

## Extended Abstract

(For Ph.D. Open Seminar)

### Multiscale Characterization of Anisotropic Rocks with Emphasis on Phyllite and Shale from the Lesser Himalaya, Uttarakhand, India

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The Himalayas make a significant contribution to the growth of the country's economy. The growing infrastructure development and the exploration of natural resources within the highly complex Himalayan terrain require an in-depth characterization of the rock masses. The detailed characterization in terms of physico-mechanical properties, geochemical behavior, morphological parameters, and pore characteristics is very critical for understanding the behavior of those complex and highly anisotropic formations. In accordance with this, the research presented in this thesis has been undertaken. The thesis is structured into nine chapters, as outlined below:

### **Chapter 1. Introduction**

This chapter outlines the definition of the problem along with a detailed literature survey in the context of the Himalayas, followed by the identified research gaps and the proposed research objectives. Two different rock types, i.e., Chandpur phyllite and Krol shale from the Lesser Himalayan region, Uttarakhand, have been taken into consideration. The Chandpur phyllite in and around the Srinagar region of Uttarakhand is a dominant lithology for the major infrastructure developments, including dams, tunnels, highways, etc., despite its strong anisotropy and highly fragile nature. The Krol shale from the Lesser Himalaya has a wide geographical distribution and a carbonaceous nature, yet this formation remains completely unexplored.

### **Chapter 2. Selection and Collection of Rock Samples**

This chapter represents the study area for this thesis, which is located in Uttarakhand, India, where the Chandpur Group phyllite and the Krol Formation shale are well-exposed and easily accessible. Both rock units belong to the Lesser Himalayan domain, which is well known for its geological complexity. Geographically, the region lies within an active tectonic belt of the Himalaya, characterized by structural features such as folding, faulting, and thrusting. The area exhibits considerable lithological diversity with varying degrees of deformation, reflecting its prolonged tectonic history.

### **Chapter 3. Multiscale Weathering Grade Classification of Chandpur Phyllite: Insights from In-situ Observation and Laboratory Investigation**

This chapter investigates the multiscale weathering grade classification of Chandpur phyllite by combining in situ observations with laboratory analyses. The results indicate the progressive

deterioration of the rock strength as weathering progresses from W1 to W4. The deterioration of the primary minerals into secondary clay minerals with progressive weathering plays an important role in altering the rock's mechanical behavior. The surface characteristics, including wettability, roughness, and frictional behavior, also change gradually with progressive weathering grades. This multiscale analysis provides a basis for understanding the weathering phenomena crucial for infrastructure development within Chandpur phyllite.

#### **Chapter 4. Impact of Water Saturation on Physico-mechanical Properties of Fragile Chandpur Phyllite at Varying Foliation Angles**

In this chapter, a study on the influence of water saturation on the physico-mechanical behavior of Chandpur phyllite has been carried out. A combined effect of water saturation and foliation angle ( $\beta$ ) has been considered. Although saturation leads to a reduction in the overall rock strength, it does not affect the failure modes, which are controlled by the micaceous foliations. The U-shaped pattern (initially decreasing and then increasing with  $\beta$  angle, having a minimum value at  $\beta=30^\circ$  and a maximum at  $\beta=0^\circ/90^\circ$ ) of the mechanical parameters under both normal and saturation conditions, shows the inherent anisotropic nature of Chandpur phyllite. However, the wave velocity did not follow a similar U-shaped pattern, but it decreased with increasing  $\beta$  angle under both normal and saturated conditions, although values are higher under saturated conditions. The findings of this study are crucial, as the effect of both anisotropy and water saturation should be considered for the design and stability of infrastructures.

#### **Chapter 5. Influence of Specimen Geometry on the Point Load Strength Anisotropy of Phyllite**

This chapter investigates the influence of specimen geometry (ratio of length to diameter,  $t/d$ ) on the axial point load strength index of Chandpur phyllite. Specimens with  $t/d$  ratios of 1, 0.8, and 0.6 across  $\beta$  angles from  $0^\circ$  to  $90^\circ$  were prepared for investigation. The results indicate that for  $t/d$  ratios of 0.8 and 1, the point load strength index  $I_{S(50)}$  increases from  $0^\circ$  to  $60^\circ$  and then decreases at higher  $\beta$  angles ( $75^\circ$  and  $90^\circ$ ). In contrast, for a  $t/d$  ratio of 0.6,  $I_{S(50)}$  continuously increases with increasing  $\beta$  angle. Up to  $\beta = 30^\circ$ , all specimens show valid failure modes along the foliation. However, at higher  $\beta$  angles ( $\geq 45^\circ$ ), specimens with  $t/d$  ratios of 0.8 and 1 exhibit invalid failure due to uneven stress distribution, while those with a  $t/d$  ratio of 0.6 maintain valid failure modes.

Statistical validation using the Student's t-test further confirms that the strength indices differ significantly among the geometries, with the t/d ratio of 0.6 showing reliable strength values. Overall, this study provides the role of specimen geometry in point load testing and suggests that a t/d ratio of 0.6 is the most suitable for obtaining accurate strength in phyllite rock masses.

## **Chapter 6. Multiscale Mechanical Characterization of Krol Shale using Brazilian Tensile Strength and Nano-indentation Tests: Implications in Hydrocarbon Recovery and CO<sub>2</sub> Sequestration**

In this chapter, a study on the multiscale mechanical characterization of the Krol shale by combining the macroscopic Brazilian tensile strength test and the microscopic nano-indentation test has been carried out. The nature of the studied shale indicates that it has strong potential for gas generation, is rich in clay minerals, and was deposited in a marine environment containing terrestrial organic matter. Mechanical parameters indicate low-to-moderate stiffness and a semi-ductile nature, supported by low values of elastic modulus and brittleness index obtained from nano-indentation and BTS tests. Features like pop-in events and elbow events in the load-displacement curve of the nano-indentation test are associated with pores/heterogeneity and phase change during unloading. Nano-indentation tests highlight the microscale mechanical heterogeneity and tectonic deformation mechanism within the Krol shale, which overall controls its macroscopic tensile strength. The relatively low BTS values and observed layer-controlled failure along bedding planes further confirm the nature of the shale. Further, a probabilistic distribution approach successfully links microscopic and macroscopic mechanical properties, showing good agreement with experimental results. The findings of this study are very important for assessing the suitability of the Krol shale for hydraulic fracturing to enhance gas recovery and for long-term CO<sub>2</sub> storage.

## **Chapter 7. Pore System Characterization of Untapped Himalayan Shale Using an Integrated Multi-analytical Approach: Bridging Nano-to Macro Scale Investigation**

This chapter investigates the pore characterization of the Krol shale using various multi-analytical techniques such as LPGA, SAXS, FE-SEM, and micro-CT. This multiscale pore characterization bridges the gap between nano- and macro-scale pores. The geochemical results indicate that the pyrite-rich black Krol shale contains Type III kerogen (gas-prone). This is supported by high

vitrite reflectance ( $>1.5\%$ ), absence of liptinite and inertinite, and high sulfur & TOC content, indicating strong gas-generation potential. The pore structure mainly consists of irregular, elongated slit- and wedge-shaped pores, as shown by LPGA isotherms and SEM images. Pore size distribution (PSD) indicates the dominance of micro- and mesopores, which overall controls the storage capacity. The fine micropores were identified through LPGA-CO<sub>2</sub> adsorption and mesopores through LPGA-N<sub>2</sub> adsorption and SAXS analysis. Micro-CT analysis suggests that pore connectivity is low to moderate having connected porosity smaller than the total porosity. Micro-CT imaging also reveals the presence of natural microfractures, with most of the connected porosity mainly concentrated along those fracture zones. Overall, this study demonstrates that despite being largely unexplored, the Krol shale possesses a complex pore network with significant potential for gas storage and flow.

### **Chapter 8. Multiscale Characterization of Pore Evolution in Lesser Himalayan Black Shale from Krol Formation under Oxidic Thermal Heating: Relevance to Untapped Shale Gas Extraction**

In this chapter, the study focuses on the evolution of the pore structure of the Krol shale during combustion to enhance untapped gas extraction. At around 100°C, pore compactness results from moisture removal, but at higher temperatures ( $>100^\circ\text{C}$ ), combustion leads to pore expansion, coalescence, and the creation of new pores due to thermal heating and organic matter breakdown. This leads to increased surface irregularity, as supported by the LPGA fractal dimension and SEM images. With increasing temperature, pore size distribution also changes, shifting from micro- and mesopores at lower temperatures to larger mesopores and macropores at higher temperatures, with a sharp rise in pore development around 400°C, confirmed by LPGA and SAXS results. This study shows the potential of combustion-induced thermal stimulation and suggests that oxidic heating can significantly alter shale microstructure, thereby offering a viable technique for shale gas recovery.

### **Chapter 9. Conclusion and Future Scope**

This chapter includes the Key findings of the research work carried out in the thesis, followed by the recommendations for future studies that can help in strengthening the findings of the current research.

## Publications from the thesis in Referred Journal

- Singh, D.,** Singh, H. K., Kumar, A., Mishra, D. A., & Vishal, V. (2026). Pore System Characterization of Untapped Himalayan Shale Using an Integrated Multianalytical Approach: Bridging Nano-to Macro-Scale Investigation. *Energy & Fuels*, 40(9), 4594-4612.
- Ram, B. K.,<sup>†</sup> **Singh, D.,**<sup>†</sup> Singh, H. K., Gupta, V., Mishra, D. A., & Kumar, S. (2026). Engineering Properties of Himalayan Rocks: A Comprehensive Review across Tectono-stratigraphic Zones. *Journal of Earth System Science*, 135, 110. (†Contributed equally)
- Singh, D.,** Nilankar, K., Singh, H. K., Kumar, A., Vishal, V., & Mustapha, K. A. (2025). Multiscale Characterization of Pore Evolution in Lesser Himalayan Black Shale from Krol Formation under Oxidic Thermal Heating: Relevance to Untapped Shale Gas Extraction. *Energy & Fuels*, 39(24), 11723-11738.
- Singh, D.,** Nilankar, K., Ram, B. K., Singh, H. K., Sharma, A., & Srivastava, P. (2025). Influence of Specimen Geometry on the Point Load Strength Anisotropy of Phyllite. *Geotechnical and Geological Engineering*, 43(8), 464.
- Singh, D.,** Nilankar, K., Singh, H. K., & Ram, B. K. (2025). Impact of Water Saturation on Physico-mechanical Properties of Fragile Chandpur Phyllite at varying Foliation Angles. *Geotechnical and Geological Engineering*, 43(1), 2.
- Singh, D.,** Ram, B. K., Singh, H. K., & Nilankar, K. Multiscale Weathering Grade Classification of Chandpur Phyllite: Insights from In-situ Observation and Laboratory Investigation. **(R2 version submitted, Under Review)**.
- Singh, D.,** Singh, H. K., Srivastava, P., & Kumar, R. Multiscale Mechanical Characterization of Krol Shale using Brazilian Tensile Strength and Nano-indentation Tests: Implications in Hydrocarbon Recovery and CO<sub>2</sub> Sequestration. **(Under Review)**.