment. Cutting is done by rotary blade. During cutting water is used for cooling purpose and for removing dust.

Figure 6 Coring

Figure 7 Cutting

Cylindrical Cores

Core plugs obtained from rotary drilling machine are of different size for different rocks because all rocks have different breaking strength. Cores are obtained to get the rocks of finite dimension which will help in calculating Density, porosity, Modulus of elasticity. They break when strain during rotation exceed the rock strength. Diameter of the cores are almost same for all rocks and is equal to 35.5 mm.

Volume of Cylinder (V)

$$V = 4\pi r^2 h$$

where r= radius of the cylinder

h= length of the cylinder

Table 5 Dimensions and weight of rocks

Sample No.	Rock name	Diameter(mm)	Length(mm)	Weight(g)
1	Meta-Basalt	35.77	103.44	311.00
2	Limestone	35.40	88.51	245.20
3	Sandstone	35.33	63.33	174.60
4	Granite Gneiss	35.50	83.87	215.60
5	Dolomite	35.40	91.93	226.20

Figure 8 Cylindrical Cores of different rocks

3.3. SEM Sample Preparation

This types of the circular chunk that will be used for SEM experiment after polishing.

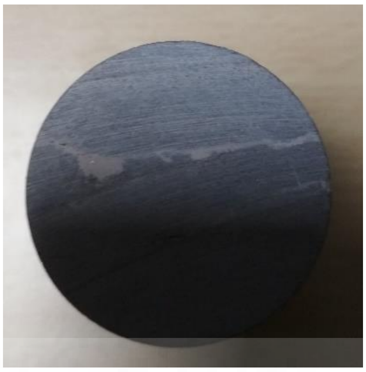

Figure 9 SEM sample

3.3.1. Polishing

The SEM samples/chunks are cut from the two ends of core. Surface of SEM samples are polished by sand papers of Grit sizes of 80, 150, 220, 320, 400, 600 and 800 to remove roughness and make surface smooth to get clear images. The chunks are also polished using polishing machine and using diamond paste for smooth and shining. More is the grit size finer it will make the surface.

Figure 10 Polishing on sand papers

Figure 11 Fine Polishing on Machine

3.3.2. Gold Coating

The surface of rocks are coated for making a non-conductive SEM sample conductive. To achieve clear and better results conductive material should be selected precisely. There is no universal coating material but can be changed depending on the material but most commonly used coating is Gold Coating. Gold coating is done in DC magnetron SEM sputter coater. Before coating Vacuum is created to remove the moisture so that material is not charged. Charge make images unclear. The coating quality depends on target material and its interaction between the target and coating material.

Most of the sample used in this thesis are non-conductive. Coating a target with highly conductive metal like Gold makes non-conductive targets conductive and to avoid the charging of target surface. It will enable to get higher resolution image due to higher target stability under electron beam. Gold have higher secondary electron yield than insulator material. 4-8 nm of gold coating increases the signal to noise ratio which results in getting crisp and clear images. Coating thickness depends on the grain size of the coating material and its interaction with the target and coating material.

The reason of choosing gold coating is that samples does not contain Au concentration which will make hinderance in interpreting the EDX data, second reason is gold is very highly conductive material which avoid charging of the SEM sample. In gold coating process gas used is air. Gold structure is not imaged due to low resolution table top SEM.

Figure 12 Diamond coating

Figure 13 carbon coater

3.3.3. Carbon Coating

Carbon coating is done for Granite Gneiss . In carbon caoting process gas used is Argon gas which is inert gas. Carbon coating is mainly done to get Black Scatter Electron(BSE) image. Carbon is not done in DC magnetron sputter coater.

Figure 14 Coated SEM samples

Chapter 4: Methodology

4.1. Water saturation method (Conventional Method)

Porosity is the most basic reservoir property. Accurate porosity measurements of the formation is still challenging and lack of accurate measurement techniques or scale leads to significant error in assessment of hydrocarbon present. Liquid saturation and immersion techniques have been used for porosity measurements by the American Petroleum Institute (API RP40), the American Society for Testing and Materials (ASTM Standard C20–00; ASTM Standard C830-00), and the International Society for Rock Mechanics (Franklin et al., 1981). The method has been employed in analysis of a wide variety of rock types including low porosity granites (Alexander et al., 1981; Katsube & Kamineni, 1983; Melnyk & Skeet, 1986), oil sands and sandstones (Barnes, 1931; Plummer & Tapp, 1943), dolomites and limestones (Goldstrand et al., 1995).

$$Porosity = \frac{Volume\ of\ void}{Total\ volume} X100$$

Through this equation porosity of all five different rock types are calculated

Assumption

- Deionised water does not react with any mineral or dissolve any mineral content

Ideal saturating fluid should satisfy following criteria.

- Low reactivity with porous rock
- Low viscosity
- Slow evaporation rate and high vapor pressure
- Safe handling properties and non-hazardous
- High wettability and low surface tension

$$\text{Porosity} = \frac{(Ws-Wi)/\rho}{v} X100$$

Where,

Ws=weight of rock after saturation

Wi=weight of rock before saturation

ρ = density of liquid

Rock samples are dip in deionised water of density of 1g/cc for around 72 hours till rock get saturated with water. Mass of the rock is measured before dipping(m_i) and after saturation(m_f). The mass difference before and after saturation gives the amount of water absorbed in the sample. Mass of water absorbed divided with density of water give volume of the water absorbed which is approximately equal to volume of the connected pores. Volume of the rock can be calculated by multiplying weight of the rock with bulk density of the rocks. Deionised water having surface tension 72.7 dynes/cm, dynamic viscosity of 1.002 centipoise and penetration coefficient of 3630.9 cm/s

Figure 15 Water saturating in rock

Deionised water has advantage that exchangeable cations present on clay mineral surface and in layer form shells of water molecules around them which control the water absorption of core. It is also stable and unreactive.

The accuracy of porosity measurement using liquid saturation method depends on immersing fluid saturation efficiency and type of pore network and stability in laboratory conditions (Melnyk

& Skeet, 1986). The driving force to saturate porous rock are surface tension, wettability, and pressure gradient.

Table 6 Weight of rocks before and after saturation

Sample No.	Rock Name	Weight before saturating(g)	Weight of rock after saturation(g)	Voume of void(cc)	Bulk volume(cc)	Porosity(%)
1	Meta Basalt	174.40	174.80	0.40	58.33	0.63
2	Limestone	96.60	97.80	1.20	34.38	2.88
3	Sandstone	128.40	129.20	0.80	45.70	1.55
4	Granite Gneiss	111.20	111.80	0.60	42.70	1.38
5	Dolomite	84.80	88.80	4.00	33.92	10.44

This porosity can be called as effective porosity or connected porosity and it will always be less than total porosity. The porosity calculated from this method will be assumed as a field porosity.

4.2 Ultrasonic Method (Non- Destructive)

3.2.1. Porosity

Ultrasonic wave propagation method is a non-destructive method. Petrophysical properties such as density, saturation and porosity have a relation on acoustic properties of rocks. Simply ultrasonic is a way of studying elastic properties by measuring the time it takes to flow through a medium of known thickness. Sound energy is converted into electrical energy which is recorded. The velocity of acoustic waves in crustal rocks may also vary with mineral composition, texture, and cementation (Vernik ,1994), and may exhibit frequency and scale-dependent dispersion (Rio et al., 1996). Therefore, there is recommendation to use acoustic measurements to calculate the porosity of the rock. There are two elastic waves called compressional/primary (P wave) wave and shear wave/secondary (S wave). The P wave also called compressional wave velocity is non -destructive wave to the rocks. The ultrasonic method of porosity calculation is based on the propagation time of P wave in the direction of receiver and transducer. Ultrasonic velocities are also used to calculate the elastic constant of the rocks. P wave velocity (V_P) vibration direction is in the direction of propagation and for S wave direction of vibration is perpendicular to the direction of propagation. S wave does not propagate in fluids and also slower than P wave velocity. The relationship between density and velocity is considered linear. Transient time depends on the pore space, pore distribution and density. This method is valid for both isotropic and anisotropic rocks.

Figure 16 Ultrasonic measurements

Wave velocity, transit time and waveform are measured from the PROCEQ instrument and plotted on Pundit software. Wave pulse is sent to the rock by electro acoustical transducer and is received by electro acoustic receiver, both of them is mounted on two opposite sides of rock. The length of all the rocks are different so path length covered by sound is different so travel time will be different because it depends on the path length. Transducer and receiver are aligned parallel to get more

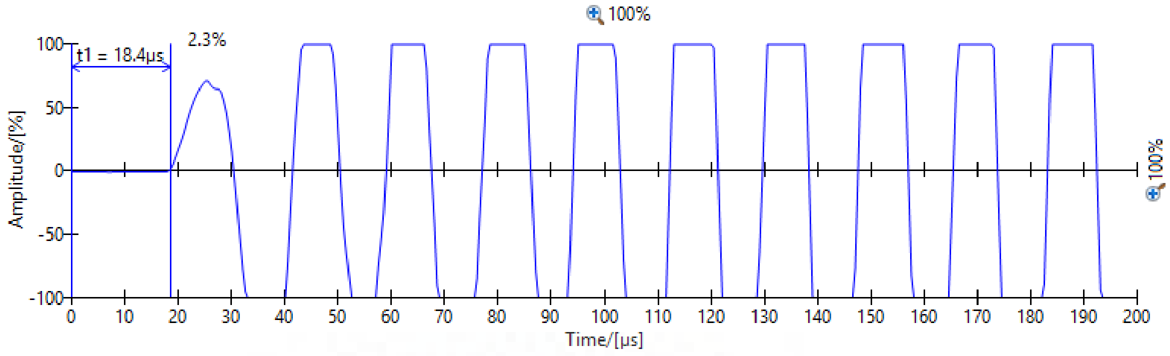

Figure 17 Vp waveform of Meta-Basalt

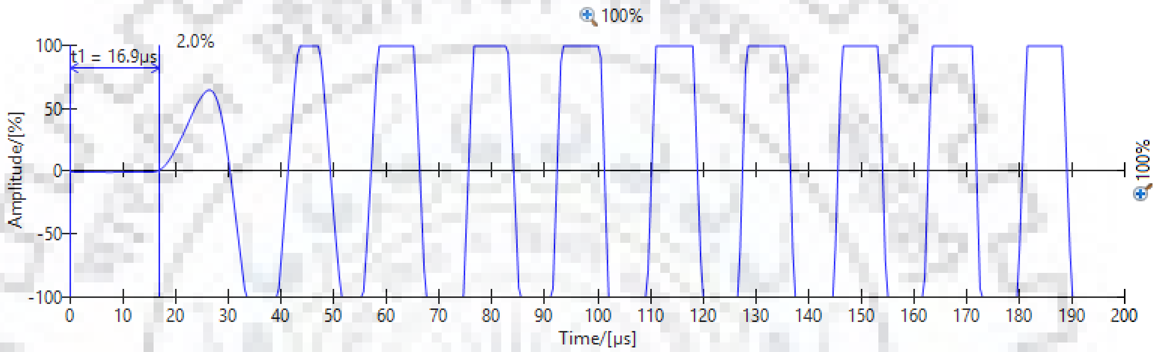

Figure 18 Vp waveform of Limestone

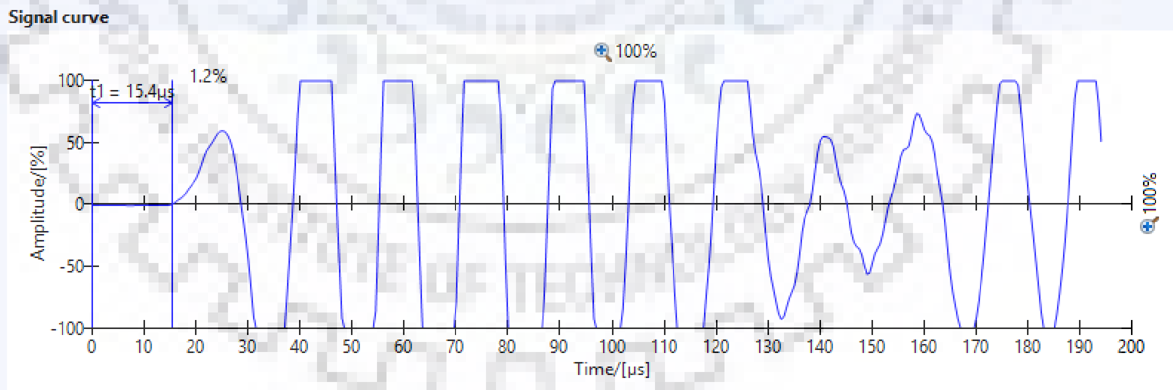

Figure 19 Vp waveform of Sandstone

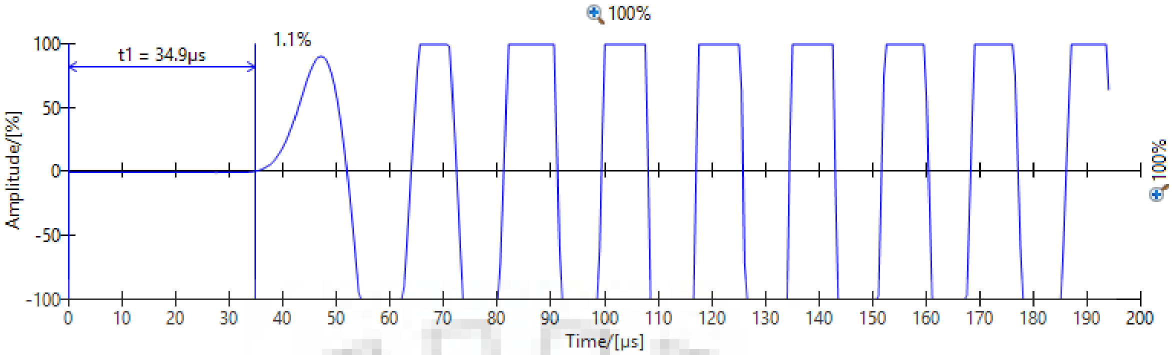

Figure 20 Vp waveform of Granite Gneiss

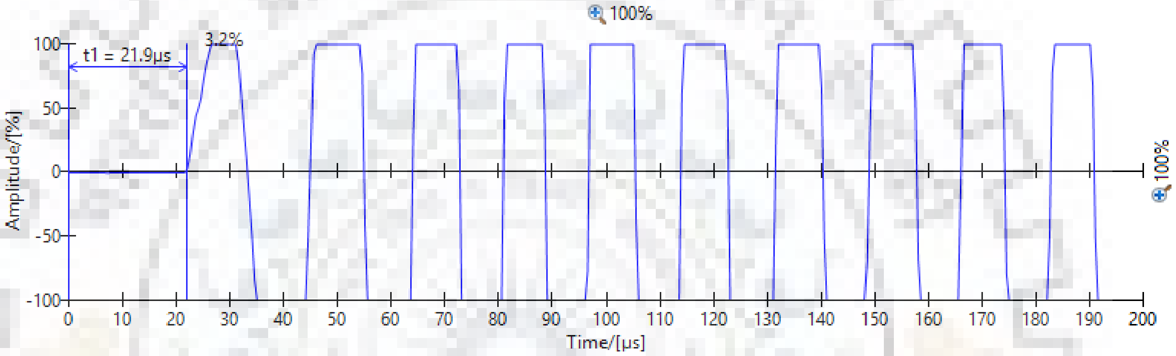

Figure 21 Vp waveform of Dolomite

accurate reading. Pulse of frequency 54 KHz, amplitude of 50V and pulse length is 2µs for P wave is transmitted from the transducer. Gel is used for smooth contact between the transmitter, rock specimen and receiver. Pulse is travelling in all direction and attenuation of wave occurs during travelling. In pundit software length (path length) of the specimen is to be inserted to calculate the travel time by using simple distance time velocity relationship.

$$V = L/t$$

Where,

V= pulse velocity

L= length of the rock(specimen)

t= time taken

Porosity calculation through ultrasonic measurements include P wave velocity in fluid, rock matrix, and bulk rock sample. Wave velocity of rock matrix and fluid will be taken from literature. These values are related through Wyllie equation.

Table 7 Vp velocity of rock matrix (Carmichael,R.S ,1982)

sample	Vp of matrix(m/s)
Meta-Basalt	7620.0
Limestone	6400.8
Sandstone	5791.2
Granite	5486.4
Dolomite	7010.4
Water	335.28

In term of velocity, v

$$\frac{1}{v} = \frac{\emptyset}{vf} + \frac{1 - \emptyset}{vma}$$

Where,

v=P wave velocity of bulk sample

vf= P wave velocity of fluid present in the sample

vma=P wave velocity of rock matrix

4.2.2. Elastic Properties of Rocks

Ultrasonic method uses transition time of elastic pulse take to travel between the two end points of rock matrix. Both compressional wave velocity (Vp) and shear wave velocity (Vs) are used for calculating elastic properties.

To measure the shear wave velocity and travel time, separate transducer and receiver are used. The gel used between the rock specimen and transducer is different from P wave measuremnets gel. Pulse length of 2.0 μs, high frequency of 250 kHz and pulse amplitude of 500 V is used to measure shear wave velocity and travel time. Shear wave is not used to calculate the porosity but has implication in calculating the elastic modulus.

In Figure 22, first arrival is p wave which is measured from S wave transducer and receiver so S wave first arrival will take approximately double time of P wave.

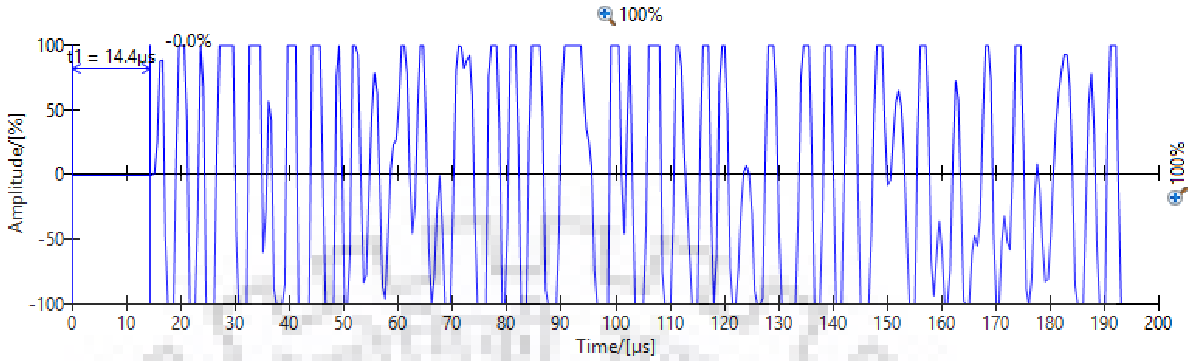

Figure 22 shear waveform of Meta-Basalt

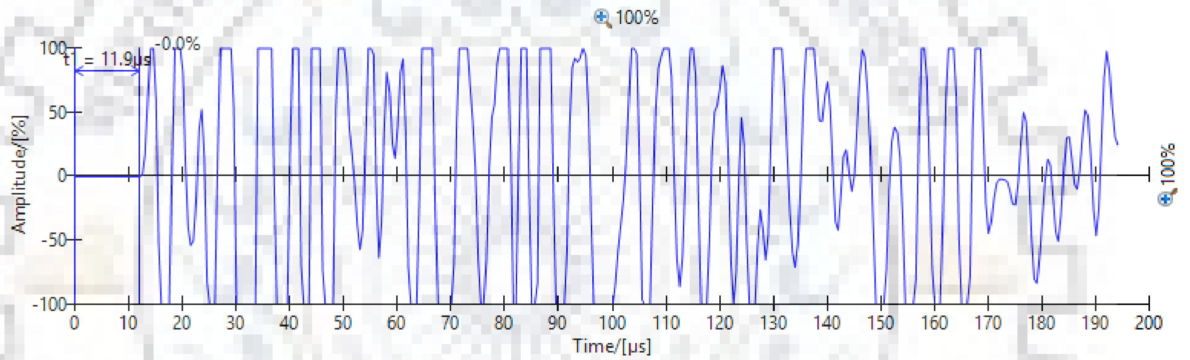

Figure 23 Shear waveform of Limestone

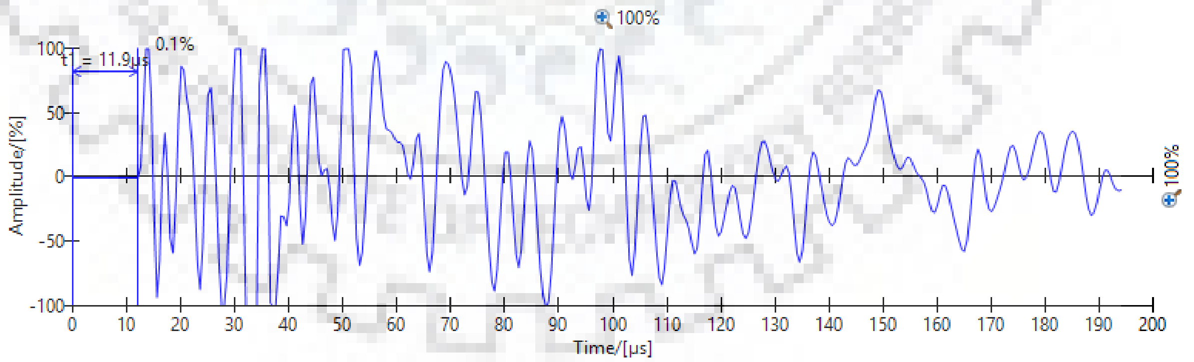

Figure 24 shear waveform of sandstone

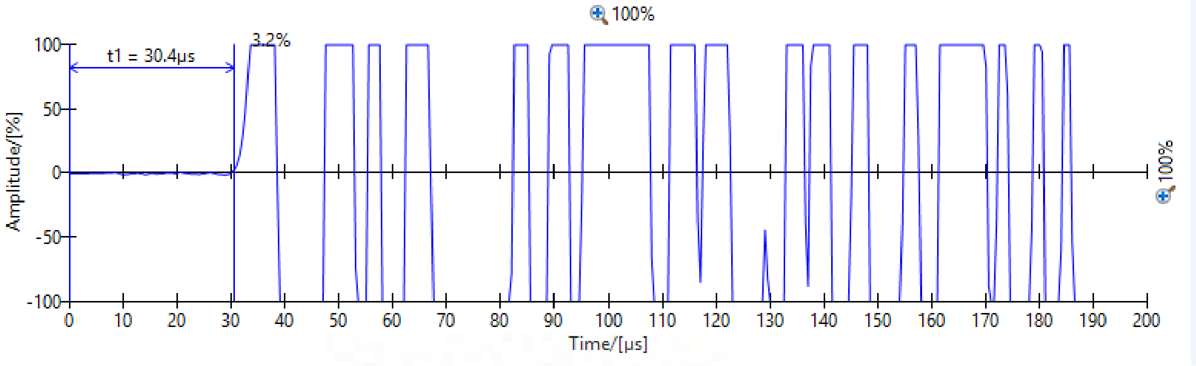

Figure 25 shear waveform of Granite Gneiss

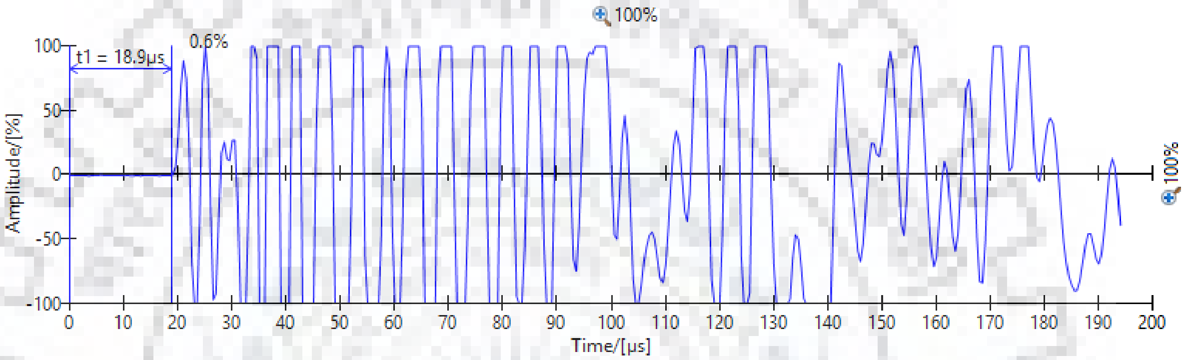

Figure 26 shear waveform of Dolomite

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

$$Vp = \sqrt{\frac{K+(\frac{4}{3})G}{\rho}}$$

$$E = \frac{\rho Vs_2(3Vp^2 - 4Vs^2)}{Vp^2 - Vs^2}$$

Where, Vp= Compressional wave velocity

Vs= Shear wave velocity

G= shear modulus

K= Bulk modulus

E= Youngs Modulus

ρ= Density of rock

4.3. Scanning Electron Microscope (SEM) Experiment (Non-Destructive)

4.2.1. Image Acquiring

A scanning electron microscope (SEM) is very vital instrument to produce micro and nano scale images. The SEM is used to obtain the detailed information of the near surface or surface region of the rock. A SEM can be split into three major parts-(i) vacuum system (ii) an electron optical column and (iii) electronics.

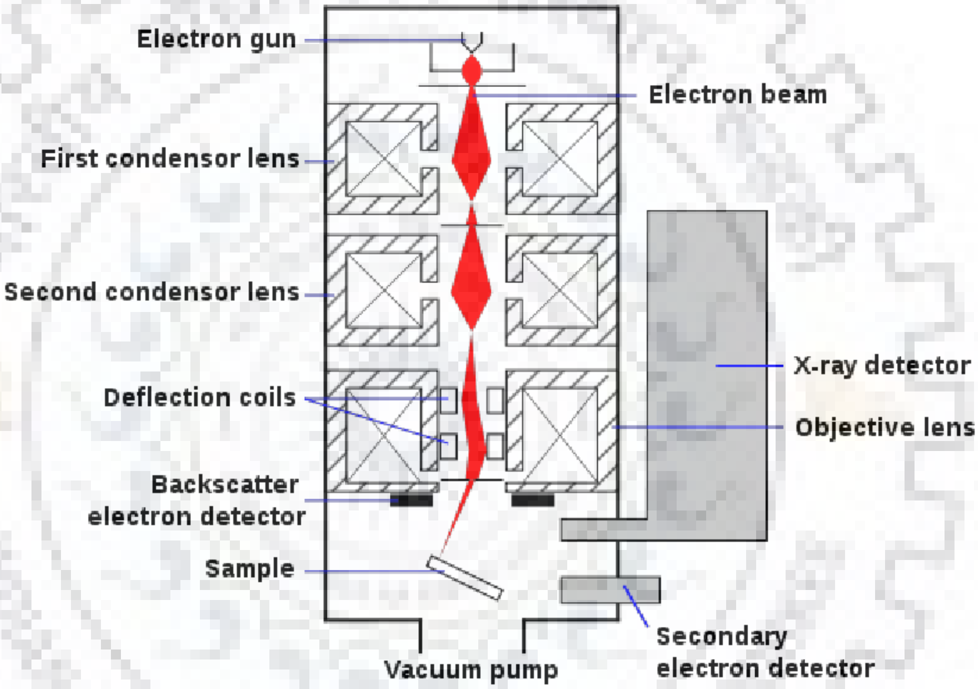

Figure 27 Schematic Diagram of Scanning Electron Microscope (SEM) (Abudayyeh,2012)

Figure 28 Scanning Electron Microscope (SEM)

The SEM images acquired by focusing a beam of high energy electrons across each rectangular target on the sample as small as 1nm. The interactions between the electron beam and the sample surface produces secondary and back-scattered electrons, which are used to image the structure of the sample. SEM images are recorded and taken by SmartSEM (Services) software (Figure.29)

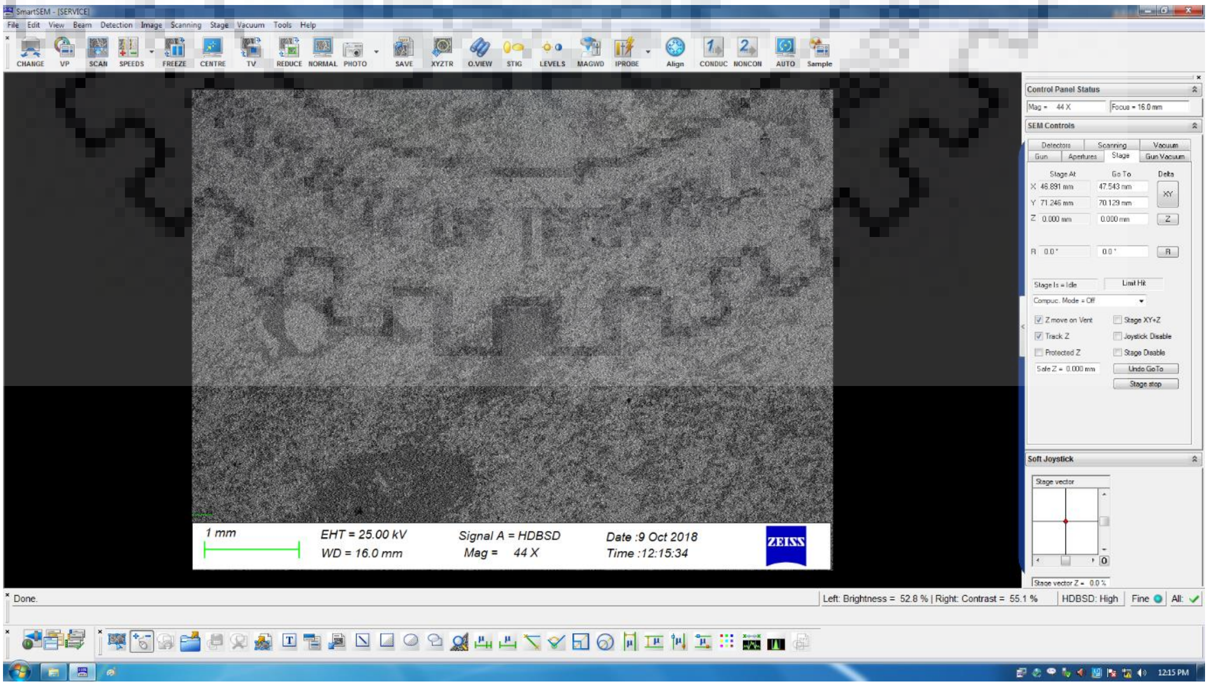

Figure 29 SmartSEM software interface

Magnification

The magnification of image is defined as ratio of the size of the displayed image and the area scanned size.

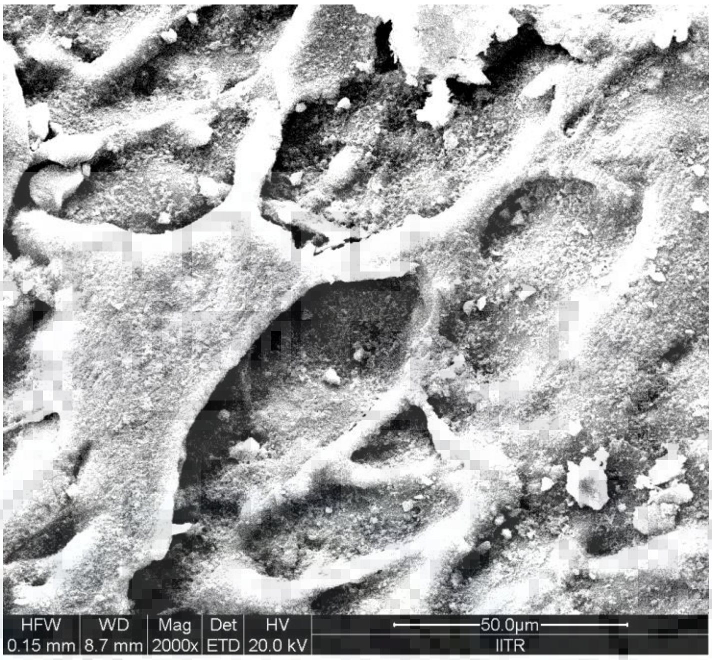

Figure 30 SEM image of Granite Gneiss

$$\text{Magnification}=\frac{size\ of\ displayed\ image}{size\ of\ the\ area\ scanned}$$

Magnification of the images can be change by changing the size of area scanned. Apparently, magnification increases by reducing the size of scanned area. The rock for SEM experiment has to be dry, vacuum compatible, dry and electrically conductive. Quality of SEM images depend on distance of the rock specimen and orientation from the final lens. The specimen movement or coordinate X, Y, Z can be controlled by computer system. These interactions also produce X-rays, which when paired with an energy-dispersive X-ray spectroscopy (EDS) sensor, can be used to determine the elements present in a sample. The elemental composition is useful in identifying the minerals present in the sample.

Due to constructive method and different scales of measurement, its use in petroleum industries is increasing.

4.2.1. Image Processing

SEM images are taken for different rock types at different resolution or magnification. The pore space is clearly distinguishable from the grains. The position of the image was randomly chosen from the SEM chunks for different rocks in order to take homogenous and heterogeneous picture. In each SEM sample, multiple images at different magnification were taken. Dark (black) colour represent pore spaces and others colour i.e grey colour are rock matrix. The images consist of 1024×768 pixels with scale bars present on the image. From the greyscale SEM images, binary images are produced using fiji (advance of ImageJ software) software. Greyscale images are converted into a binary image by adjusting the threshold limit on pores and grains, and thus binary image (pores and grain are segmented) formed.

ImageJ/ Fiji software

ImageJ can be analyze, edit , display and process upto 32 bit color, gray scale image in almost all formats including TIFF image. It can also calculate area and pixel values statistics. It can also measure grain size. It also support many in built thresholding algorithm. It also helps in image processing function by applying arithmetical and logical operations, brightness adjustment and contrast adjustment and sharpening of images.

Pixel Values

Pixel values are numbers in digital images which is not subjective to colour but photon counts. These number represent the type of signal entered the equipment and its interaction with environment and after interaction information is processed in the digital format. Tagged Image File Format (TIFF) image is produced through SEM experiment because it is non lossy formats. TIFF images preserve any calibration applied to images without compressing the file.

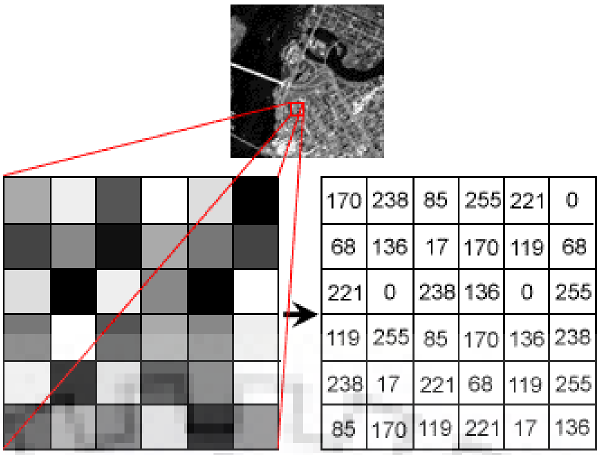

Figure 31 Pixel values in a square grid (ai.Stanford.ed)

4.2.1.2. Binary image

In order to differentiate pore with rock matrix, grayscale image (0 to 255 grey scale value) obtained from SEM experiment need to be converted to binary image (0 and 1). A digital image that has only two possible pixel values for each pixel is called binary image. Typically, black (for pores) and white (for rock matrix) are used in binary image although any colour may be used. Edge of the rock matrix pores are very vague. In order to clearly differentiate rock matrix and pore in SEM grayscale images, a threshold pixel value is needed to divide the image into two groups white and black. Separating objects and image features by their cut off pixel value called threshold value. Common image binarization method are Isodata, otsu method, max entropy, mean method etc. These auto binarization method did not give accurate results for images used in this thesis, so manual thresholding is required after applying one of these method.

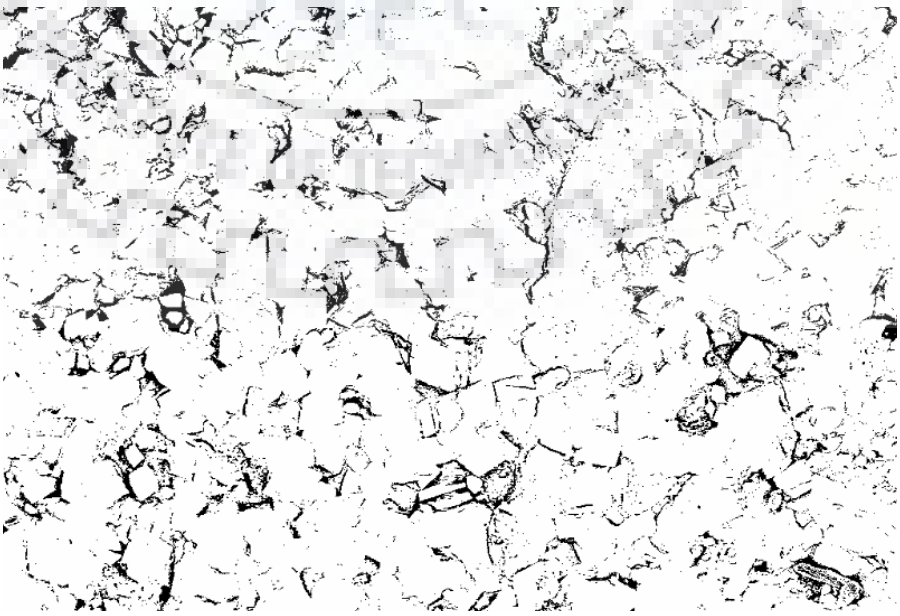

Figure 32 Binary Image

4.2.2. Surface Porosity Calculation Procedure

Only pores with size above the SEM imaging resolution is taken into account. Surface porosity of the two ends of the core samples of different rocks are calculated. Due to the heterogenous pore distribution in rocks, surface porosity calculated is different for same rock. In order to find surface porosity distribution within the same rock, homogenous and heterogeneous section of the rock surface is analysed. To calculate the approximate bulk porosity of rock from surface porosity, average of homogenous and heterogeneous section's surface porosity of the two end is interpolated.

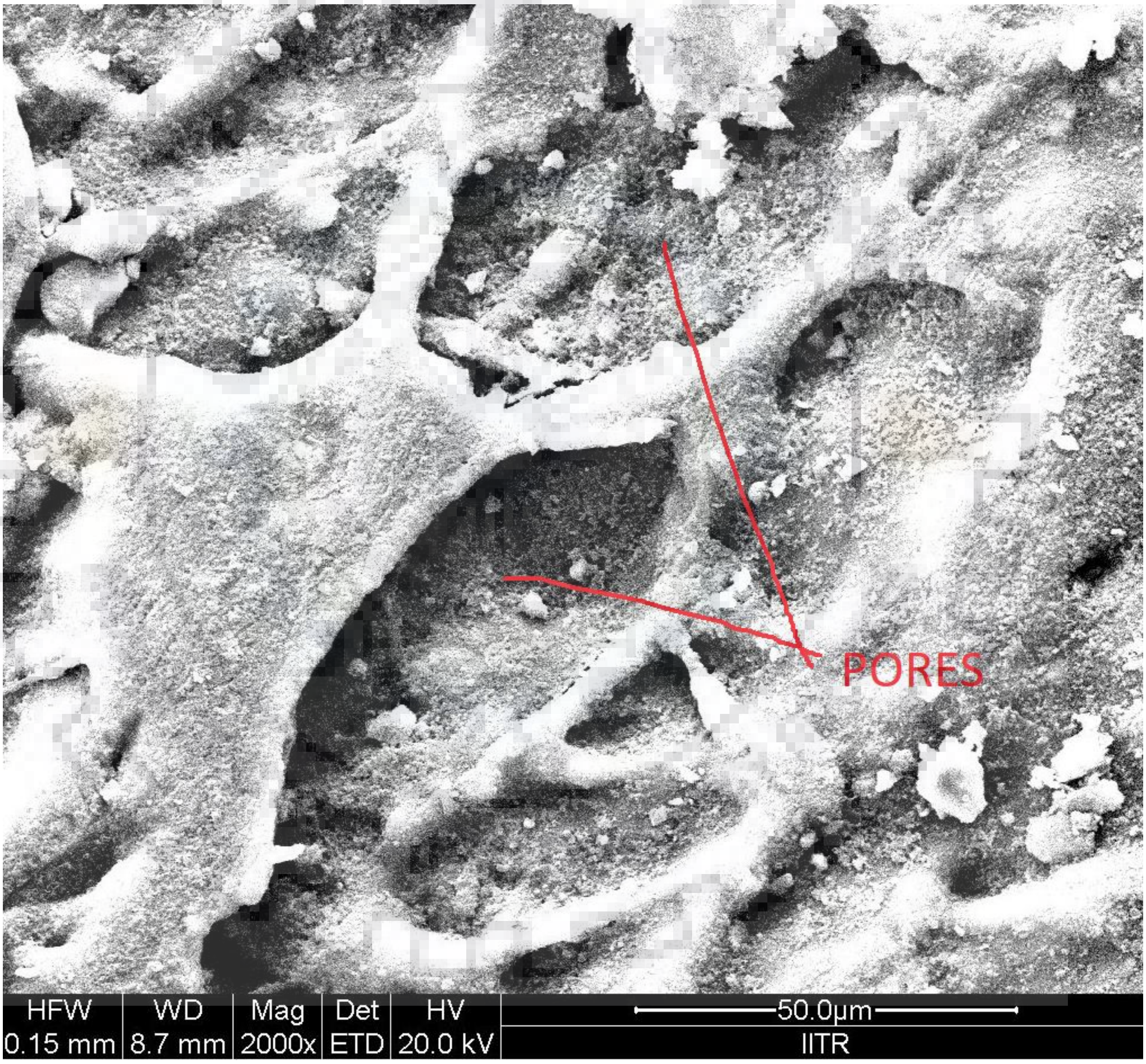

Figure 33 SEM image of Granite Gneiss indicating Pores

Step 1: Open the image in Fiji software.

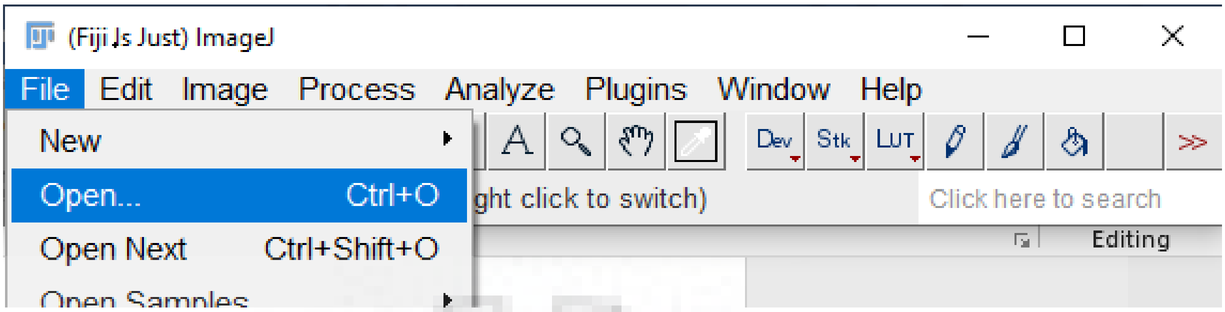

Figure 34 Fiji software interface

As the image should only consist of the surface of interest so scale bar and other bars is to remove, so duplicate image of the area of interest is produced for analysis.

Step 2: To make duplicate image, right click on the image and click on duplicate. New image of the selected area will appear.

Step 3: Select the type of image from the menu bar

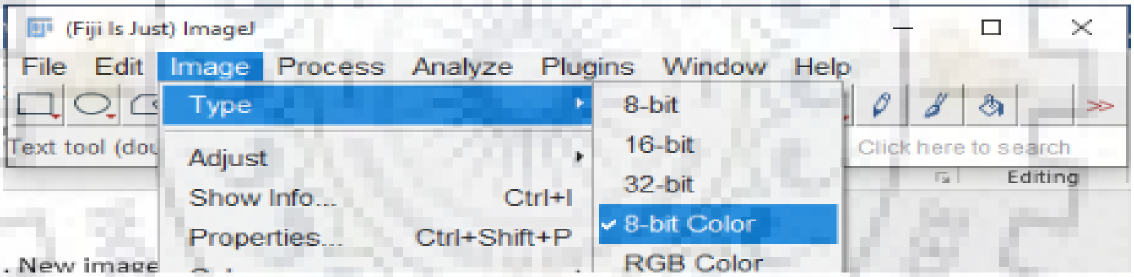

Figure 35 Selecting type of image in fiji software

Step 4: For thresholding go to 'Image' option in the menu bar -> Adjust->Threshold

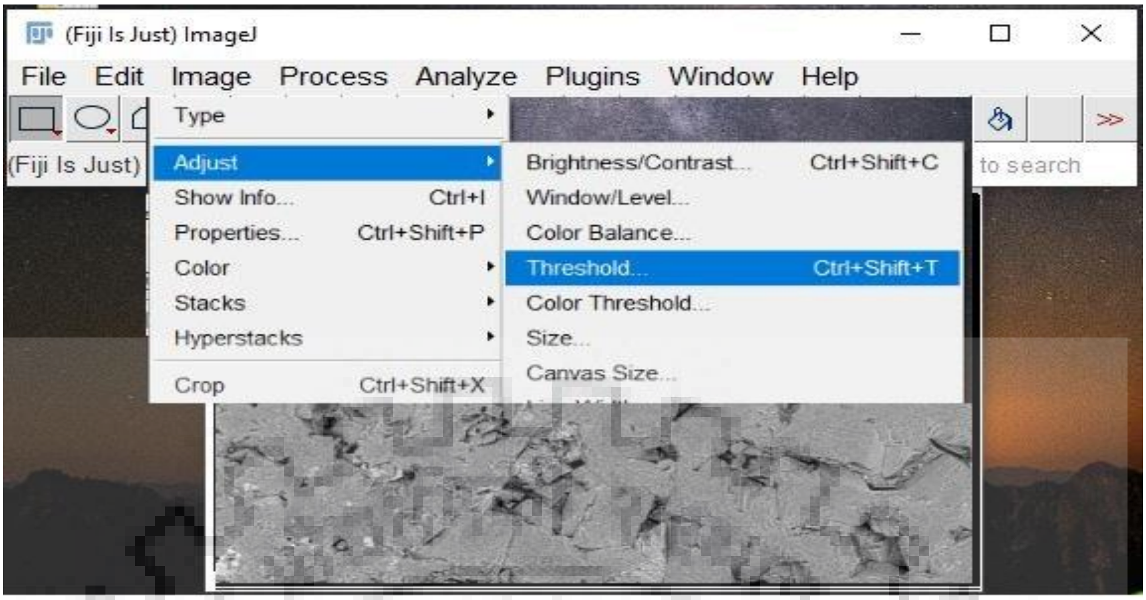

Figure 36 Figure showing thresholding option in Fiji software

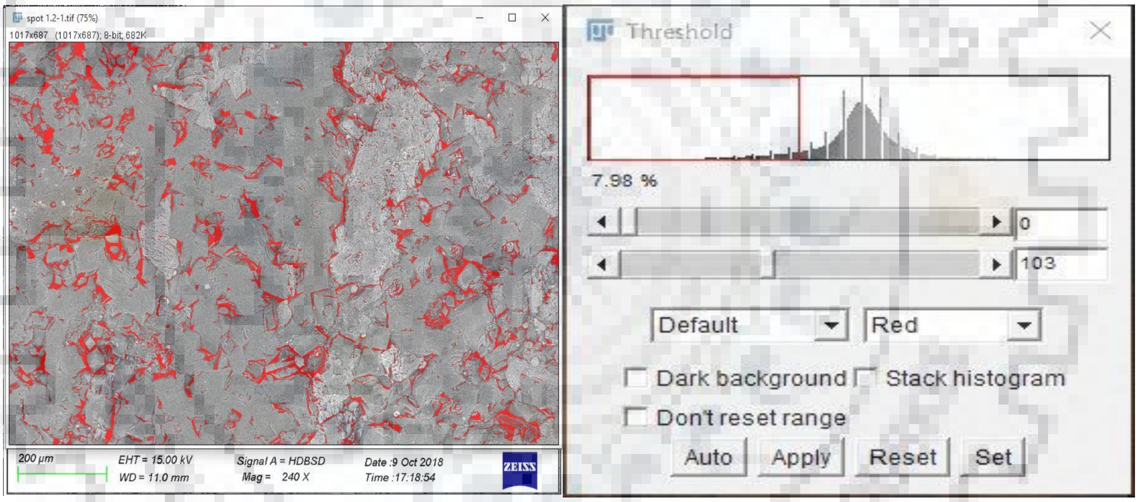

Figure 37 Threshold limit set on dolomite SEM image

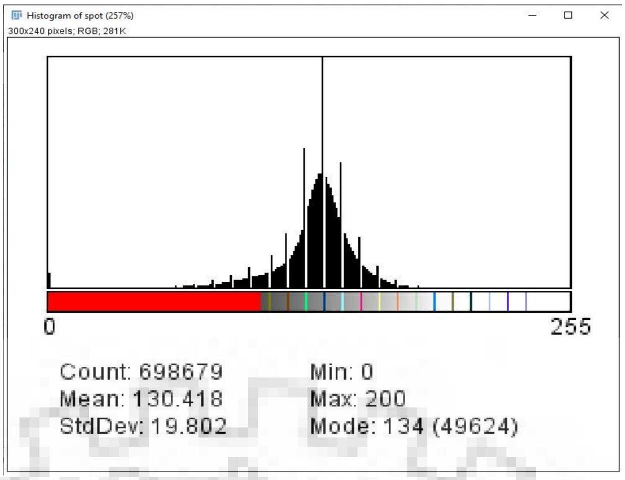

Figure 38 Histogram of grey values of dolomite

Different thresholding algorithm is applied to the image and none of the method able to differentiate the pores and matrix properly so I choose default algorithm which is IsoData (Iterative selection method) thresholding algorithm for thresholding and also did manual thresholding based on the visualisation and histogram of frequency and grey scale value to get better results in differentiating pores and matrix.

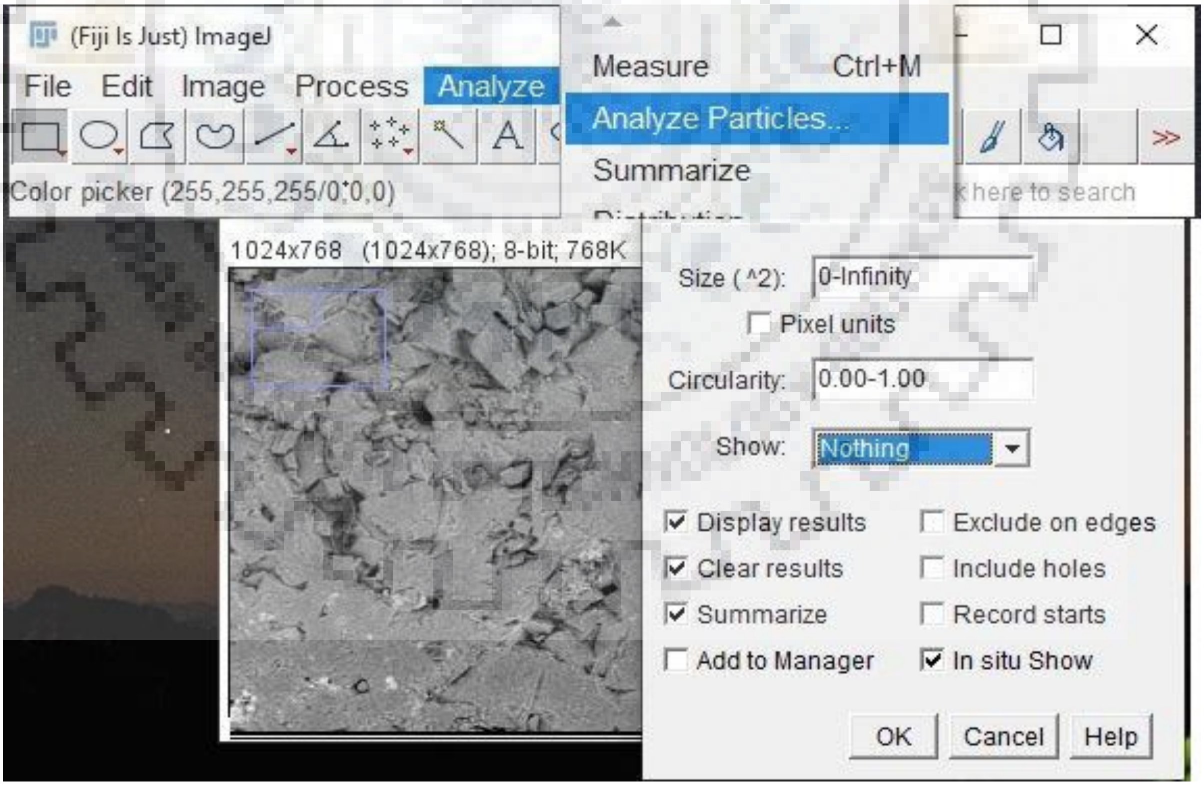

Figure 39 setting Limit to analyse particle in Fiji software

Step 5: Before click on ‘Apply’ button create a histogram by going on ‘Analyze’ and create histogram.

After applying the thresholding limit grey scale image get converted into binary image

Step 5: Go to ‘Analyze’ option on the menu bar -> anlalyze particles. After clicking OK, it will automatically calculate the percentage area of total pores and also of individual pores based on the grey scale value of pores.

IsoData Algorithm

This is based on the iteration algorithm. In this algorithm image is divided into background and object by taking some initial threshold value. Average of the pixels above the threshold and below the threshold are computed. The threshold is increased by iterating until the threshold is larger than composite average.

Threshold= (Average objects+ average background) /2

4.3.4. SEM Images and Its Binary Images

4.3.4.1. Sandstone

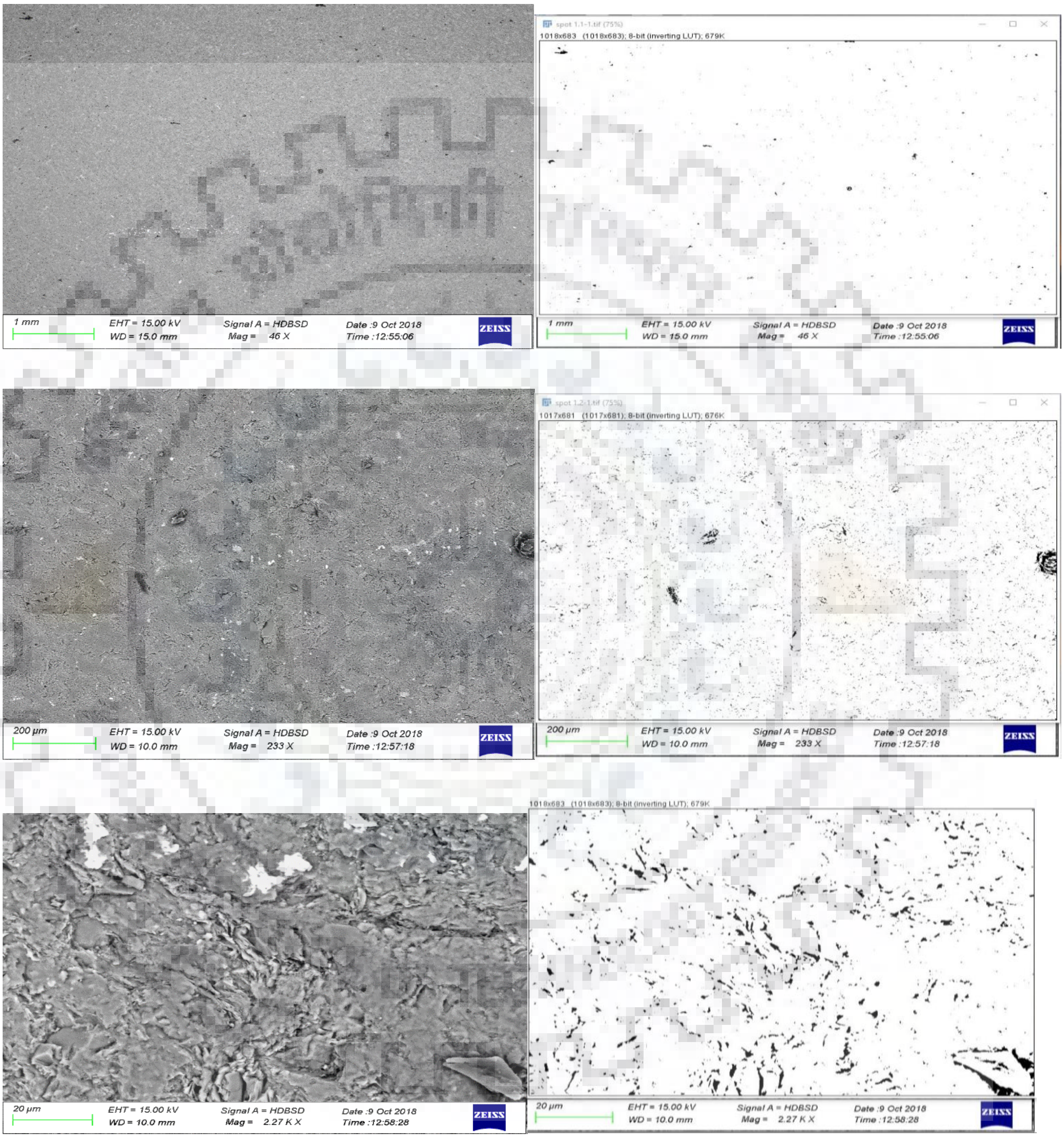

Figure 40 SEM and Binary image of Homogeneous Sandstone surface

4.3.4.2. Dolomite

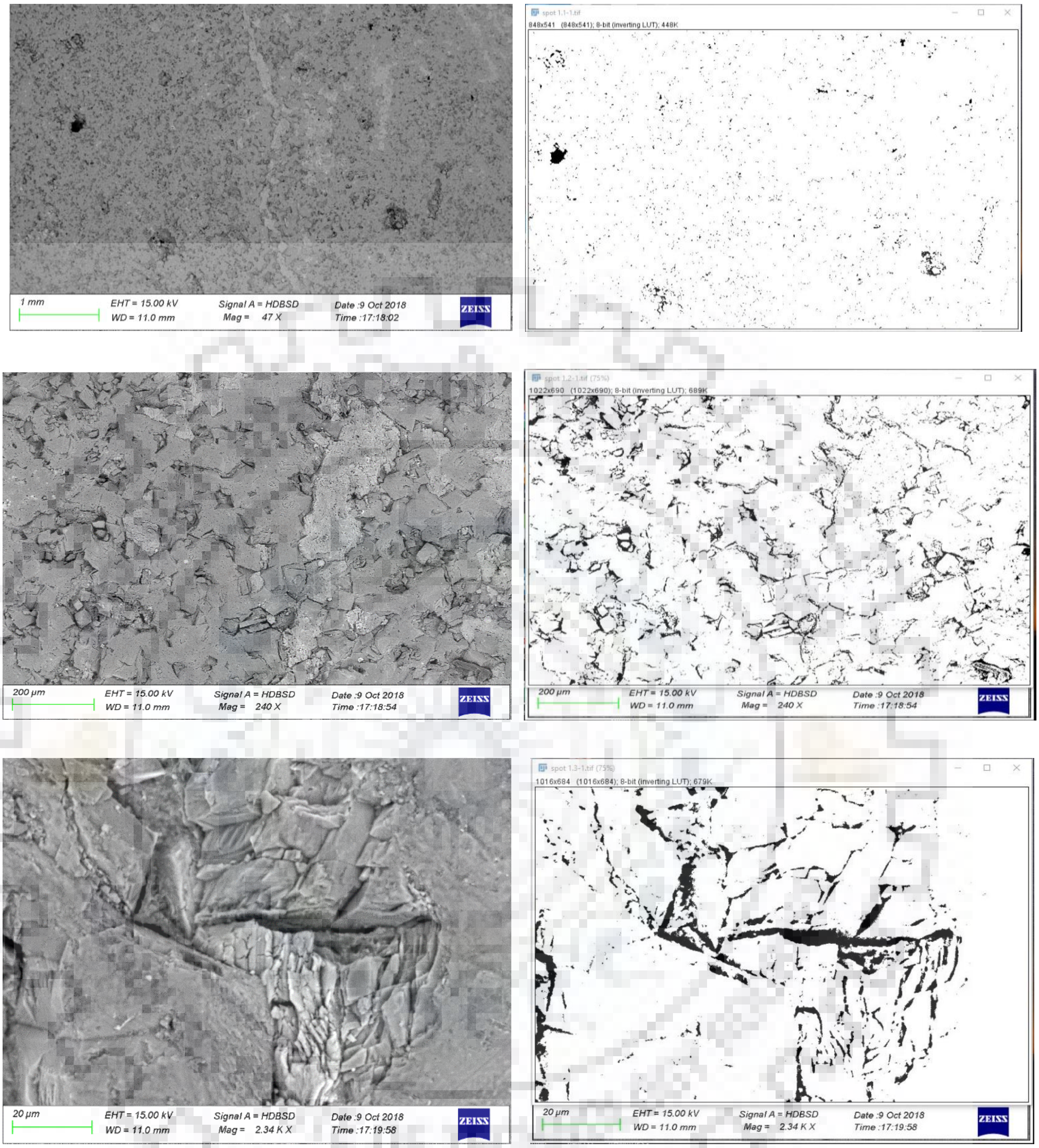

Figure 41 SEM and Binary image of Homogeneous Dolomite surface

4.3.4.3. Meta- Basalt

Homogeneous Meta- basalt

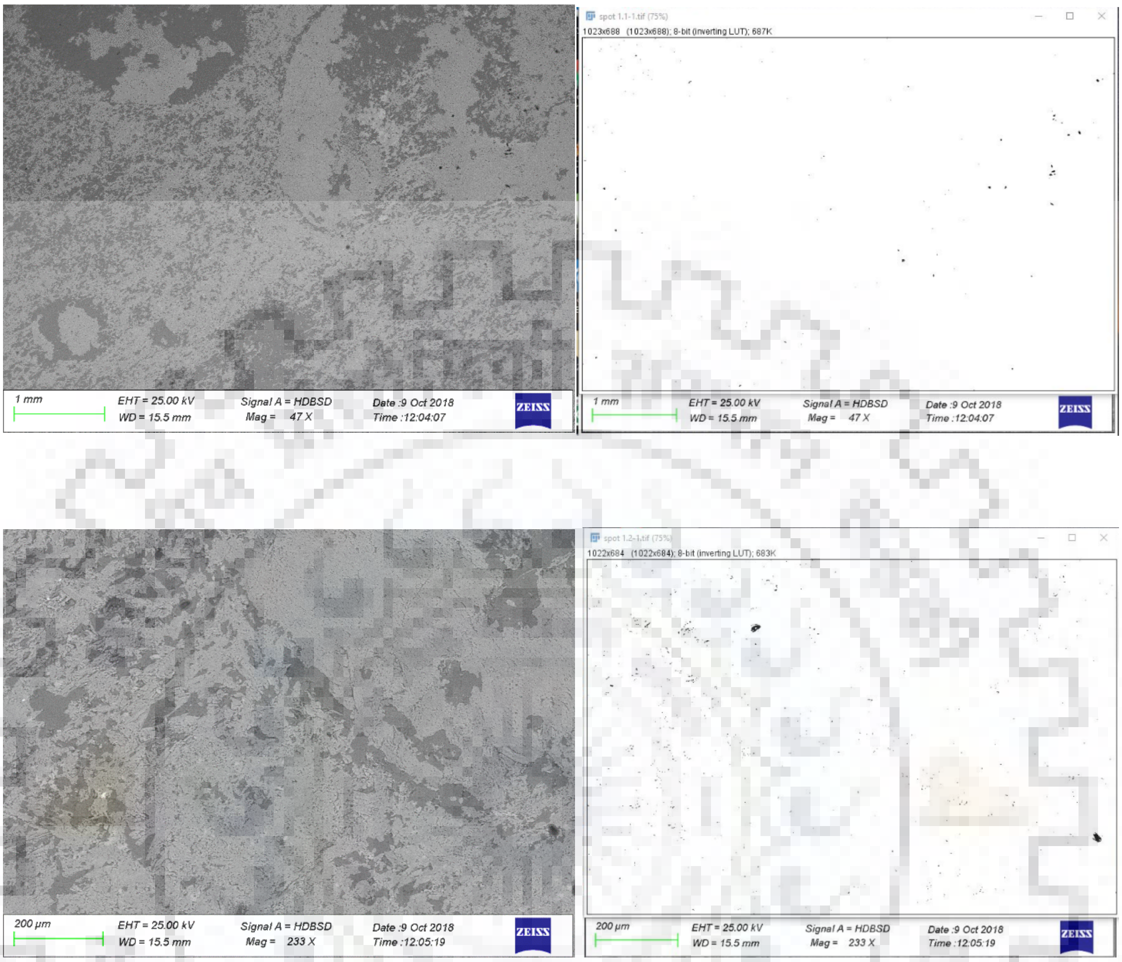

Figure 42 SEM and Binary image of homogeneous Meta-basalt

Heterogeneous Meta- basalt

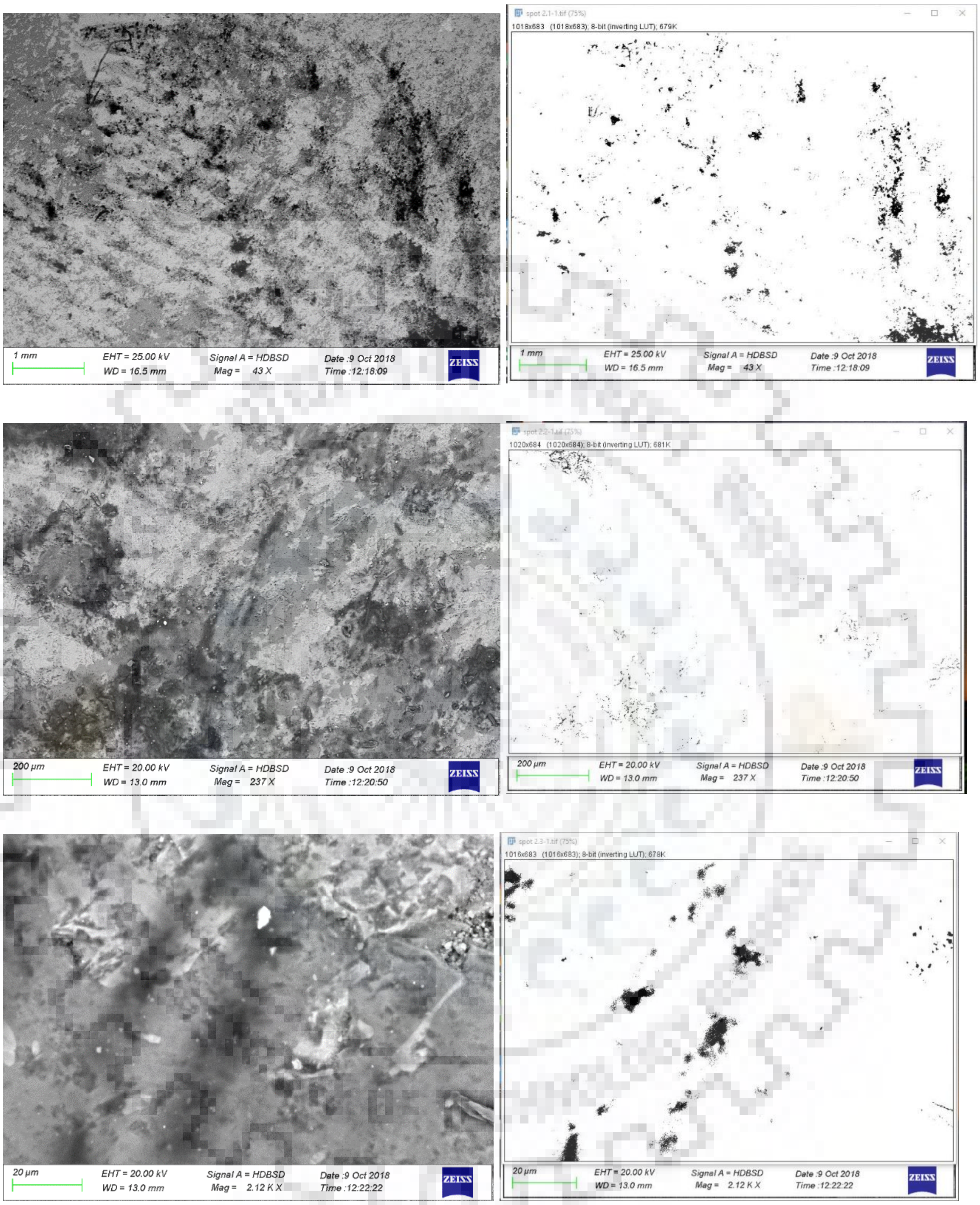

Figure 43 SEM image of heterogeneous meta-basalt at different resolution

4.3.4.4. Limestone

Homogeneous Limestone

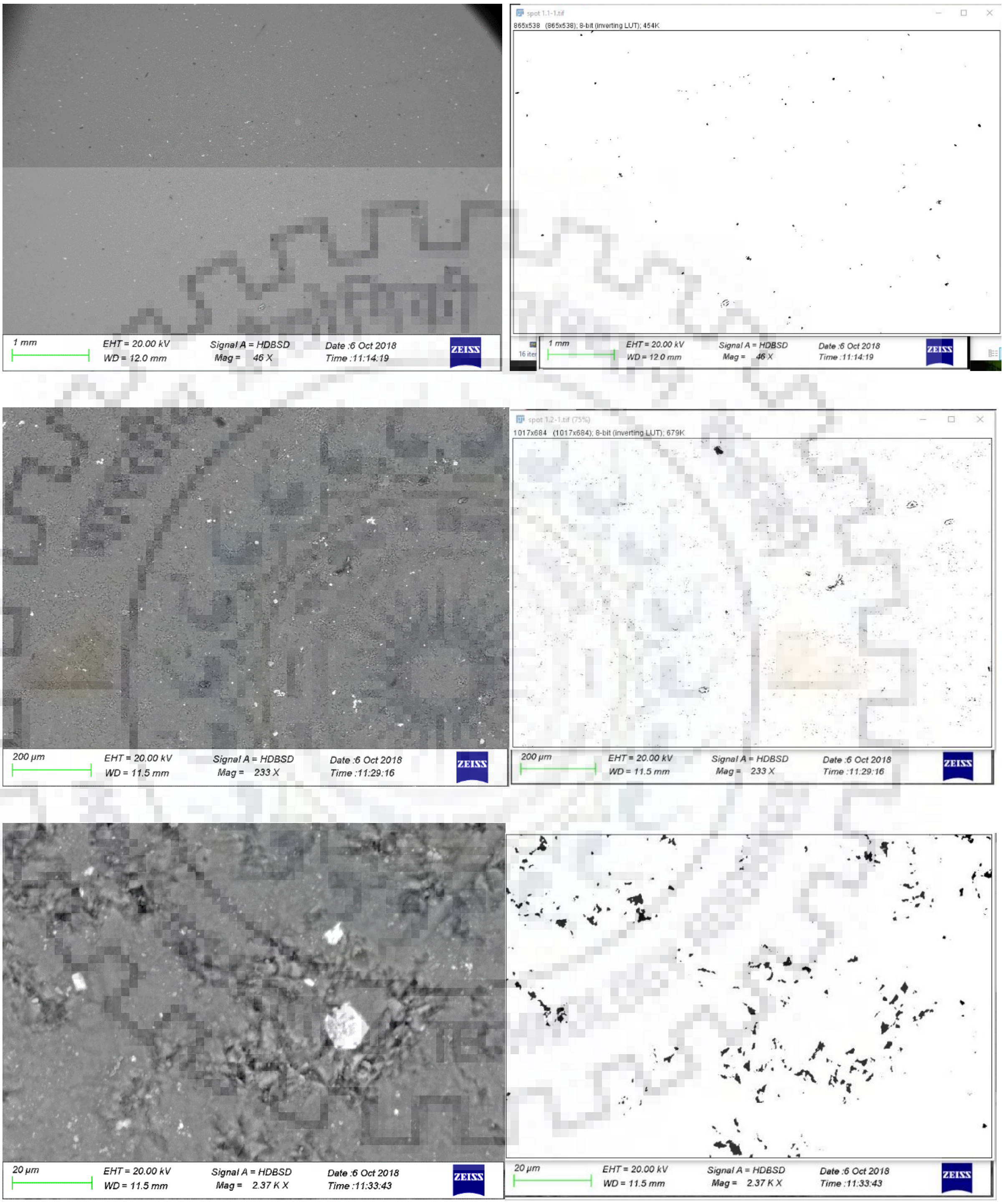

Figure 44 SEM of Homogeneous Limestone surface at different resolution

4.3.4.5 Granite Gneiss

Homogeneous Granite Gneiss

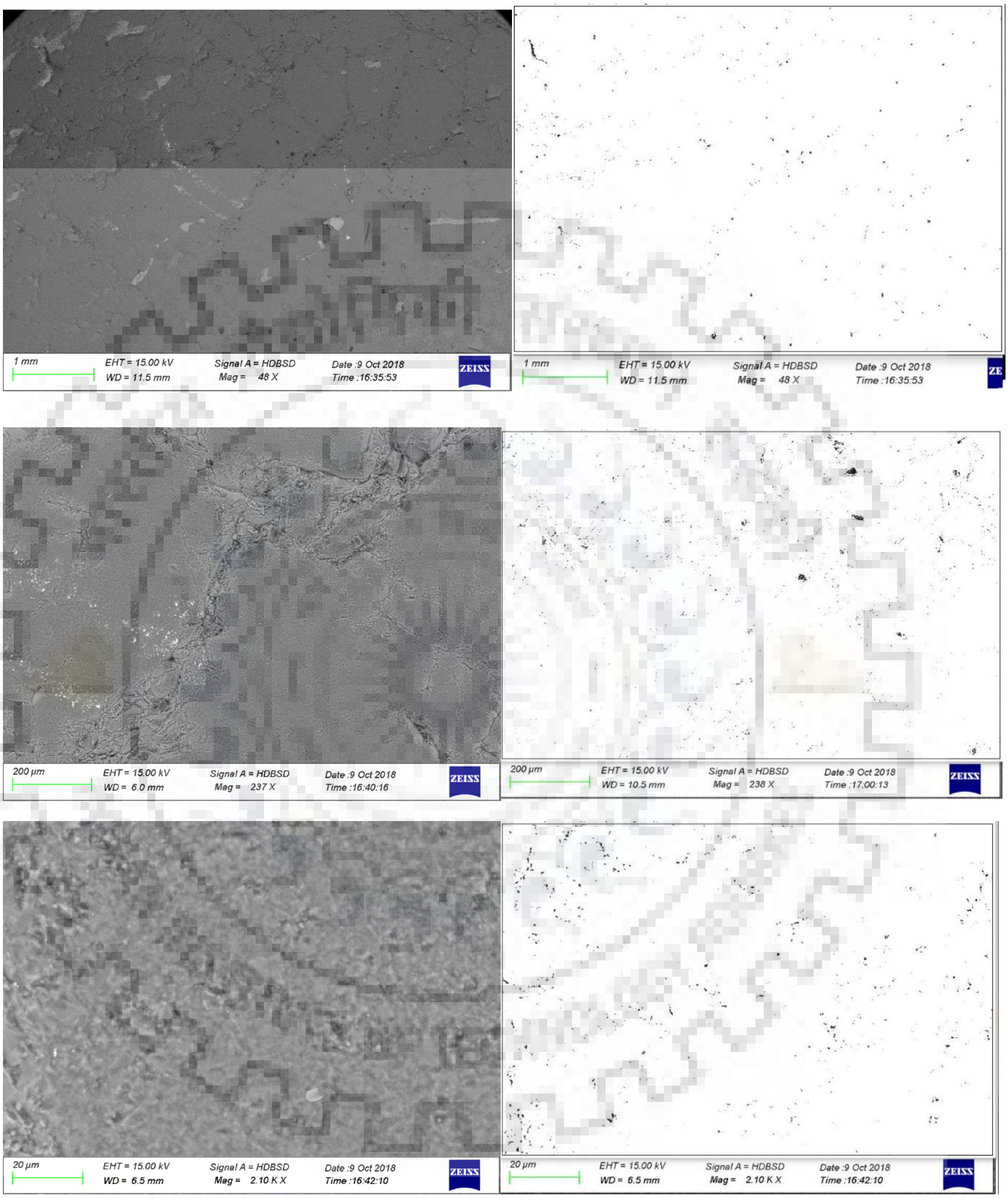

Figure 45 SEM images of homogeneous Granite Gneiss at different resolution

4.3.5. Calculation of Bulk porosity form surface porosity

3D porosity or bulk porosity can be calculated from 2D porosity by taking average of ends or edges of core surface porosity

$$\text{3D porosity} = \left(\sum_{k=0}^{n} \text{2D porosity}\right)/\text{n}$$

This is interpolation technique.

Chapter 5 : Results and Discussion

These are the results of the porosity calculation from SEM images at a scale of 20µm (1st order), 200µm (2nd order), 1000µm (3rd order), Scale of 10000µm (4th order) represents the core porosity obtained from the conventional method (Water immersion method) and scale of 100000µm (5th order) represent the porosity calculated from the ultrasonic measurements. Due to limited availability of rock sample and advancement if imaging technique we are able to obtained the SEM image of homogeneous and heterogeneous surface. The conforming results for field porosity values were obtained using water immersion porosity method on all the five samples. Relation of modulus of elasticity with porosity is also seen in all five types of rocks.

5.1. 2D Porosity from SEM

5.1.1. Homogeneous Surface

Table 8 Surface porosity of homogenous rock surface of top and bottom ends

		Top end			Bottom end		
Sample no	**Rock name**	**1000µm Mag=47X**	**200µm Mag=240X**	**20µm Mag=2.34 KX**	**1000µm**	**200µm**	**20µm**
1	Meta-Basalt	0.05	0.21	0.17	0.18	0.26	1.42
2	Limestone	0.11	0.60	1.62	0.11	0.46	2.23
3	Sandstone	0.21	0.23	2.19	0.22	2.08	4.27
4	Granite Gneiss	0.57	0.74	2.70	0.25	0.58	0.57
5	Dolomite	0.45	10.55	9.72	1.31	7.97	8.44

5.1.2. Heterogenous surface

Table 9 Surface porosity of heterogenous rock surface of top and bottom ends

		Top end			Bottom end		
Sample no	**Rock name**	**1000µm Mag=47X**	**200µm Mag=240X**	**20µm Mag=2.34 KX**	**1000µm**	**200µm**	**20µm**
1	Meta-Basalt	0.46	3.45	9.48	2.76	0.47	2.26
2	Limestone	0.78	2.78	4.57	0.21	0.68	3.91
3	Sandstone	0.26	2.12	4.91	0.37	2.42	4.65
4	Granite Gneiss	0.84	1.26	2.32	0.55	2.24	0.73
5	Dolomite	1.45	10.86	12.85	1.07	8.96	12.84

5.2. 3D Porosity From SEM

5.2.1. 3D porosity of Homogeneous images

Table 10 3D porosity of Homogenous rock at different scale

Sample no	**Rock name**	**1000µm Mag=47X**	**200µm Mag=240X**	**20µm Mag=2.34 KX**
1	Meta-Basalt	0.12	0.24	0.80
2	Limestone	0.11	0.53	1.93
3	Sandstone	0.24	1.16	3.23
4	Granite Gneiss	0.66	0.66	1.64
5	Dolomite	0.88	9.26	9.08

5.2.3. 3D Porosity of Heterogenous images

Table 11 3D porosity of Heterogenous rock at calculate from SEM images

Sample no	**Rock name**	**1000µm Mag=47X**	**200µm Mag=240X**	**20µm Mag=2.34 KX**
1	Meta-Basalt	1.61	1.96	5.87
2	Limestone	0.50	1.73	4.24
3	Sandstone	0.29	2.27	4.78
4	Granite Gneiss	0.70	1.75	1.53
5	Dolomite	1.26	9.91	12.85

5.3. Bulk Porosity at 5 Different Scale

5.3.1. Porosity of Homogeneous rock at 5 Different scales

From the results of porosity displayed in Table 12 and Figure 46, it can be seen that the porosity distribution has a large variation in dolomite at a 3rd order of scale (1mm) and is very much dependent on scale of observation while granite gneiss also exceptionally large porosity on scale of 5th order (10 cm). Sandstone, Limestone and Meta-Basalt porosity does not vary much with scale of observation.

Table 12 Porosity of homogeneous rocks at 5 different scale

Sample no	**Rock**	**20µm (1st order)**	**200µm (2nd order)**	**1000µm (3rd order)**	**10000µm (4th order)**	**100000µm (5th order)**
1	Meta-Basalt	0.80	0.24	0.12	0.63	1.54
2	Limestone	1.93	0.53	0.11	2.88	1.19
3	Sandstone	3.23	1.16	0.24	1.55	2.42
4	Granite Gneiss	1.64	0.66	0.66	1.38	8.51
5	Dolomite	9.08	9.26	0.88	10.44	3.54

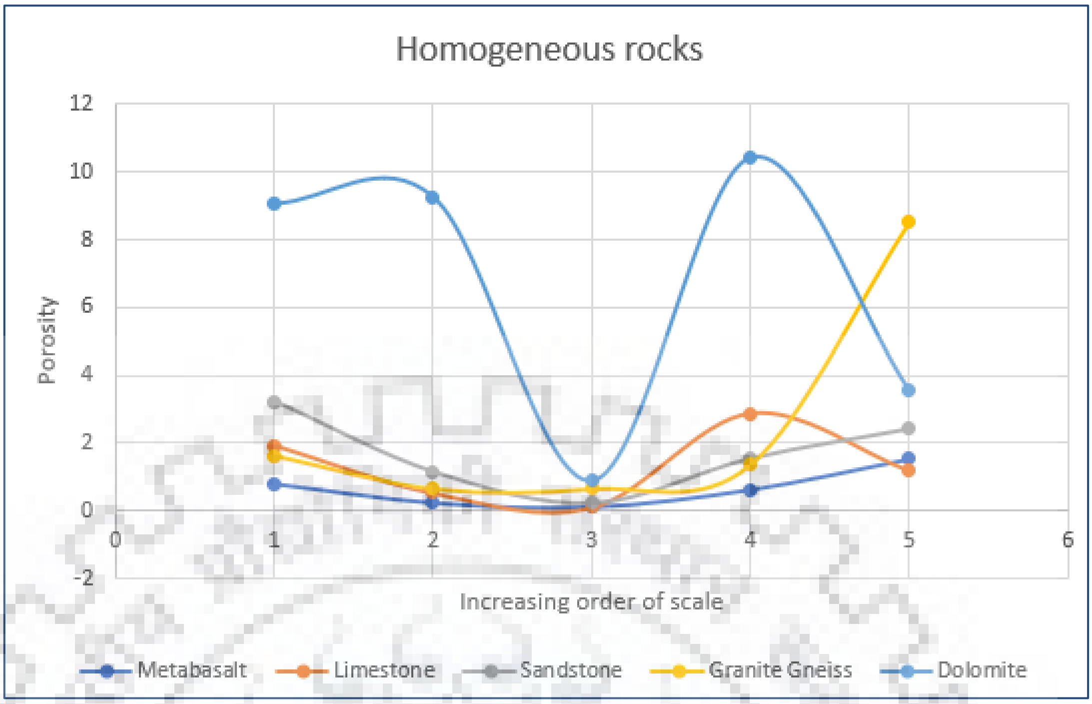

Figure 46 Porosity of different homogeneous rocks at 5 different scale

5.3.2. Porosity of Heterogenous rock at 5 different scales

From the results displayed in Table 13 and Figure 47, it can be seen that porosity distribution has large variation in dolomite in heterogeneous rocks and is much dependent on scale of observation. Dolomite has significant low porosity at scale of 1 cm while Granite Gneiss also showing exceptionally large porosity on scale of 10 cm. Sandstone, Limestone and Meta-basalt does not vary much with scale of observation.

Table 13 Porosity of heterogenous rocks at 5 different scale

Sample no	**Rock**	**20µm (1st order)**	**200µm (2nd order)**	**1000µm (3rd order)**	**10000µm (4th order)**	**100000µm (5th order)**
1	Meta-Basalt	5.87	1.96	1.61	0.63	1.54
2	Limestone	4.24	1.73	0.50	2.88	1.19
3	Sandstone	4.78	2.27	0.29	1.55	2.42
4	Granite Gneiss	1.53	1.75	0.70	1.38	8.51
5	Dolomite	12.85	9.91	1.26	10.44	3.54

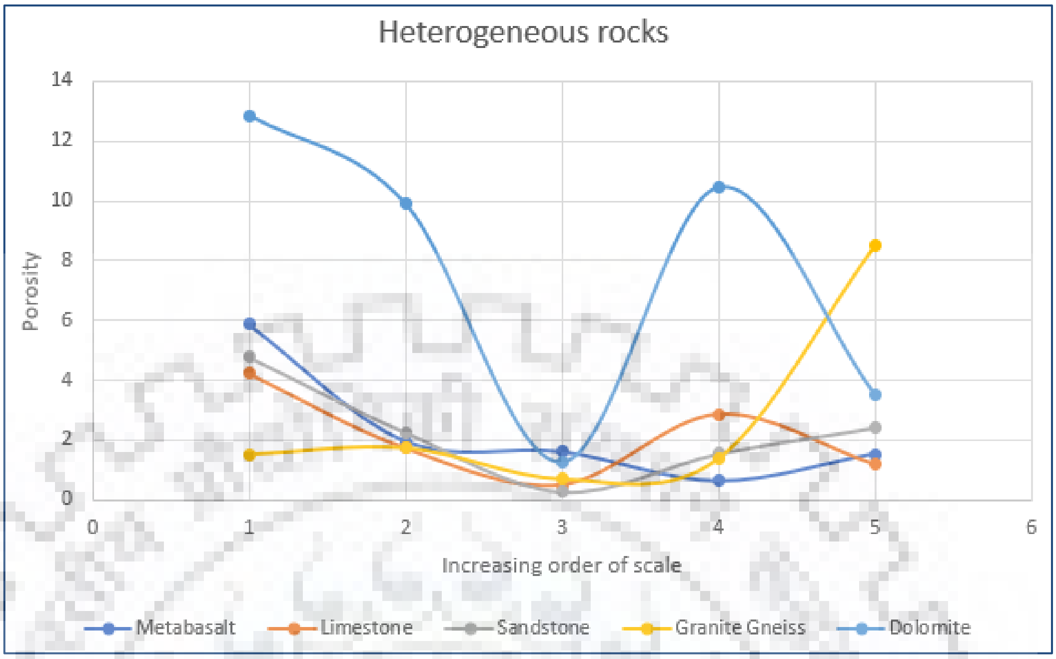

Figure 47 Porosity of different heterogeneous rocks at 5 different scale

5.3.3. Average of Homogeneous and Heterogeneous rocks

From the results displayed in Table 14 and Figure 48, the bulk porosity can be obtained by taking the average of both homogeneous and heterogenous porosity. It is found in Figure 48 that porosity is minimum at 4th order scale and almost same at 2nd order and 3rd order scale.

Table 14 Bulk Porosity of rocks at 5 different scale

Sample no	**Rock**	**20µm (1st order)**	**200µm (2nd order)**	**1000µm (3rd order)**	**10000µm (4th order)**	**100000µm (5th order)**
1	Meta-Basalt	3.335	1.1	0.865	0.63	1.54
2	Limestone	3.085	1.13	0.305	2.88	1.19
3	Sandstone	4.005	1.715	0.265	1.55	2.42
4	Granite Gneiss	1.585	1.205	0.68	1.38	8.51
5	Dolomite	10.965	9.585	1.07	10.44	3.54

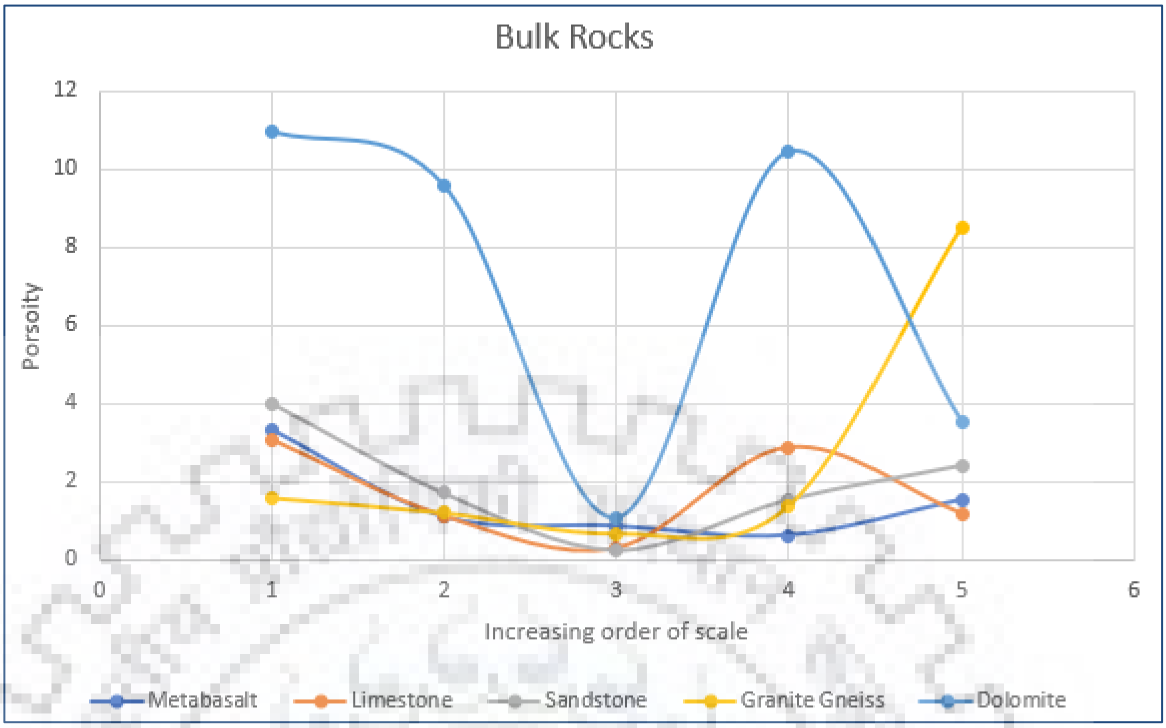

Figure 48 Porosity of different Bulk rocks at 5 different scale

5.4. Porosity variation in same type of rock based on the homogeneity

5.4.1. Meta-Basalt

From the results displayed in Figure 49, difference in porosity of homogeneous and heterogeneous surface is significantly higher at a scale of 1st order (20μm) than other scales. Difference in porosity in heterogeneous and homogeneous is more at low scale. Porosity range from 0.12% to 1.54 % for homogeneous surface and 0.63% to 5.87% for heterogenous and as bulk rock porosity range from 0.63 to 3.34%.

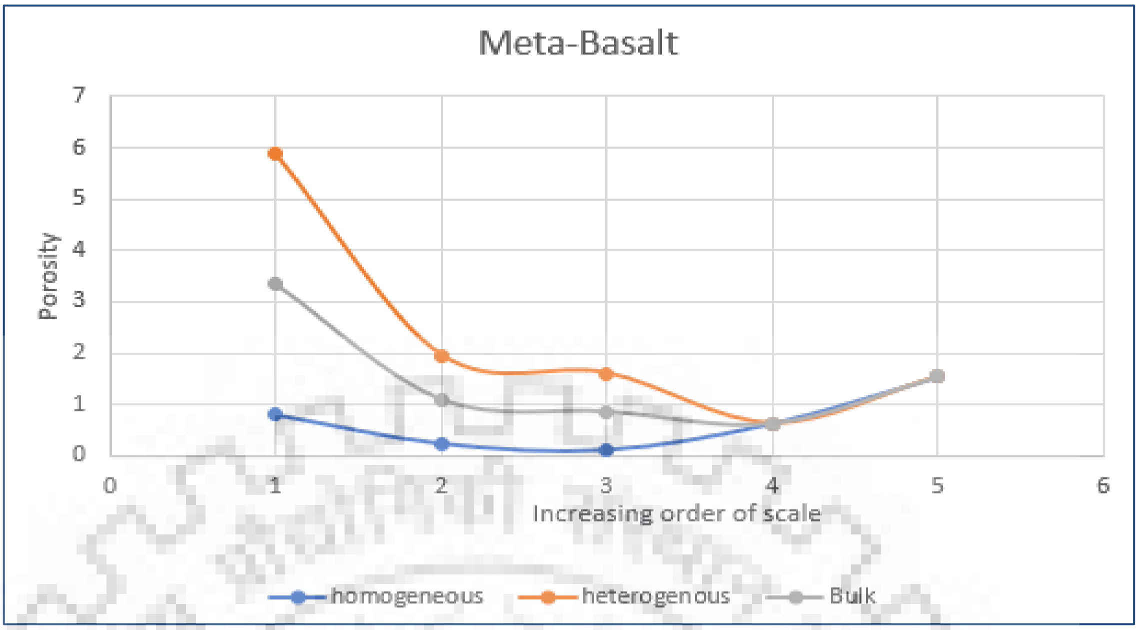

Figure 49 variation of Porosity of Meta-Basalt of homogeneous and heterogeneous with the bulk at 5 different scale

5.4.2. Limestone

From the results displayed in Figure 50, difference in porosity of homogeneous and heterogeneous surface is significantly higher at scale of 1st order (20µm) than other scales. Difference in porosity in heterogeneous and homogeneous is more at low scale. Porosity range from 0.11% to 2.88 % for homogeneous surface and 0.50% to 4.24% for heterogenous and as bulk rock porosity range from 0.30% to 3.09%.

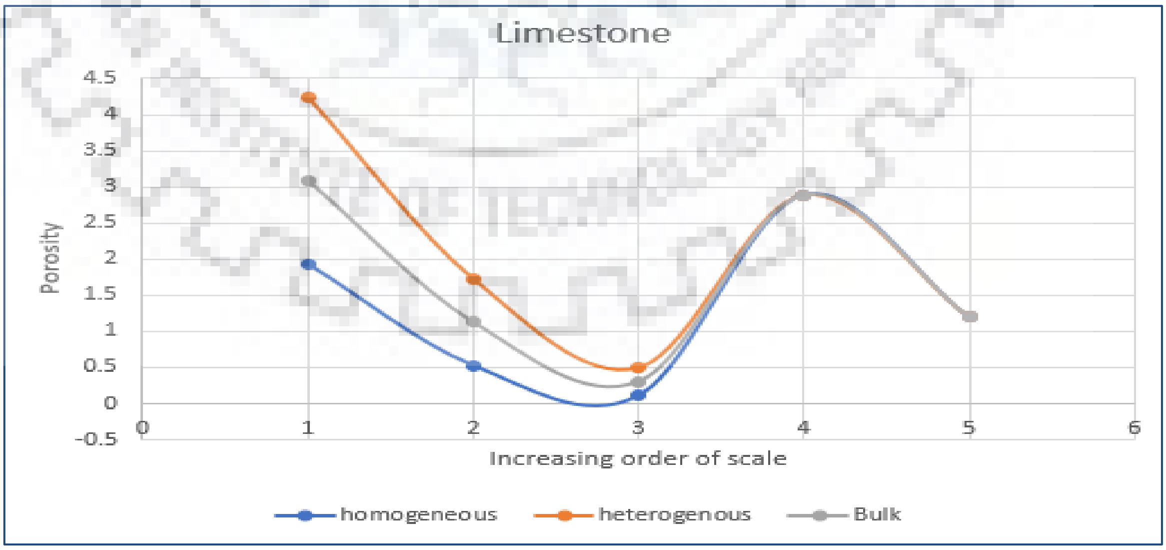

Figure 50 variation of Porosity of Limestone of homogeneous and heterogeneous with the bulk at 5 different scale

5.4.3. Sandstone

From the results displayed in Figure 51, difference in porosity of homogeneous and heterogeneous surface is significantly higher at a scale of 1st order (20μm) than other scales. Difference in porosity in heterogeneous and homogeneous is more at low scale. Porosity of homogeneous and heterogeneous surface is same at a scale of 1mm. Porosity range from 0.24% to 3.23 % for homogeneous surface and 0.29% to 4.78% for heterogenous and as bulk rock porosity range from 0.27% to 4.01%.

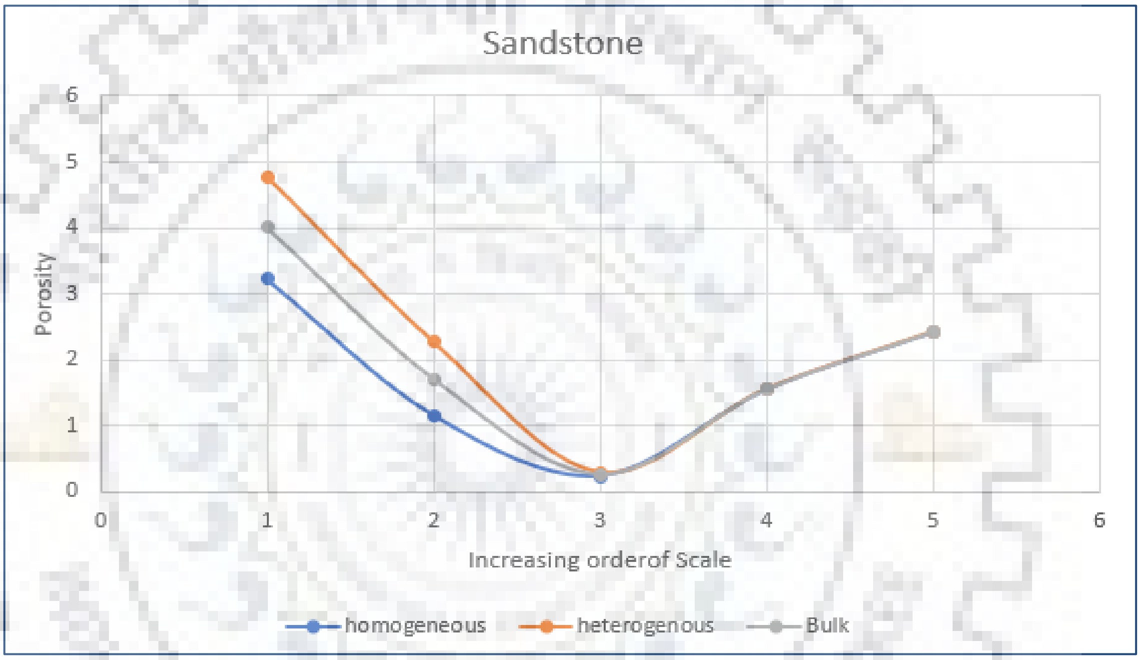

Figure 51 variation of Porosity of Sandstone of homogeneous and heterogeneous with the bulk at 5 different scale

5.4.4. Granite Gneiss

From the results displayed in Figure 52, difference in porosity of homogeneous and heterogeneous surface is significantly higher at scale of 2nd order (200μm) than other scales. Difference in porosity in heterogeneous and homogeneous is almost negligible at 20μm and 1mm scale. Porosity range from 0.66% to 8.51 % for homogeneous surface and 0.7% to 8.51% for heterogenous and as bulk rock porosity range from 0.68% to 8.51%.

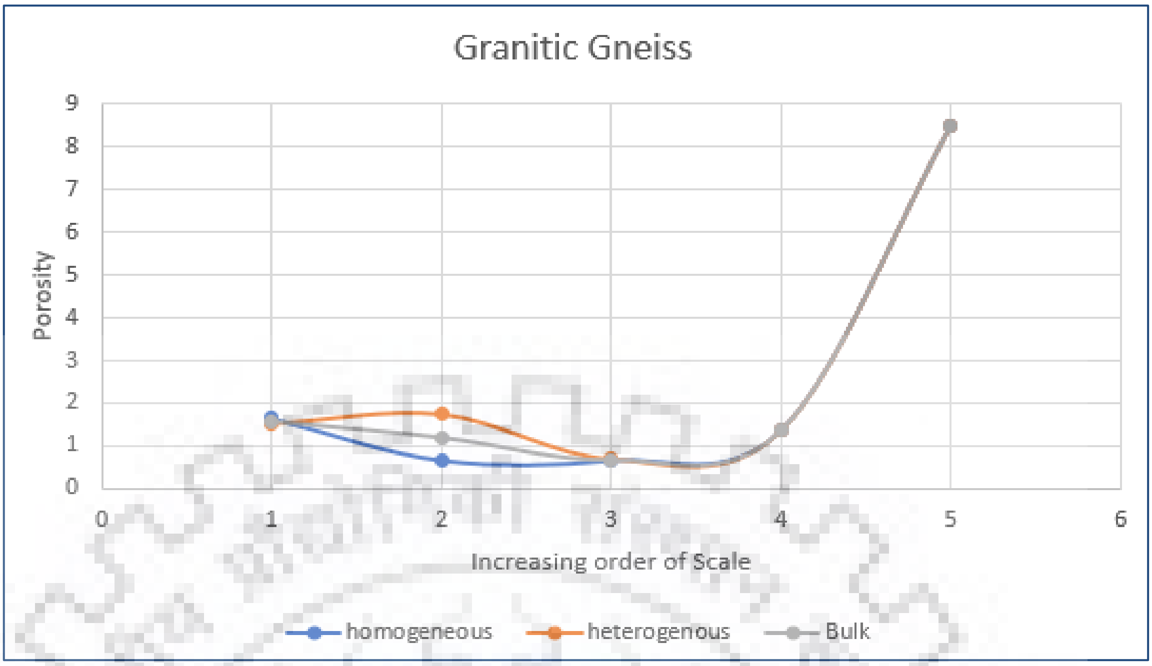

Figure 52 variation of Porosity of Granitic Gneiss of homogeneous and heterogeneous with the bulk at 5 different scale

5.4.5. Dolomite

From the displayed results in Figure 53, there is porosity difference in homogeneous and heterogeneous surface at scale of 1st order (20μm). Porosity range from 0.88% to 10.44 % for homogeneous surface and 1.26% to 12.85% for heterogenous and as bulk rock porosity range from 1.07% to 10.97%.

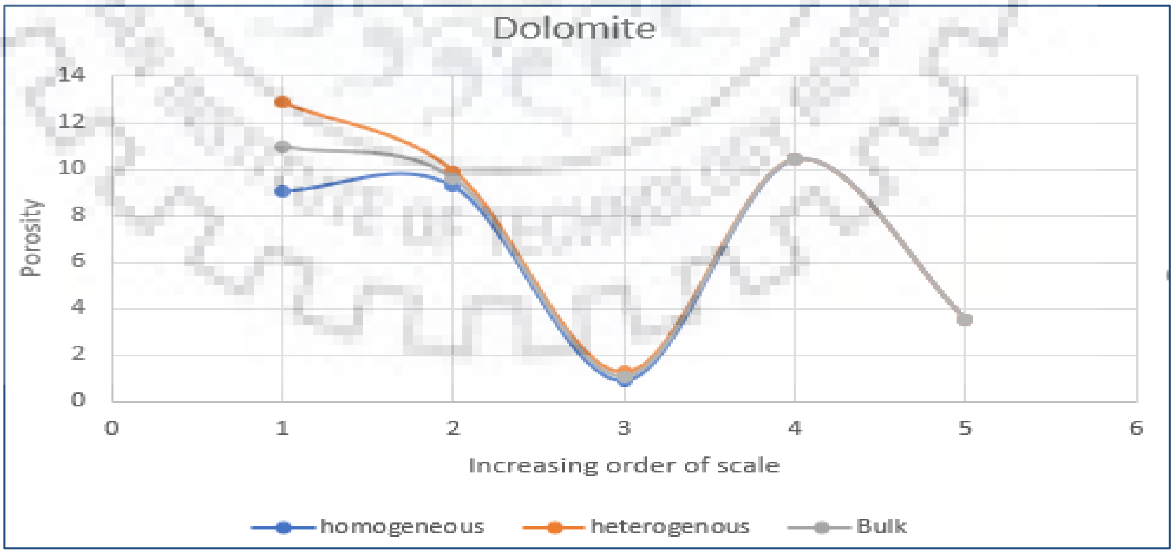

Figure 53 variation of Porosity of Dolomite of homogeneous and heterogeneous with the bulk at 5 different scale

5.5. Relation of Porosity with Bulk Density

From the displayed results in Table 15 and Figure 54, it can be found that Porosity decrease with increase in Bulk density.

Table 15 Relation of Porosity and Bulk density of different rocks

Rock	Bulk density (calculated)	Porosity(%)
Metabasalt	2.99	0.63
Limestone	2.81	2.88
Sandstone	2.81	1.55
Granite Gneiss	2.60	1.38
Dolomite	2.50	10.44

.

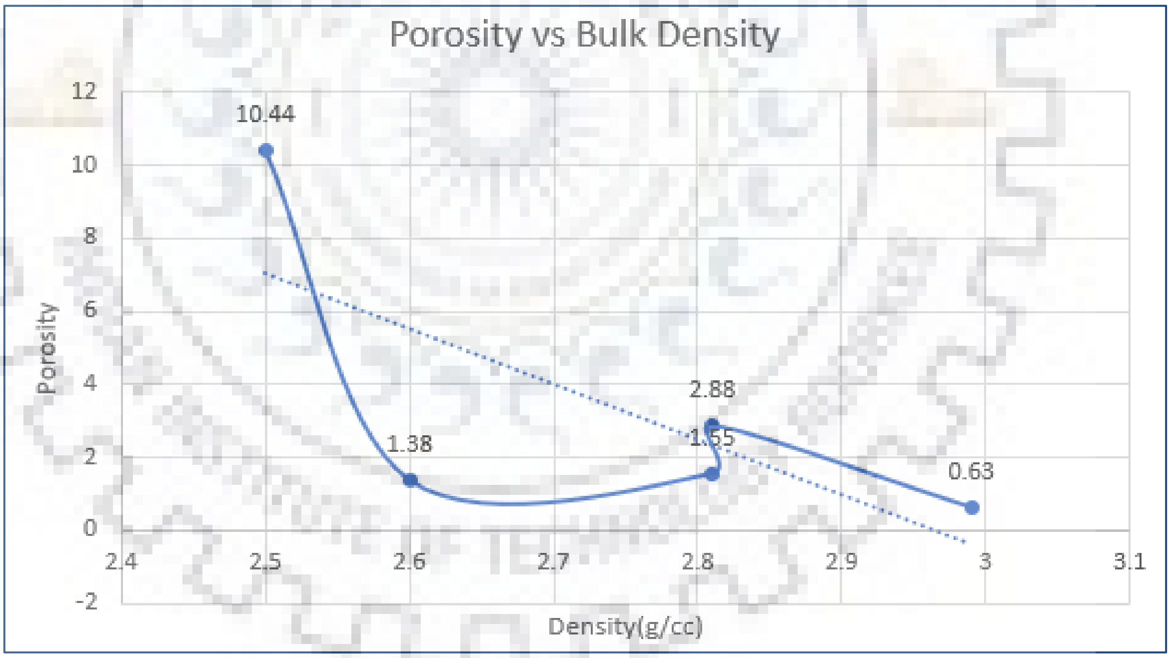

Figure 54 Porosity and Bulk Density Relation

5.6. Relation of Porosity with Compressional wave velocity (V_p)

From the displayed results in Table 16 and Figure 55, it can be clearly seen that the Porosity decrease in rocks which have higher compressional wave velocity.

Table 16 Relation of Vp and Porosity of rocks

Sample Name	**V_p (m/s)**	**V_s(m/s)**	**V_{ma}(m/s)**	**V_f(m/s)**	**porosity(P)**
Meta Basalt	5707	3535	7620	335.28	1.54
Limestone	5266	3724	6400.80	335.28	1.19
Sandstone	4156	2883	5791.20	335.28	2.42
Granite Gneiss	2378	1441	5486.40	335.28	8.51
Dolomite	4110	2932	7010.40	335.28	3.54

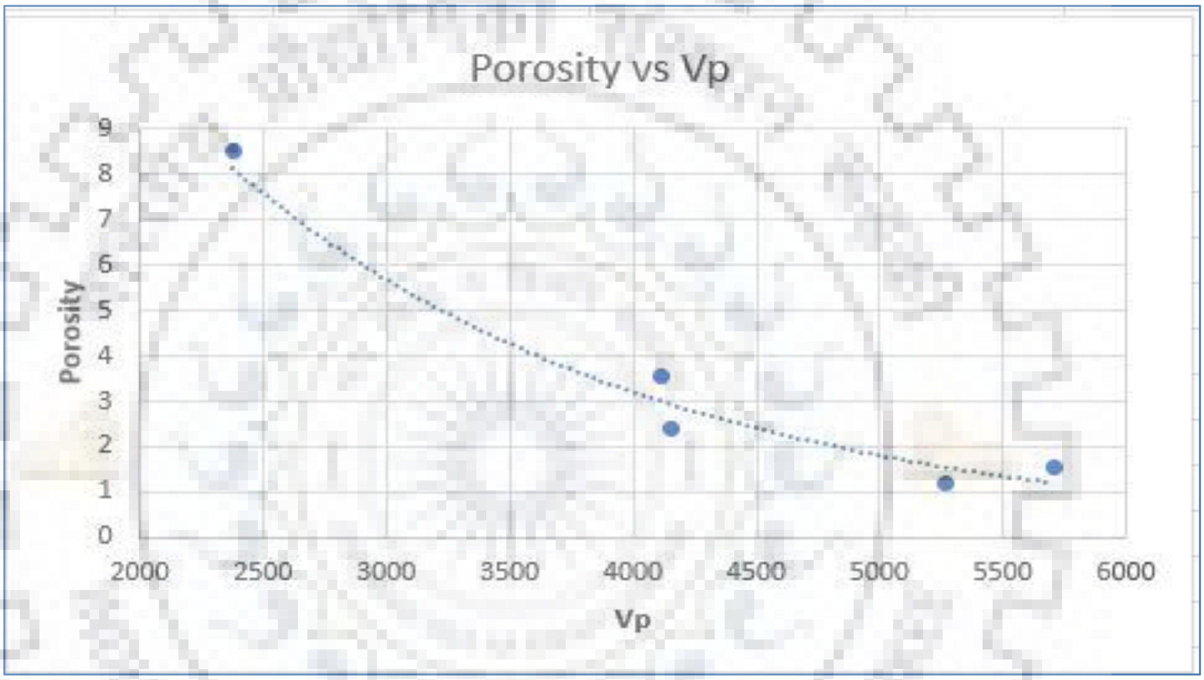

Figure 55 Porosity and V_p Relation

5.7. Relation of Porosity with Modulus

Table 17 Porosity and Elastic Modulus

Rock	**Bulk Modulus**	**Shear Modulus**	**Young's Modulus**	**Porosity**
Meta-Basalt	47.58	37.38	88.86	1.54
Limestone	22.7	41.48	82.94	1.19
Sandstone	15.39	24.86	51.52	2.42
Granite Gneiss	6.4	6.21	15.03	8.51
Dolomite	7.91	25.71	50.5	3.55

5.7.1. Relation of Shear Modulus with Porosity

From displayed results in Figure 56, exponential relationship between porosity and modulus of elasticity is found. Shear modulus of rocks is increasing with decrease in porosity.

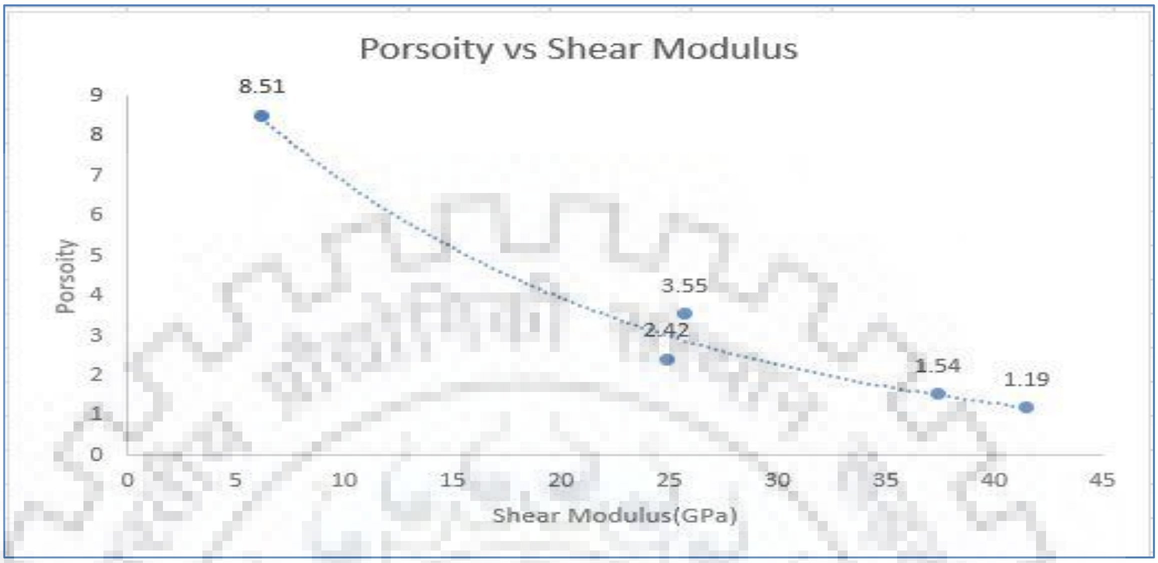

Figure 56 Relation of Porosity with shear modulus for different rocks

5.7.2. Relation of Bulk Modulus with Porosity

From displayed results in Figure 57, linear relationship between porosity and modulus of elasticity (in GPa) could be interpreted. Bulk modulus of rocks is decreasing with increase in porosity.

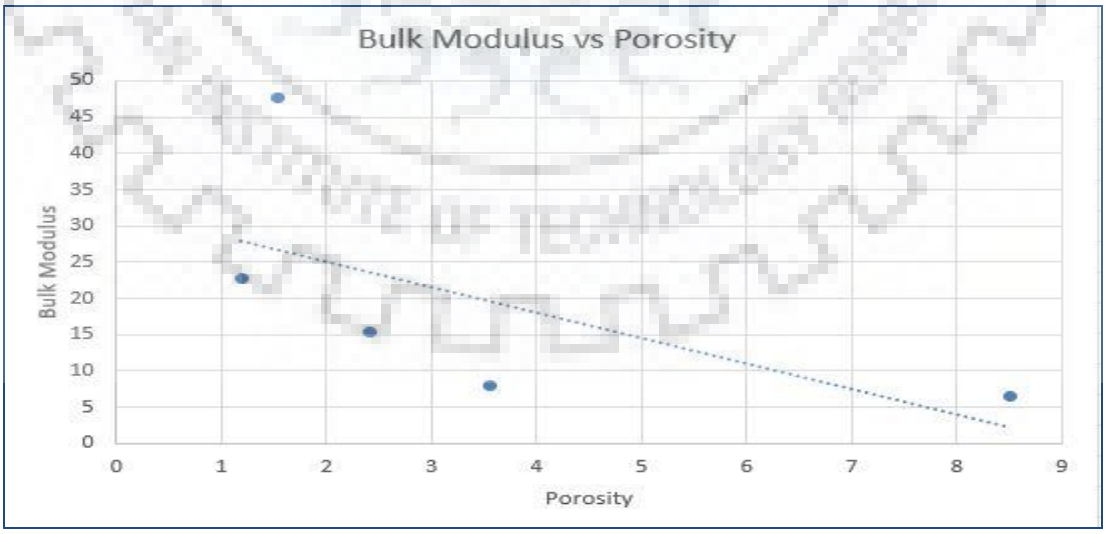

Figure 57 Relation of porosity with Bulk modulus for different rocks

5.7.3. Relation of Young's Modulus with Porosity

From the displayed results in Figure 58, linear relationship between porosity and Young's modulus could be seen. Youngs modulus of rocks is decreasing with increase in porosity.

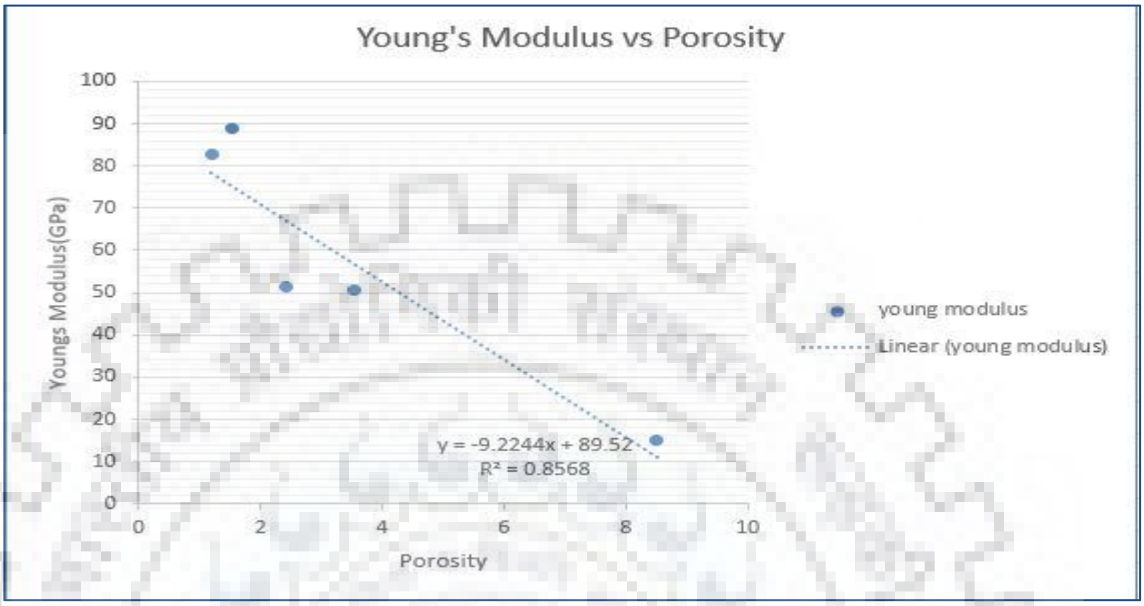

Figure 58 Relation of young's modulus with porosity for different rocks

5.7.4. Relation of Modulus of Elasticity with porosity

From Figure 59, linear relationship between porosity and modulus of elasticity could be seen. Youngs modulus, Bulk modulus, and shear modulus all are decreasing with increase in porosity.

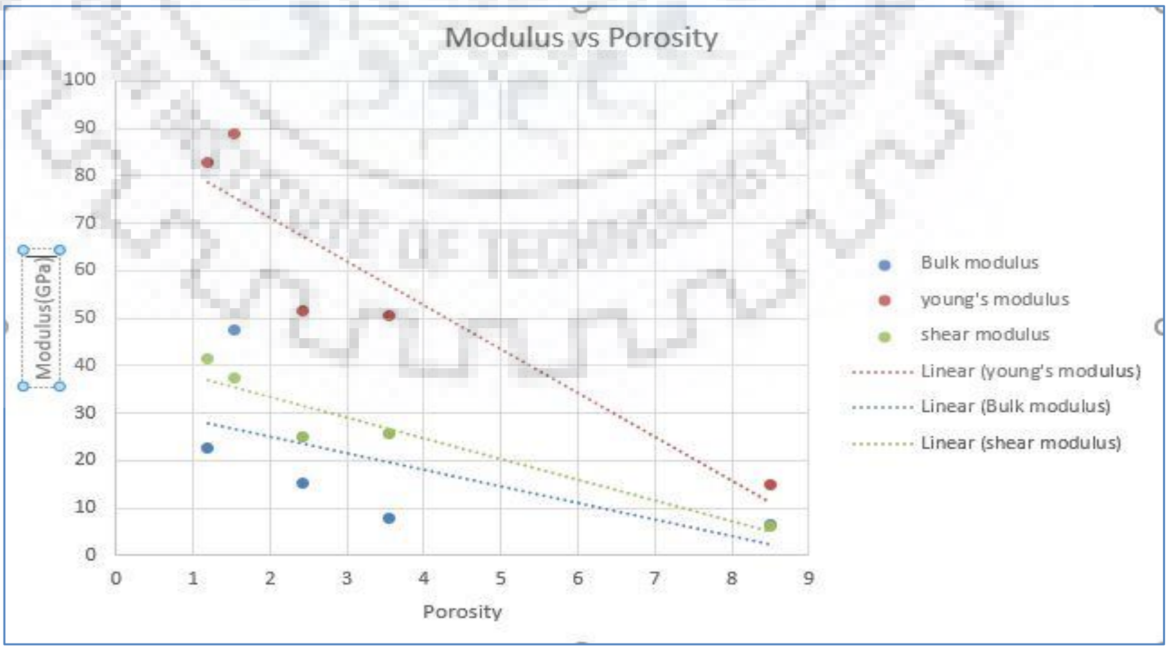

Figure 59 Relation of elastic modulus with porosity

Chapter 6: Conclusion

- Modulus of elasticity shows a dependence on porosity. Young's, bulk and shear modulus decrease with increase in porosity.
- Porosity is method and scale dependent for same rock and also different for different rocks
- V_p decrease with increase in porosity which in general decrease with increase in density
- Assuming field porosity to be a standard values at scale of 1cm, it can be concluded that Meta-Basalt show optimum porosity at scale of 200µm and 1000µm. Limestone show optimum porosity at 20µm scale. Granitic Gneiss and Dolomite show optimum porosity at 20µm and 200µm scale.
- 200µm scale is found to be most suitable scale for all five types of rocks for porosity calculation because it matches with the field porosity.
- Dolomite showing exceptionally low porosity at scale of 1mm which shows underestimated results in comparison to field porosity.
- Granitic Gneiss showing exceptionally high porosity at scale of 10 cm, which shows overestimated results in comparison to field porosity.

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