ENGINEERING BEHAVIOR OF OIL SHALE UNDER HIGH PRESSURE AFTER THERMAL TREATMENT

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by

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Dedicated to my

Grandparents, Parents and Sisters

for their support and belief

This humble work is a sign of my love to you

CERTIFICATE

This is to certify that the thesis entitled, **"Engineering Behavior of Oil Shale under High Pressure after Thermal Treatment**", being submitted by **Mr. Ankesh Kumar** to the Indian Institute of Technology Delhi, for the award of **Doctor of Philosophy'** is a record of the Bonafide research work carried out by him under my supervision of this thesis, which to the best of my knowledge has reached the requisite standard. The material contained in this thesis has not been submitted in part or fully to any other university or institute for the award of any degree or diploma.

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ABSTRACT

The petroleum reserves are one of the major source of energy for both developed and developing nations, and the depletion of existing resources has increased the need for the alternative energy sources to petroleum. Therefore, the oil/gas shales are emerged as a viable substitute, due to the improved technologies such as hydro-fracturing. The oil shale is an unconventional resource, which is formed due to the simultaneous deposition of the fine-grained mineral debris and organic matter. The extraction process involves borehole construction, fracturing, injection to achieve communication, heating the formation at 300 to 400 ^{o}C, and fluid migration at the underground location. Therefore, the understanding of petrology, physical properties, permeability and mechanical properties of shale rocks at high pressure and temperature is very important for the effective recovery of shale oil/gas. Hence, the oil shale rock from the upper Assam region is considered for the present work. To understand the temperature effect, the investigation is performed on the oil shale samples which are thermally treated at a temperature between 100 to 800 ^{o}C.

The investigation related to physical properties suggests that the saturated water content, effective porosity and sonic wave velocity of oil shale decrease with increase in anisotropic angle. This variation is observed due to the fine laminated structure of the rock. The investigation related to the effect of thermal treatment on the physical properties revealed that the dry density and sonic wave velocity of oil shale decrease with increase of thermal treatment from 300 ^{o}C to 600 ^{o}C.

The petrographic investigations of oil shale rocks suggest that these rocks contain clay size particles. The inorganic matrix consists of quartz silt (Q), kaolinite, illite, pyrite, plagioclase, K-feldspar, mixed calcite, and siderite. It is observed that these rocks contain the thin laminated structure of micron thickness. Presence of irregular shaped micro level voids (V) and internal cracks (IC) along the grain boundaries are also observed. Furthermore, it is observed that the oil shale from Assam contains 10–17% extractable organic matter. The change in color of the oil shale from dark grey at 100 ^{o}C to red at 600 ^{o}C is also observed. The change in the color is due to the thermal decomposition of the ferrous minerals. The investigation through XRD suggests that the dehydration takes place up to 400^{o}C. Further, the dehydroxylation of illite and kaolinite takes place up to 600^{o}C, and due to this the kaolinite is decomposed to amorphous metakaolin, but the illite is stable at this temperature. The transition of α quartz to β – quartz is observed at 600^{o}C temperature. The SEM analysis shows that the frequency of cracks increases with increase in thermal treatment.

The engineering behaviour of oil shale is investigated by considering strength anisotropy. The specimens are prepared at 0^{o}, 30^{o}, 45^{o}, 60^{o} and 90^{o} orientation angles. The effect of foliation plane (fp) and loading angle on the tensile strength of oil shale rock is examined. It is observed that

the tensile strength is maximum for foliation angle (Ψ) of 90^o, and minimum at 30^o. It is found that the Brazilian strength increases as the β increases from 0^o to 90^o for the 0^o Ψ. The investigation shows that the tensile strength is a function of both Ψ and β. It is also observed that the axial point load strength increases with increase in loading angle. Further, the investigations related to diametral case suggest that the diametral point load strength decreases with increase in loading angle.

It is found through experimentation that the permeability of oil/gas shale rock is a function of porosity (φ), pore compressibility (K_p), effective stress (σ_{eff}) and permeability at the surface (k_o). It is noticed that the hydraulic conductivity (K) and permeability (k), for all anisotropic angles, decrease as confinement pressure increases. It is also found that the hydraulic conductivity and permeability values decrease as the anisotropy angle varies from 0^o to 90^o. The effect of β angle on permeability is observed due to the laminations, which allows the water to permeate at a higher rate through them. Further, the applicability of the power law and exponential model of stress-dependent permeability is investigated, and the required modifications are carried out to incorporate the anisotropic effect.

The uniaxial and triaxial testing is conducted on the samples after thermal treatment at 100, 300, 450 and 600 ^oC for 16 hours. It is found that the compressive strength is maximum for $\beta = 90^o$ and minimum at $\beta = 30^o$ in all the cases of thermal treatment. The thermally treated oil shale rock shows plastic – elastic – plastic behaviour at 300, 450 and 600 ^oC. The Young's modulus E_t is maximum at $\beta = 0^o$ and minimum at $\beta = 30^o$. It is also observed that the E_t value decreases as thermal treatment temperature changes from 300 to 600 ^oC. It is also noted that the Poisson's ratio decreases as thermal treatment temperature changes from 300 to 600 ^oC. Therefore it can be concluded that increase in crack volume and alteration of mineralogy with temperature affected the strength of the rock. The oil shale rock shows "Shoulder type" anisotropy due to the presence of single set of lamination of foliation plane. The temperature effect on strength is investigated through thermal degradation ratio, which is the ratio of uniaxial compressive strength at β angle after thermal treatment at T ^oC ($\sigma_{c\beta T}$) to uniaxial compressive strength at β angle after thermal treatment at 100 ^oC ($\sigma_{c\beta 100}$). It is observed that with increase in temperature, the thermal degradation ratio decreases for all angles of β.

The failure strength (σ_1) of thermally treated oil shale is determined as a function of confining pressure (σ_3) and orientation angle (β). The experimental investigation suggests that the failure strength and Young's modulus vary non-linearly with the confinement stress. It is observed that the variation of cohesion and friction angle with orientation angle is similar to the strength anisotropy behaviour. Further, the material constants of Ramamurthy et al. (1988) criterion and modified Hoek-Brown criterion for thermally treated oil shale rock are investigated. The predicted strength values through both the criteria show good agreement with experimental values at 100 ^{o}C. But the predicted values for 450 and 600 ^{o}C from Ramamurthy et al. (1988) do not match with the experimental values. Whereas, the number of triaxial tests for modified Hoek-Brown failure criterion is very large. Therefore, the above strength criteria are modified by introducing the thermal degradation ratio (R_{cT}) in equation.

The R_{cT} values $\left(\frac{\sigma_{c\beta T}}{\sigma_{c\beta 100}}\right)$ can be calculated by performing unconfined compressive strength test at $\beta = 0^{o}$ anisotropy angle. The coefficient of correlation r^2 calculated for the modified criterion show that the proposed criteria fits well with the experimental values. The considerable amount of improvement is observed in the r^2 value for Ramamurthy et al. (1988) criterion.

Finally, the values obtained through physical, mineralogical and mechanical testing of oil shale rock can be used for the investigations of well bore stability. Further, it can be concluded that the strength and deformation behaviour of rocks is dependent on both in-situ stress state and temperature conditions. The modified equations proposed for the strength prediction of thermally treated anisotropic rocks in the present research can be used for the proper engineering designs and long term stability assessment of deep underground structures, such as underground caverns for storage of radioactive waste, boreholes for oil/gas production, and wells for injection of carbon dioxides, etc.

सार

पेट्रोलियम भंडार विकसित और विकासशील दोनों देशों के लिए ऊर्जा के प्रमुख स्रोत में से एक है, और मौजूदा संसाधनों की कमी ने पेट्रोलियम के लिए वैकल्पिक ऊर्जा स्रोतों की आवश्यकता को बढ़ा दिया है। इसलिए, हाइड्रो-फ्रैक्चरिंग जैसी बेहतर तकनीकों के कारण, तेल / गैस शेल एक व्यवहार्य विकल्प के रूप में उभरे हैं। ऑयल शेल एक अपरंपरागत संसाधन है, जो महीन दानेदार खनिज मलबे और कार्बनिक पदार्थों के एक साथ जमाव के कारण बनता है। निष्कर्षण प्रक्रिया में बोरहोल निर्माण, फ्रैक्चरिंग, संचार को प्राप्त करने के लिए इंजेक्शन, 300 से 400 °C पर गठन को गर्म करना और भूमिगत स्थान पर द्रव प्रवास शामिल है। अतेव, उच्च दबाव और तापमान पर शेल चट्टानों के पेट्रोलॉजी, भौतिक गुणों, पारगम्यता और यांत्रिक गुणों की समझ, शेल तेल / गैस की प्रभावी वसूली के लिए बहुत महत्वपूर्ण है। इसलिए, वर्तमान कार्य के लिए ऊपरी असम क्षेत्र के तेल शेल रॉक का उपयोग किया गया है। तापमान प्रभाव को समझने के लिए, तेल शेल नमूनों पर जांच की गई है, जिनका 100 से 800 °C के बीच तापमान पर थर्मली उपचारित किया गया है।

तेल शेल रॉक के भौतिक गुणों से संबंधित जांच बताती है कि एनिस्ट्रोपिक कोण में वृद्धि के साथ संतृप्त पानी की मात्रा, प्रभावी सरंध्रता और ध्वनि तरंग वेग में कमी होती है। चट्टान के लैमिनेटेड संरचना के कारण यह भिन्नता देखी जाती है। भौतिक गुणों पर थर्मल उपचार के प्रभाव से संबंधित जांच से पता चला है कि तेल शेल रॉक का सूखा घनत्व और ध्वनि तरंग वेग थर्मल उपचार की वृद्धि (300 °C से 600 °C) के साथ कम हो जाता है।

तेल शेल चट्टानों की पेट्रोग्राफिक जांच से पता चलता है कि इन चट्टानों में मिट्टी के आकार के कण होते हैं। अकार्बनिक मैट्रिक्स में क्वार्ट्ज गाद, केओलिनइट, इलाइट, पाइराइट, प्लाजियोक्लेज़, के-फेल्डस्पार, मिश्रित कैल्साइट और साइडराइट शामिल हैं। यह भी देखा गया है कि इन चट्टानों में माइक्रोन मोटाई की पतली परतदार संरचना होती है। दाने की सीमाओं के साथ अनियमित आकार के सूक्ष्म स्तर छिद्र (V) और आंतरिक दरारें (IC) की उपस्थिति भी देखी जाती है। इसके अलावा, यह देखा गया है कि असम से तेल शेल चट्टानों में 10 से 17% निकालने योग्य कार्बनिक पदार्थ मौजूद हैं। गहरे भूरे रंग (100 °C पर) से लाल रंग (600 °C पर) में परिवर्तन भी देखा गया है। तेल शेल रॉक के रंग में यह परिवर्तन लौह खनिजों के थर्मल अपघटन के कारण होता है। X.R.D के माध्यम से जांच से पता चलता है कि निर्जलीकरण 400 °C तक होता है। इसके अलावा, इलाइट और केओलिनइट का निर्जलीकरण 600°C तक होता है, और इसके कारण केओलिनइट को अनाकार मेटाकॉलिन से विघटित कर दिया जाता है, लेकिन इस तापमान पर इलाइट स्थिर होता है। α - क्वार्ट्ज से β - क्वार्ट्ज का संक्रमण 600°C तापमान पर देखा गया है। S.E.M विश्लेषण से पता चलता है कि थर्मल उपचार में वृद्धि के साथ दरारों की आवृत्ति बढ़ जाती है।

तेल शेल के इंजीनियरिंग व्यवहार की जांच शक्ति ऐनआइसोट्रॉपी पर विचार करके की गई है। तेल शेल के नमूने 0°, 30°, 45°, 60° और 90° अभिविन्यास कोणों पर तैयार किए गए है। तेल शल रॉक की तन्य शक्ति पर फोलिएशन प्लेन (fp) और लोडिंग कोण के प्रभाव की जांच की गई है। यह देखा गया है कि तन्य शक्ति 90° के फोलिएशन कोण (of) के लिए अधिकतम है, और 30° पर न्यूनतम है। यह पता चला है कि ψ 0° के लिये, 0° से 90° तक लोडिंग कोण में वृद्धि के साथ ब्राज़ीलियन तन्य शक्ति में भी वृद्धि होती जाती है। जांच से पता चलता है कि तन्य शक्ति, Ψ और β दोनों पर निर्भर है। यह भी देखा गया

है कि लोडिंग कोण में वृद्धि के साथ अक्षीय बिंदु भार शक्ति बढ़ जाती है। इसके अलावा, व्यास संबंधी मामले से संबंधित जांच से पता चलता है कि लोडिंग कोण में वृद्धि के साथ व्यास संबंधी बिंदु भार शक्ति घट जाती है।

यह प्रयोग के माध्यम से पाया गया है कि तेल / गैस शेल रॉक की पारगम्यता, सरंध्रता (φ), छिद्र संपीडन (K_p), प्रभावी तनाव (σ_{eff}) और सतह पर पारगम्यता (k_o) पर निर्भर है। सभी ऐनआइसोट्रोपिक कोणों के लिए, यह पाया जाता है कि कार्मिक दबाव में वृद्धि के साथ द्रवचालित प्रवाहिता (K) और पारगम्यता (k) घट जाती है। यह भी पाया गया कि 0° से 90° तक ऐनआइसोट्रोपिक कोण में परिवर्तन के साथ द्रवचालित प्रवाहिता और पारगम्यता मान कम हो जाते हैं। पारगम्यता पर β कोण का प्रभाव लामिनेशन के कारण देखा गया है, जो पानी को उनके माध्यम से उच्च दर पर प्रवाहित करने की अनुमति देता है। इसके अलावा, शक्ति कानून और तनाव-निर्भर पारगम्यता के घातीय मॉडल की प्रयोज्यता की जांच की गई है, और ऐनआइसोट्रोपिक प्रभाव को शामिल करने के लिए आवश्यक संशोधनों को किया गया है।

नमूनों को 16 घंटे के लिए 100, 300, 450 और 600 °C पर गर्म किया गया है, और इन नमूनों पर ऊष्मीय उपचार के बाद एकपक्षीय और त्रिअक्षीय परीक्षण किया गया है। यह पाया गया है कि ऊष्मीय उपचार के सभी मामलों में संकुचित शक्ति β = 90° के लिए अधिकतम है और β = 30° पर न्यूनतम है। 300, 450 और 600 °C पर ऊष्मीय उपचारित तेल शेल रॉक प्लास्टिक - लोचदार - प्लास्टिक व्यवहार को दर्शाता है। यंग का मापांक β = 0° पर अधिकतम है, और β = 30° पर न्यूनतम है। यह भी देखा गया है कि ऊष्मीय उपचार तापमान में 300 से 600 °C तक के बदलाव के साथ E_t वैल्यू घट जाती है। यह भी पाया गया है कि ऊष्मीय उपचार तापमान में 300 से 600 °C के बदलाव के साथ पॉइसन का अनुपात कम हो जाता है। इसलिए यह निष्कर्ष निकाला जा सकता है कि दरार की मात्रा में वृद्धि और तापमान के साथ खनिज विज्ञान के परिवर्तन के साथ चट्टान की ताकत प्रभावित होती है। तेल शेल रॉक में फोलिएशन प्लेन के परतबंदी के एकल सेट की मौजूदगी "शोल्डर टाइप" ऐनआइसोट्रोपि को दर्शाती है। तापीय क्षरण अनुपात के माध्यम से ताकत पर तापमान प्रभाव की जांच की गई है, जो कि β कोण पर T °C पर ऊष्मीय उपचार के बाद अक्षीय संकुचित शक्ति ($\sigma_{c\beta T}$) और 100 °C पर ऊष्मीय उपचार के बाद अक्षीय संकुचित शक्ति ($\sigma_{c\beta 100}$) का अनुपात है। यह देखा गया है कि सभी β कोणों के लिए, तापमान में वृद्धि के साथ थर्मल क्षरण अनुपात में कमी होती है।

ऊष्मीय उपचारित तेल शेल की विफलता की ताकत (σ_1), सीमित दबाव (σ_3) और अभिविन्यास कोण (β) पर निर्भर करती है। प्रयोगात्मक जांच से पता चलता है कि विफलता की ताकत और यंग का मापांक गैर-रेखीय रूप से सीमित दबाव के साथ भिन्न होता है। यह देखा गया है कि अभिविन्यास कोण के साथ सामंजस्य और घर्षण कोण की भिन्नता, शक्ति ऐनआइसोट्रोपि व्यवहार के समान है। इसके अलावा, ऊष्मीय उपचारित तेल शेल रॉक के लिए राममूर्ति एट अल. (1988) कसौटी और संशोधित होएक-ब्राउन कसौटी की सामग्री स्थिरांक की जांच की की गई है। दोनों मानदंडों के माध्यम से अनुमानित शक्ति मान 100 °C पर प्रयोगात्मक मूल्यों के साथ अच्छा समझौता दिखाते हैं। लेकिन राममूर्ति एट अल. (1988) से 450 और 600 °C के लिए अनुमानित मूल्य प्रयोगात्मक मूल्यों के साथ मेल नहीं खाते हैं। जबकि, संशोधित होएक-ब्राउन विफलता मानदंड के लिए त्रिअक्षीय परीक्षण की संख्या बहुत

बड़ी है। इसलिए, समीकरण में तापीय क्षरण अनुपात (R_{cT}) को पेश करके उपरोक्त शक्ति मानदंड को संशोधित किया गया है।

R_{cT} मान $\left(\frac{\sigma_{c\beta T}}{\sigma_{c\beta 100}}\right)$ की गणना $\beta = 0^o$ कोण पर अपुष्ट संपीड़ित शक्ति परीक्षण करके की जा सकती है। संशोधित मानदंड के लिए गणना सहसंबंध r^2 के गुणांक से पता चलता है कि प्रस्तावित मानदंड प्रयोगात्मक मूल्यों के साथ अच्छी तरह से काम करता है। राममूर्ति एट अल. (1988) की कसौटी के लिए r^2 मूल्य में काफी सुधार देखा गया है।

अंत में, तेल शेल रॉक के भौतिक, खनिज और यांत्रिक परीक्षण के माध्यम से प्राप्त मूल्यों का उपयोग अच्छी तरह से बोर स्थिरता की जांच के लिए किया जा सकता है। इसके अलावा, यह निष्कर्ष निकाला जा सकता है कि चट्टानों की ताकत और विरूपण व्यवहार दोनों इन-सीटू तनाव स्थिति और तापमान की स्थिति पर निर्भर है। वर्तमान शोध में ऊष्मीय उपचारित अनिसोट्रोपिक चट्टानों की ताकत की भविष्यवाणी के लिए प्रस्तावित संशोधित समीकरणों का उपयोग उचित अभियांत्रिकी रचनाओं और गहरी भूमिगत संरचनाओं के दीर्घकालिक स्थिरता मूल्यांकन के लिए किया जा सकता है, जैसे कि रेडियोधर्मी कचरे के भंडारण के लिए भूमिगत गुफा, तेल या गैस उत्पादन के लिए बोरहोल, और कार्बन डाइऑक्साइड के इंजेक्शन के लिए कुओं, आदि में।

TABLE OF CONTENT

CERTIFICATE i

ACKNOWLEDGEMENTS ii

ABSTRACT iv

CONTENT vii

LIST OF FIGURES xv

LIST OF TABLES xxxii

LIST OF NOTATIONS xxxv

CHAPTER 1 1

INTRODUCTION 1

1. General 1

1.1 Objectives of the Thesis 3

1.2 Thesis Outline 3

CHAPTER 2 5

LITERATURE REVIEW 5

2. General 5

2.1 Depositional Process and World Deposits 5

2.1.1 Environment of Deposition and Lithology 5

2.1.2 World Distribution of Oil/Gas Shales 6

2.2 Classification of Oil/Gas Shale 9

2.3 Mineralogy of Shale and Oil/Gas Shale 11

2.4 Recovery Process 13

2.4.1 Surface Retorting 14

2.4.2 In Situ Retorting 15

2.5	Factors Influencing the Strength of Rocks	17
2.5.1	Geological Factors	17
2.5.2	Lithological Factor	17
2.5.3	Physical Properties	18
2.5.4	Mechanical Factors	20
2.5.5	Environmental Factors	20
2.6	Stress Dependent Permeability	21
2.6.1	Models for Stress Dependent Permeability	22
2.7	Anisotropy	24
2.7.1	Type of Anisotropy	25
2.8	Engineering Behaviour	26
2.8.1	Organic Content Determination	27
2.8.2	Thermo Gravimetric Analysis (TGA)	28
2.8.3	Tensile Strength	30
2.8.3.1	Effect of Loading Angle	31
2.8.3.2	Effect of Organic Content	38
2.8.4	Uniaxial Compressive Strength	38
2.8.4.1	Effect of Anisotropy	39
2.8.4.2	Effect of Organic Content	42
2.8.5	Triaxial Compressive Strength	43
2.8.5.1	Effect of Confinement on Anisotropy	44
2.8.6	Thermo – Mechanical Behaviour	45
2.9	Effect of Thermal Treatment	51
2.9.1	Physical Properties	51
2.9.2	Tensile Strength	52

2.9.3 Uniaxial Compressive Strength 54

2.9.4 Triaxial Compression 58

2.10 Intact Rock Classification 61

2.11 Brittle – Ductile Behaviour of Rocks 61

2.12 Failure Criteria for Rocks 65

2.12.1 Strength Criteria for Isotropic Rocks 65

2.12.2 Strength Criteria for Anisotropic Rocks 68

2.13 Failure Patterns 73

2.14 Critical Observations 75

2.15 Conclusions 79

CHAPTER 3 81

GEOLOGICAL SETTING AND EXPERIMENTAL INVESTIGATION 81

3. General 81

3.1 Oil/Gas Shale Deposits and Tectonic Setting 81

3.1.1 Cambay Basin 83

3.1.2 Krishna Godavari Basin 86

3.1.3 Cauvery Basin 88

3.1.4 Damodar Valley Basin 91

3.1.5 Upper Assam Basin 93

3.2 Sampling Location 101

3.2.1 Rock Formation 101

3.2.2 Stratigraphy 102

3.2.2.1 Disang Group 102

3.2.2.2 Barail Group 103

3.2.2.2.1 Tikak Parbat Formation 103

3.2.2.2.2 Baragolai Formation 105
3.2.2.2.3 Naogaon Sandstone 105
3.2.2.3 Tipam Group 106
3.2.2.4 Dihing Group 106
3.3 Sampling Process 106
3.3.1 Coring in Laboratory 109
3.3.2 In Situ Coring 110
3.3.3 Sample Preservation 111
3.4 Specimen Preparation 114
3.4.1 Procedure for Specimen Preparation 114
3.5 Experimental Investigation 116
3.5.1 Thermal Treatment 116
3.5.2 Geotechnical Behaviour 119
3.5.2.1 Brazilian Test 119
3.5.2.2 Point Load Test 120
3.5.2.3 Unconfined Compression Test 122
3.5.2.4 Triaxial Compression Test 122
3.5.2.5 High Pressure Triaxial Cell 123
3.5.2.6 Loading and Load Monitoring System 128
3.5.2.7 Strains Measuring System 130
3.5.2.8 Permeability Testing 131
3.6 Physical Index Properties 132
3.6.1 Density and Specific Gravity 132
3.6.2 Porosity and Water Absorption 133
3.6.3 Ultrasonic Velocity 135

3.6.4 Effect of Temperature 136
3.7 Conclusions 139
CHAPTER 4 141
PETROLOGY AND CHEMICAL STUDIES 141
4. General 141
4.1 Thin Section Analysis 141
4.1.1 Thin Section Preparation 142
4.1.2 Petrology through Thin Section 143
4.2 X-Ray Diffraction Method (XRD) 152
4.2.1 Material and Methods 152
4.2.2 Qualitative and Quantitative Analysis 153
4.3 Scanning Electron Microscopy (SEM) 158
4.3.1 Instrument and Sample Preparation 158
4.3.2 Surface Morphology 160
4.3.3 Pore Characterization 171
4.4 Transmission Electron Microscopy (TEM) 177
4.5 Elemental Analysis 179
4.5.1 Energy-Dispersive X-Ray Spectroscopy (EDX) 180
4.5.2 CHNS Analysis 182
4.6 Thermal Gravimetric Analysis (TGA) 185
4.7 Chemical Test for Inorganic Carbon 188
4.8 Free Swell Index Test 189
4.9 Thermal Degradation 190
4.9.1 Texture and Weight Loss Analysis 191
4.9.2 X-Ray Diffraction Analysis 193

4.9.3 Scanning Electron Microscopy 200

4.10 Comparision of Mineral Composition of Different Oil/Gas Shales 205

4.11 Conclusions 207

CHAPTER 5 209

STRENGTH INDEX PROPERTIES AND STRESS DEPENDENT PERMEABILITY 209

5. General 209

5.1 Tensile Strength 209

5.1.1 Brazilian Strength (σ_t) 209

5.1.2 Point Load Strength (I_{s50}) 214

5.2 Unconfined Compressive Strength 218

5.3 Strength and Deformation Behaviour 220

5.3.1 Stress – Strain Curve 220

5.3.2 Young’s Modulus and Poisson’s Ratio 224

5.3.3 Intact Rock Classification 227

5.4 Anisotropy 229

5.4.1 Compressive Strength Anisotropy 229

5.4.2 Tensile Strength Anisotropy 229

5.4.3 Young’s Modulus Anisotropy 230

5.4.4 Strain Anisotropy Ratio 231

5.5 Effect of Thermal Treatment 231

5.6 Failure Patterns in Unconfined State 233

5.7 Stress-Dependent Permeability 236

5.7.1 Bulk Compressibility 237

5.7.2 Effective Porosity 243

5.7.3 Permeability 244

5.7.4 Model for Stress-Dependent Permeability for Shales 248
5.8 Conclusions 256
CHAPTER 6 259
STRENGTH AND DEFORMATION BEHAVIOUR OF OIL SHALE 259
6. General 259
6.1 Prediction of Unconfined Compressive Strength 259
6.2 Stress-Strain Behaviour under Confined State 261
6.3 Effect of Confinement on Deformation Behaviour 270
6.3.1 Effect of Confinement on Young's Modulus (E_{t50}) 270
6.3.2 Poisson's Ratio under Confined State 280
6.3.3 Effect of Confinement on Failure Strain 282
6.3.3.1 Axial Strain (ε_a) 282
6.3.3.2 Diametral Strain (ε_d) 282
6.4 Strength Behaviour in Triaxial Compression 286
6.5 Effect of Confinement on Shale Anisotropy 292
6.6 Prediction of Failure Strength for Oil/Gas Shales 294
6.7 Proposed Criterion for Thermally Treated Rocks 307
6.8 Shear Strength Parameters 311
6.9 Brittle – Ductile Transition 323
6.10 Failure Pattern in Triaxil Stress State 326
6.11 Comparison 331
CHAPTER 7 335
SUMMARY AND CONCLUSIONS 335
7. General 335
7.1 Geology of the Study Area 335

7.2 Physical Index Properties 336
7.3 Petrology and Chemical Studies 336
7.4 Strength Index Properties 337
7.4.1 Brazilian strength 338
7.4.2 Point Load Strength Index 338
7.4.3 Uniaxial Compressive Strength 338
7.4.4 Strength and Deformation Behaviour 339
7.4.5 Anisotropy 339
7.4.6 Effect of Thermal Treatment 339
7.5 Stress Dependent Permeability 340
7.5.1 Permeability 340
7.6 Strength and Deformation Behaviour of Oil Shale 342
7.7 Effect of Confinement on Deformation Behaviour 342
7.8 Prediction of Failure Strength for Oil/Gas Shales 342
7.9 Proposed Criterion for Thermally Treated Rocks 343
7.10 Shear Strength Parameters 343
7.11 Brittle – Ductile Transition 344
7.12 Future Scope of Work 344
REFERENCES 347
BRIEF BIO-DATA OF THE AUTHOR 391

LIST OF FIGURES

Figure No.	Description	Page No.
2.1	Major oil shale deposits in the world (Rajeshwar et al. 1979; Eseme et al. 2007; US Energy Information Administration)	9
2.2	Classification of oil shales adapted from Hutton (1987)	11
2.3	In situ conversion process (a) freeze wall concept (b) heating after fracturing (Wong et al. 2007)	16
2.4	Variation of uniaxial compressive strength with density for carbonate rocks from different locations (Madhubabu et al. 2016; Festa et al. 2018)	19
2.5	Variation of density with total organic carbon for oil shale rock (Ambrose 2014; Chen et al. 2015; Wang et al. 2015)	19
2.6	Variation of permeability with porosity for mudstone (Yang and Aplin, 2010)	21
2.7	Effect of thermal treatment temperature on the permeability of granite rock (He et al. 2018)	22
2.8	Possible variation of σc with β angle for inherent anisotropy	26
2.9	TGA–MS weight loss and derivative curves for the two higher heating rates (Tiwari and Deo, 2012)	29
2.10	TGA weight loss curve for different oil shales (Maaten et al. 2016)	30
2.11	Experimental arrangement adopted for tensile strength testing of anisotropic rocks by considering foliation plane angle (Ψ) and loading angle (β)	32
2.12	Tensile strength results in polar coordinates for (a) Postaer sandstone, and (b) Leubsdorfer gneiss (Dan et al. 2013)	32

2.13	Regression surface deduced from multiple regression analysis of tensile results for (a) Postaer sandstone, and (b) Leubsdorfer gneiss (Dan et al. 2013)	33
2.14	Different failure modes in tensile strength test (Tan et al. 2015; Chong et al. 2017; Ma et al. 2018)	35
2.15	Plot of maximum versus minimum Brazilian tensile strength for different anisotropic rocks (Ma et al. 2018)	36
2.16	Variation of tensile strength with % organic content for different oil shale (Chong et al. 1979, 82 and 84)	38
2.17	Variation of uniaxial compressive strength with anisotropic angle for different schist rock (Behrestaghi et al. 1996)	41
2.18	Variation of uniaxial compressive strength with organic content (Chong et al. 1976; Chong et al. 1979-80)	42
2.19	p-q Diagram for different rocks of oil yield (McLamore and Gray 1967; Hoshino et al. 1972; Chong et al. 1983; Zeuch 1983; Dusseault and Loftsson 1985; Ramamurthy et al. 1993)	44
2.20	Variation of stress ratio with orientation angle (β) for (a) martinsburg slate, (b) slate, (c) green river shale, and (d) fractured sandstone (Rao et al. 1986)	45
2.21	Variation of thermal conductivity with temperature (Rajeshwar 1979 and Alishaev et al. 2012)	47
2.22	Variation of differential stress with temperature at different confining pressure for oil shale (Zeuch, 1983)	50
2.23	Variation of differential stress with temperature for different rocks (Ibanez. and Kronenberg, 1993; Zeuch, 1983; Zhang et al. (2010); Zhao et al. 2012)	50
2.24	Variation of bulk density with thermal treatment temperature for carbonate rocks (Yavuz et al. 2010)	53

2.25 Change in sonic velocity (VP) related to initial velocity (VPI) at different thermal treatment temperature for carbonate rocks (Yavuz et al., 2010) 53

2.26 Variation of tensile strength with thermal treatment temperature at different loading angle (Roy and Singh 2016) 55

2.27 Variation of uniaxial compressive strength with temperature after air and water cooled granite (Shao et al. 2014). FG – fine grained, MG – medium grained, and CG – coarse grained 56

2.28 Stress-strain response of the thermally treated rock for (a) rapidly cooled specimen, and (b) slowly cooled specimen (Isaka et al. 2018) 57

2.29 Variation of (a) UCS, (b) Young's modulus, (c) strain at failure, and (d) Poisson's ratio and shear modulus with thermal treatment temperature (Isaka et al. 2018) 57

2.30 Stress-strain curves for marble under confinement after thermal treatment at (a) 20^{o}C, (b) 200 ^{o}C, (c) 400 ^{o}C, and (d) 600 ^{o}C (Yao et al. 2016; Peng et al. 2016) 59

2.31 Stress – strain curves of thermally treated granite at different confining pressure (a) unconfined, (b) 5 MPa, (c) 10 MPa, (d) 15 MPa (Zhang et al. 2018) 60

2.32 Deere and Miller's Classification of oil and gas shales from different locations 62

2.33 Mohr diagram showing possible modes of failure and fracturing 64

2.34 Fracture mechanisms in different regions of stress space (Franklin 1971) 74

2.35 Evaluation of parameters for a Ramamurthy et al. 1985 criterion for diffrerent anisotropic rocks (McLamore and Gray 1967; Hoshino et al. 1972; Chong et al. 1983; Zeuch 1983; Dusseault and Loftsson 1985; Ramamurthy et al. 1993) 76

3.1	Resource Assessment for Shale Gas and Oil in India (Geological Survey of India)	82
3.2	Depth of black shale of Cambay basin, India (EIA/ARI)	85
3.3	North-South geological cross section across Cambay basin (Sudhakar and Basu 1973)	85
3.4	Structural and tectonic map of Krishna-Godavari Basin (Bastia et al. 2007)	87
3.5	Geological Cross section of Krishna-Godavari Basin (Bastia et al. 2007)	88
3.6	Prospective area of Cauvery basin (EIA/ARI)	90
3.7	Geological Section showing the discovered hydrocarbon pools in Cretaceous sediments of South Cauvery region (Sastri et al. 1981)	91
3.8	Damodar valley basin for shale gas and oil exploration (EIA/ARI)	92
3.9	Cross section of Raniganj sub basin of Damodar valley (Ghosh 2002)	93
3.10	Map of North-East states of India and Assam – Arakan basin (NDR)	97
3.11	Tectonic and geological age map of North - East India (Nandy 2000)	98
3.12	Tectonic and stratigraphic map of Assam and adjoining areas (Gupta and Biswas 2000)	99
3.13	Oil fields of the Upper Assam shelf (NDR)	100
3.14	Map of Tinsukia district of Upper Assam Region (Srivastava et al. 2013)	102
3.15	Oil Shale samples collection location (Srivastava et al. 2013)	103
3.16	Tikak colliery (a) geological map and (b) section along A-A’	107
3.17	Site for sample collection (a) Tikak parbat and (b) blasted material	108
3.18	Coring procedure performed in the laboratory (a) shale blocks, (b) wet coring (c) disintegrated block due to water, (d) dry coring, (e) cored samples, and (f) cube cutting	112

3.19	In situ coring of the specimen (a) Shale blocks, (b) block selected for coring (c) portable coring equipment, (d) wet coring, (e) cored sample, and (f) preserved samples	113
3.20	Preservation of cored samples (a) paraffin wax coating and (b) core box	114
3.21	Portable core cutting and grinding equipment (a) front view (b) isometric view (c) cutting machine and (d) clamping system	115
3.22	Flowchart of experimental program on oil shale for varying orientation (β) of the foliation plane with respect to the direction of major principal stress σ_1	118
3.23	Specimens prepared for the thermal treatment	119
3.24	Thermally treated specimens at different temperatures	119
3.25	Specimen of L/D = 0.5 at different anisotropic angles (Ψ) for Brazilian test	120
3.26	Testing program for Brazilian strength test	120
3.27	Specimen of L/D = 1.0 at different anisotropic angles point load test	121
3.28	Testing program for point load strength test (a) axial and (b) diametral	121
3.29	Test program for uniaxial compression test	124
3.30	Specimens prepared for the UCS and triaxial testing (a) anisotropy variation and (b) heat treated	125
3.31	Test program for Triaxial test	126
3.32	Complete triaxial setup for UCS and triaxial testing (a) loading frame, (b) data accusation system, (c) confinement unit, and (d) triaxial cell	127
3.33	Schematic diagram of high pressure triaxial cell	128
3.34	Complete view of (a) triaxial cell, (b) specimen placed, (c) confinement unit	129
3.35	Loading platens designed and fabricated at IIT Delhi	130

3.36 Axial and diametral strain measurement unit 130

3.37 Schematic diagram of permeability test apparatus (Rao 1984) 131

3.38 Apparatus for permeability testing of rocks 132

3.39 Density of the oven dried specimens 133

3.40 Specific gravity test for (a) mudstone, (b) brown shale, (c) black shale, (d) green shale, and (e) oil shale 134

3.41 Variation of saturated water content and porosity with anisotropic angle 134

3.42 Variation of sonic wave velocity (V_p) with anisotropic angle 135

3.43 Variation of density with thermal treatment 136

3.44 Variation of density with anisotropy angle for different temperatures 137

3.45 Variation of sonic velocity (V_p) with anisotropy angle after thermal treatment at different temperatures 137

4.1 Procedure for thin section preparation (a), (b) rock sample, (c) grinding disc, (d) finished samples fixed to glass slide using epoxy, (e) thin layer of rock after cutting, (f) lapping with coarse grid, (g) lapping with 400 grit size, (h) and (i) thin section 142

4.2 Thin Section of (a) mudstone, (b) coal, (c) brown shale(d) green shale, (e) oil shale, (f) across lamination, and (g) along lamination 143

4.3 Thin Section images of mudstone at (a) location 1 and (b) location 2 145

4.4 Thin Section images of coal at (a) location 1 and (b) location 2 146

4.5 Thin Section images of brown shale at (a) location 1 and (b) location 2 147

4.6 Thin Section images of green shale at (a) location 1 and (b) location 2 148

4.7 Photomicrograph of oil shale samples (a) across lamination, and (b) along lamination (location 1) 149

4.8 Photomicrograph of oil shale samples (a) magnified at 50X, and (b) location 2 150

4.9 Photomicrograph of oil shale samples (a) magnified at 50X under plane polarised, and (b) cross polarised 151

4.10 Complete set up of XRD apparatus (Ringaku, Min flax600) used for the analysis 153

4.11 X-Ray diffraction pattern of mudstone (red), shale (green), and coal (black) 154

4.12 SEM apparatus available at Central Research Facility, Indian Institute of Technology Delhi 159

4.13 Finished specimen of different rocks are mounted on disc for SEM analysis 160

4.14 SEM images of mudstone at magnification 1000X showing (a) grain boundry (GB), and (b) organic void (OV) 162

4.15 SEM images of mudstone at magnification 5000X showing (a) voids (V), and (b) ouartz grain 163

4.16 SEM images of coal at magnification (a) 81X and (b) 5000X 164

4.17 SEM images of brown shale at magnification (a) 1000X and (b) 5000X 165

4.18 SEM images of green shale at magnification (a) 1000X and (b) 5000X 166

4.19 SEM images of oil shale at magnification (a) 2500X and (b) 5000X 167

4.20 SEM images across lamination at magnification (a) 2500X and (b) 5000X 168

4.21 SEM images along lamination at magnification (a) 2500X and (b) 5000X 169

4.22 SEM images of oil shale at magnification (a) 7000X and (b) 25000X 170

4.23 Pore size distribution for (a) mudstone, (b) oil shale along lamination, and (c) across lamination 173

4.24 Pore area distribution for (a) mudstone, (b) oil shale along lamination, and (c) across lamination 175

4.25 Pore aspect ratio distribution for (a) mudstone, (b) oil shale along lamination, and (c) across lamination 176

4.26 Transmission Electron Microscope (TEM) facility at DMSE, IIT Delhi 177

4.27 Clay minerals in oil shale observed under TEM (a) illite, and (b) kaolinite 178

4.28 Oil shale containing quartz silt observed under TEM 178

4.29 Pyrite mineral in oil shale observed under TEM (a) 200nm, and (b) 400nm 179

4.30 Calcite in oil shale observed under TEM (a) calcite nanoparticles, and (b) vaterite 179

4.31 Area scanned on the sample for EDX analysis 181

4.32 Working principal of CHNS analyzer 183

4.33 Thermal gravimetric analyzer facility of Chemical Engineering Department, Indian Institute of Technology Delhi 186

4.34 TGA-MS weight loss and derivative of weight loss curve for oil shales, mudstone and coal from the Upper Assam region 187

4.35 Free swell index test of oil shale rock 190

4.36 Texture change after thermal treatment (a) front view, and (b) top view 192

4.37 Weight loss in oil shale with temperature for different anisotropic angles 193

4.38 XRD patterns for oil shale after thermal treatment at 100, 300, 450, 600, and 800 ^oC 196

4.39 XRD scans for thermally treated oil shale at different 2θ intervals (a) 8 to 26^o, (b) 26 to 28^o, and (c) 26 to 70^o 199

4.40 SEM images of oil shale at 2500X magnification after thermal treatment at 100 ^oC temperature 201

4.41 SEM images of oil shale at 2500X magnification after thermal treatment at 300 ^oC temperature 202

4.42	SEM images of oil shale at 2000X magnification after thermal treatment at 450 ^{o}C temperature	202
4.43	SEM images of oil shale at 5000X magnification after thermal treatment at 450 ^{o}C temperature	203
4.44	SEM images of oil shale at 5000X magnification after thermal treatment at 600 ^{o}C temperature (location 1)	203
4.45	SEM images of oil shale at 5000X magnification after thermal treatment at 600 ^{o}C temperature (location 2)	204
4.46	SEM images of oil shale at 5000X magnification after thermal treatment at 800 ^{o}C temperature (W)	204
4.47	SEM images of oil shale at 5000X magnification after thermal treatment at 800 ^{o}C temperature (R)	205
4.48	Ternary diagram showing comparison of the mineral composition of several oil/gas-bearing shales	207
5.1	Experimental arrangement for Brazilian testing of rock anisotropy relative to foliation angle (Ψ) and loading angle (β). Where P is applied load, Z is axis along the sample length, and fp is foliation plane	210
5.2	Variation of tensile strength with foliation loading angle for different foliation angle	211
5.3	Variation of tensile strength with foliation angle for different loading angle	211
5.4	Average tensile strength variation of the oil shale in polar diagram	212
5.5	Failure patterns exhibited by oil shale under Brazilian test mode at different foliation angle (ψ) and loading angle (β)	213
5.6	Variation of diametral and axial point load strength index with Ψ for different β	216

5.7	Failure patterns exhibited by oil shale under diametral and axial point load tests at different Ψ and β	217
5.8	Variation of unconfined compressive strength (UCS) of oil shale with β after thermal treatment	220
5.9	Stress – strain curves at different β for oil shale after 100 ^{o}C heating	221
5.10	Stress – strain curves at different β for oil shale after 300 ^{o}C heating	221
5.11	Stress – strain curves at different β for oil shale after 450 ^{o}C heating	222
5.12	Stress – strain curves at different β for oil shale after 600 ^{o}C heating	222
5.13	Stress – strain curves of thermally treated oil shale at β = 0^{o}	223
5.14	Stress – strain curves of thermally treated oil shale at β = 90^{o}	223
5.15	Variation of Young's modulus with β for oil shale after thermal treatment	225
5.16	Variation of Poisson's ratio with β for oil shale after thermal treatment	225
5.17	Variation of axial strain with β for oil shale after thermal treatment	226
5.18	Variation of diametral strain with β for oil shale after thermal treatment	226
5.19	Deere and Miller's classification for rock tested and comparison with oil and gas shale	228
5.20	Variation of thermal degradation ratio with thermal treatment temperature at different β	232
5.21	Failure patterns of oil shale after thermal treatment at 100 ^{o}C under unconfined compressive strength test at different anisotropy angles	234
5.22	Failure patterns of oil shale after thermal treatment at 300 ^{o}C under unconfined compressive strength test at different anisotropy angles	234
5.23	Failure patterns of oil shale after thermal treatment at 450 ^{o}C under unconfined compressive strength test at different anisotropy angles	235

5.24 Failure patterns of oil shale after thermal treatment at 600 ^{o}C under unconfined compressive strength test at different anisotropy angles 235

5.25 Variation of axial and radial strain with mean stress 239

5.26 Variation of volumetric strain with mean stress (hydrostatic pressure) 240

5.27 Variation of bulk compressibility (C_{bc}) with mean stress for oil shale from upper Assam region (Barail shale) 240

5.28 Variation of bulk compressibility (C_{bc}) with anisotropic angle (β) at different mean stress (hydrostatic condition) 241

5.29 Variation of bulk compressibility (C_{bc}) with mean stress for oil/gas shale from different locations 241

5.30 Variation of effective porosity with anisotropy angle (β) 244

5.31 Variation of hydraulic conductivity with effective stress for different β angle 246

5.32 Variation of permeability with effective stress for different β angle 246

5.33 Exponential fit for stress-dependent permeability at different β angle and effective stress range (a) 1.38 – 11.38 MPa, (b) 0.34 – 1.38 MPa, and (c) 3.10 – 11.38 MPa 252

5.34 Power law fit for stress-dependent permeability at a different β angle 253

5.35 Variation of different parameters of exponential model and power law with β angle for oil shale rock 254

6.1 Variation of experimental and predicted σc with β angle of oil shale rock after thermal treatment at 100 ^{o}C, 300 ^{o}C, 450 ^{o}C and 600 ^{o}C 260

6.2 Stress-Strain behaviour of oil shale after thermal treatment at 100^{o}C under different σ_3 for $\beta = 0^o$ 262

6.3 Stress-Strain behaviour of oil shale rock after thermal treatment at 100^{o}C under different σ_3 for $\beta = 15^o$ 262

6.4 Stress-Strain behaviour of oil shale rock after thermal treatment at 100^{o}C under different σ_3 for $\beta = 30^o$ 263

6.5 Stress-Strain behaviour of oil shale rock after thermal treatment at 100^{o}C under different σ_3 for $\beta = 45^o$ 263

6.6 Stress-Strain behaviour of oil shale rock after thermal treatment at 100^{o}C under different σ_3 for $\beta = 60^o$ 264

6.7 Stress-Strain behaviour of oil shale rock after thermal treatment at 100^{o}C under different σ_3 for $\beta = 90^o$ 264

6.8 Stress-Strain behaviour of oil shale rock after thermal treatment at 450^{o}C under different σ_3 for $\beta = 0^o$ 265

6.9 Stress-Strain behaviour of oil shale rock after thermal treatment at 450^{o}C under different σ_3 for $\beta = 30^o$ 265

6.10 Stress-Strain behaviour of oil shale rock after thermal treatment at 450^{o}C under different σ_3 for $\beta = 45^o$ 266

6.11 Stress-Strain behaviour of oil shale rock after thermal treatment at 450^{o}C under different σ_3 for $\beta = 60^o$ 266

6.12 Stress-Strain behaviour of oil shale rock after thermal treatment at 450^{o}C under different σ_3 for $\beta = 90^o$ 267

6.13 Stress-Strain behaviour of oil shale rock after thermal treatment at 600^{o}C under different σ_3 for $\beta = 0^o$ 267

6.14 Stress-Strain behaviour of oil shale rock after thermal treatment at 600^{o}C under different σ_3 for $\beta = 30^o$ 268

6.15 Stress-Strain behaviour of oil shale rock after thermal treatment at 600^{o}C under different σ_3 for $\beta = 45^o$ 268

6.16 Stress-Strain behaviour of oil shale rock after thermal treatment at 600^{o}C under different σ_3 for $\beta = 60^o$ 269

6.17 Stress-Strain behaviour of oil shale rock after thermal treatment at 600°C under different σ_3 for $\beta = 90^o$ 269

6.18 Variation of Young's modulus with anisotropy angle β, for thermally treated oil shale rock at 100°C 271

6.19 Variation of Young's modulus with anisotropy angle β, for thermally treated oil shale rock at 450°C 271

6.20 Variation of Young's modulus with anisotropy angle β, for thermally treated oil shale rock at 600°C 272

6.21 Variation of E_t/σ_3 with β angle for oil shale after thermal treatment at 100°C 273

6.22 Variation of E_t/σ_3 with β angle for oil shale after thermal treatment at 450°C 273

6.23 Variation of E_t/σ_3 with β angle for oil shale after thermal treatment at 600°C 274

6.24 Variation of E_t/σ_3 with σ_c/σ_3 for oil shale after thermal treatment at 100°C 276

6.25 Variation of E_t/σ_3 with σ_c/σ_3 for oil shale after thermal treatment at 450°C 276

6.26 Variation of E_t/σ_3 with σ_c/σ_3 for oil shale after thermal treatment at 600°C 277

6.27 Comparison of experimental and predicted values of Young's modulus for oil shale rock, thermally treated at 100°C 277

6.28 Comparison of experimental and predicted values of Young's modulus for oil shale rock, thermally treated at 450°C 278

6.29 Comparison of experimental and predicted values of Young's modulus for oil shale rock, thermally treated at 600°C 278

6.30 Variation of Poisson's ratio with β angle under different confinement pressure for oil shale after thermal treatment at 100°C 280

6.31 Variation of Poisson's ratio with β angle under different confinement pressure for oil shale after thermal treatment at 450°C 281

6.32 Variation of Poisson's ratio with β angle under different confinement pressure for oil shale after thermal treatment at 600°C 281

6.33 Variation of failure strain in axial direction with β angle under different confining pressures for 100 °C treatment temperature 283

6.34 Variation of failure strain in axial direction with β angle under different confining pressures for 450 °C treatment temperature 284

6.35 Variation of failure strain in axial direction with β angle under different confining pressures for 600 °C treatment temperature 284

6.36 Variation of failure strain in diametral direction with β angle under different confining pressures for 100 °C treatment temperature 285

6.37 Variation of failure strain in diametral direction with β angle under different confining pressures for 450 °C treatment temperature 285

6.38 Variation of failure strain in diametral direction with β angle under different confining pressures for 600 °C treatment temperature 286

6.39 Variation of σ_1 with σ_3 at different β angle for 100 °C thermal treatment 288

6.40 Variation of σ_1 with σ_3 at different β angle for 450 °C thermal treatment 288

6.41 Variation of σ_1 with σ_3 at different β angle for 600 °C thermal treatment 289

6.42 Variation of σ_1 with β under different σ_3 for thermal treatment at 100 °C 289

6.43 Variation of σ_1 with β under different σ_3 for thermal treatment at 450 °C 290

6.44 Variation of σ_1 with β under different σ_3 for thermal treatment at 600 °C 290

6.45 Variation of stress ratio with β for oil shale rock, thermally treated at 100°C 293

6.46 Variation of stress ratio with β for oil shale rock, thermally treated at 450°C 293

6.47 Variation of stress ratio with β for oil shale rock, thermally treated at 600°C 294

6.48 Plots between $(\sigma_1 - \sigma_3)/\sigma_3$ and σ_{cj}/σ_3 for the thermally treated oil shale at 100 °C 296

6.49 Plots between $(\sigma_1 - \sigma_3)/\sigma_3$ and σ_{cj}/σ_3 for the thermally treated oil shale at 450 °C 297

6.50 Plots between $(\sigma_1 - \sigma_3)/\sigma_3$ and σ_{cj}/σ_3 for the thermally treated oil shale at 600 °C 297

6.51 Comparison between observed and predicted failure strength of oil shale after thermal treatment at 100 °C 299

6.52 Comparison between observed and predicted failure strength of oil shale after thermal treatment at 450 °C 299

6.53 Comparison between observed and predicted failure strength of oil shale after thermal treatment at 600 °C 300

6.54 Comparison between experimental and predicted failure strength for 100 °C case by using IIT Delhi criterion 301

6.55 Variation of k_β with anisotropy angle β for thermally treated oil shale rock 303

6.56 Comparison of experimental failure strength value with predicted values using modified Hoek-Brown criterion for 100 °C 304

6.57 Comparison of experimental failure strength value with predicted values using modified Hoek-Brown criterion for 450 °C 305

6.58 Comparison of experimental failure strength value with predicted values using modified Hoek-Brown criterion for 600 °C 305

6.59 Comparison of experimental and predicted failure strength using Eq. (6.11) for (a) 450 °C, and (b) 600 °C 308

6.60 Comparison of experimental and predicted failure strength using Eq. (6.12) for (a) 450 °C, and (b) 600 °C 309

6.61 Mohr plots for oil shale rock, thermally treated at 100 °C at $\beta = 0^o$ 312

6.62 Mohr plots for oil shale rock, thermally treated at 100 °C at $\beta = 30^o$ 312

6.63 Mohr plots for oil shale rock, thermally treated at 100 °C at $\beta = 45^o$ 313

6.64 Mohr plots for oil shale rock, thermally treated at 100 °C at β = 60° 313

6.65 Mohr plots for oil shale rock, thermally treated at 100 °C at β = 90° 314

6.66 Mohr plots for oil shale rock, thermally treated at 450 °C at β = 0° 314

6.67 Mohr plots for oil shale rock, thermally treated at 450 °C at β = 30° 315

6.68 Mohr plots for oil shale rock, thermally treated at 450 °C at β = 45° 315

6.69 Mohr plots for oil shale rock, thermally treated at 450 °C at β = 60° 316

6.70 Mohr plots for oil shale rock, thermally treated at 450 °C at β = 90° 316

6.71 Mohr plots for oil shale rock, thermally treated at 600 °C at β = 0° 317

6.72 Mohr plots for oil shale rock, thermally treated at 600 °C at β = 30° 317

6.73 Mohr plots for oil shale rock, thermally treated at 600 °C at β = 45° 318

6.74 Mohr plots for oil shale rock, thermally treated at 600 °C at β = 60° 318

6.75 Mohr plots for oil shale rock, thermally treated at 600 °C at β = 90° 319

6.76 p – q plot at different β for thermally treated oil shale rock at 100 °C 319

6.77 p – q plot at different β for thermally treated oil shale rock at 450 °C 320

6.78 p – q plot at different β for thermally treated oil shale rock at 600 °C 320

6.79 Variation of shear strength parameters with β angle for thermally treated oil shale (a) cohesion, c, and (b) friction angle, φ 321

6.80 Brittle – ductile transition for thermal treatment oil shale rock at 100 °C 325

6.81 Brittle – ductile transition for thermal treatment oil shale rock at 450 °C 325

6.82 Brittle – ductile transition for thermal treatment oil shale rock at 600 °C 326

6.83 Failure pattern of oil shale rock at different β angles after thermally treated at 100 °C under confining pressure of 5 MPa 327

6.84 Failure pattern of oil shale rock at different β angles after thermally treated at 100 ^{o}C under confining pressure of 10 MPa 327

6.85 Failure pattern of oil shale rock at different β angles after thermally treated at 100 ^{o}C under confining pressure of 15 MPa 327

6.86 Failure pattern of oil shale rock at different β angles after thermally treated at 100 ^{o}C under confining pressure of 20 MPa 328

6.87 Failure pattern of oil shale rock at different β angles after thermally treated at 100 ^{o}C under confining pressure of 30 MPa 328

6.88 Failure pattern of oil shale rock at different β angles after thermally treated at 450 ^{o}C under confining pressure of (a) 2, (b) 5, and (c) 10 MPa 329

6.89 Failure pattern of oil shale rock at different β angles after thermally treated at 600 ^{o}C under confining pressure of (a) 2, (b) 5, and (c) 10 MPa 330

6.90 Variation of R_{cT} with thermal treatment temperature for different rocks 332

LIST OF TABLES

Table No.	Description	Page No.
2.1	Major oil shale deposits in the world (Rajeshwar et al. 1979; Eseme et al. 2007)	7
2.2	Different correlations proposed for the stress dependent permeability	23
2.3	Brazilian tensile strength and anisotropy index for anisotropic rocks (Behrestaghi et al. 1996; Ma et al. 2018)	37
2.4	Strength anisotropy of different rocks	40
2.5	Classification of rock on the basis of anisotropy ratio (Ramamurthy et al. 1993)	41
2.6	Comparison of mechanical properties of oil shales with other anisotropic rocks (Eseme et al. 2007; Nasseri et al. 2003; Ramamurthy et al.1993; Behrestaghi et al.1996)	43
2.7	Mechanical properties of two grades of oil shales (Clossmann and Bradley 1977; Chu and Chang, 1980; Means, 1990)	49
2.8	Table 2.8 Values of parameter B for different rocks (Rao, 1984; Ramamurthy et al., 1985)	67
2.9	Comparison of physical, mechanical properties and mineralogy of oil shales with other anisotropic rocks (Eseme et al. 2007; Nasseri et al. 2003; Ramamurthy et al. 1993; Behrestaghi et al.1996; McLamore and Gray 1967)	77
2.10	Observed values of parameters of Ramamurthy et al. (1985) criterion for different rocks (McLamore and Gray 1967; Hoshino et al. 1972; Chong et al. 1983; Zeuch, 1983; Dusseault and Loftsson 1985; Ramamurthy et al. 1993)	78

3.1	Resource Potential of Different Basins for Shale Gas/Oil Extraction	83
3.2	The Stratigraphic succession in the Makum coalfield (Srivastava et al. 2013)	104
3.3	Final drilling length for different drilling angles	111
3.4	Tests and test procedures	117
3.5	Physical properties of oil shale rock	138
4.1	Quantitative mineral composition of oil shale, mudstone, and coal	157
4.2	Essential specifications of EVO50 apparatus (CRF, I.I.T. Delhi)	159
4.3	Elemental analysis of oil shale sample from upper Assam region	181
4.4	Results of CHNS Analysis of oil shale, brown shale, mudstone and coal	183
4.5	Characterization of soil or rock by the free swell index	189
4.6	Quantitative analysis of thermally treated oil shale rock	197
4.7	The total composition of oil shale rock	200
5.1	Brazilian strength value of oil shale at different foliation and loading angles	210
5.2	Axial and diametral point load strength of oil shale for different Ψ and β	216
5.3	Unconfined compressive strength (UCS) of oil shale at different β after thermal treatment	219
5.4	Anisotropy ratio for different rocks	230
5.5	Anisotropy ratio from engineering properties of oil shale	231
5.6	Thermal Degradation Ratio of Thermally Treated Oil Shale	232

5.7	Calculated values of bulk compressibility for different β angles	242
5.8	Values of hydraulic conductivity and permeability of Indian oil shale rock	247
5.9	Parameters determined using curve fitting method for the exponential model and power law of stress-dependent permeability	253
6.1	Observed and calculated parameters of the Young's modulus prediction model	279
6.2	Failure strength of thermally treated oil shale rock	291
6.3	Parameters evaluated for observed and calculated values for oil shale rock	298
6.4	Observed and calculated strength parameters of IIT Delhi criterion	301
6.5	Observed parameters of modified Hoek-Brown criterion for oil shale rock	303
6.6	Values of coefficient of determination (r2) for Eq. (6.11) and (6.12)	310
6.7	Observed shear strength parameters at different β angles	322
6.8	Observed values of the parameters of Ramamurthy et al. (1985) criterion for different rocks	333

LIST OF NOTATIONS

A	material constant for oil shale
A’	empirical constant for rock type
a_o	half-aperture at reference stress p_o
a	material constant for permeability
b	material constant for permeability
B	material constant
$B_{i,j}$	material constant depends on material response to stress state
B_j	material constant at β angle
CHNS	carbon, hydrogrn, nitrogrn, sulpher
c	cohesive strength
c_{i0}	cohesion at low confining stress
c_w	cohesion at plane of weakness
$c_{\beta 0}$	cohesion at orientation angle β
C_{bc}	bulk compressibility
$\bar{C}_{pc}$	pore compressibility of rock
D	constants in Donath (1961) approach
E_{t50}	deformational modulus at 50% failure strength
EDX	energy dispersive x-ray microanalysis
fp	foliation plane
g, h	constant for power law model
k_β	parameter describing the anisotropy effect
k	permeability at effective stress p
k(P)	permeability at effective stress P
K	hydraulic conductivity
k_o	fluid conductance reference stress p_o
M	Fischer assay oil yield in U.S. gallons per ton
m	material constant in Hoek and Brown criterion for intact rocks
m, n	constant for exponential model
O_c	organic content
OY	oil yield from oil shale
p	material constant ranges between 1.2 to 1.8 for clay and clay-rich rocks

r^2	coefficient of determination
R_c	anisotropy strength ratio
R_{cT}	thermal degradation ratio
S	material constant in Hoek and Brown criterion for intact rock
SEM	scanning electron microscope
T	temperature
TGA	thermo gravimetric analysis
TEM	transmission electron microscopy
TOC	total organic carbon
UCS	unconfined compressive strength
V_d	volume of the soil specimen in graduated cylinder filled with water
V_k	volume of the soil specimen in graduated cylinder containing kerosene.
V_p	sonic wave velocity
V_{PI}	initial sonic velocity
W_o	organic content in weight percentage
W_t	% weight loss due to retorting processes
XRD	X-ray diffraction
X	material constant
P	dry density
$\Delta\sigma$	change in effective stress
σ_o	initial stress state
β	orintation angle between major principal stress and foliation plane
β_m	orientation angle corresponding to the minimum strength or cohesion
Ψ	foliation plane angle
φ	angle of internal friction
φ_{i0}	friction angle at low confining stress
φ_w	internal friction angle at plane of weakness
$\varphi_{\beta 0}$	friction angle at orientation angle β
σ_c	uniaxial compressive strength
σ_{ci}	uniaxial compressive strength of intact rock
σ_{cmax}	maximum uniaxial compressive strength
σ_t	Brazilian tensile strength
σ_{ta}	axial point load strength

σ_{td}	diametral point load strength
σ_{tmax}	maximum tensile strength
σ_{tmin}	minimum tensile strength
σ_1	maximum failure stress (major principal stress)
σ_3	confinment stress (minoe principal stress)
σ_{cj}	uniaxial compressive strength at β angle
σ_{c90}	compressive strength at $\beta=90^o$
$\sigma_{c\beta}$	uniaxial compressive strength at β angle
$\sigma_{c\beta T}$	maximum uniaxial compressive strength at β angle for T ^{o}C temperature
σ_{crt}	critical confining pressure for rocks.
τ	shear stress
σ_n	normal stress
α	the slope of the line plotted in log – log scale
α_j	the slope of the line plotted in log – log scale at β angle
ε_a	axial strain
ε_d	diametrical strain
ε_v	volumetric strain
I_{s50}	point load strength
ρ	density
υ	Poisson’s ratio
μ_β	porosity (%) at that β angle
μ_0	porosity at 0^o β angle
μ	dynamic viscosity of the fluid
ϕ	porosity