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# EXPLORING THE PORE SYSTEM OF THE NEW ALBANY SHALE USING COMPLEMENTARY PORE CHARACTERIZATION TECHNIQUES

*Khawaja Hasnain Iltaf [1], Qinhong Hu [1,2], Majie Fan [1], Prince Oware [1], Qiming Wang [2], Chen Zhao [2], Tao Zhang [1], Rizwan Sarwar Awan [3], Sajjad Ahmad Shah [4]*

*[1]Department of Earth and Environmental Sciences, The University of Texas at Arlington, Arlington, TX 76019, USA; [2]National Key Laboratory of Deep Oil and Gas, China University of Petroleum (East China), Qingdao 266580, China; [3]School of Resources and Environmental Engineering, Hefei University of Technology, Hefei; [4]Department of Geosciences, Boise State University 1910 University Dr. Boise, ID 83725 USA*

UTA Earth and Environmental Sciences

## Introduction

The extraction of hydrocarbons from shale rock formations has become increasingly significant in recent years. To analyze the composition and pore structure of these rocks and assess their hydrocarbon extraction potential, various techniques, including petrographic analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), mercury injection porosimetry (MIP), and small-angle X-ray scattering (SAXS) were employed. The aim of this study was to characterize the mineral composition, surface morphology, and pore size distribution of the Upper Devonian New Albany Shale (NAS) formation in the Illinois Basin. The petrographic analyses provided insights into the mineral composition and texture of New Albany Shale, revealing the presence of quartz, clay minerals (e.g., illite and smectite), feldspars, micas, and pyrite. The shale exhibited a laminated appearance with alternating layers of different mineral compositions, and organic matter was observed as dark, amorphous material under the optical microscope. XRD was employed to determine the mineralogy e.g., identifying quartz, clay minerals, feldspars, calcite, and pyrite; the analysis of peak intensities and positions revealed details about the abundance and crystal structure of different minerals in the shale. SEM, MIP, $N_2$ physisorption and SAXS were utilized to characterize the pore structures of shale rocks, revealing intricate pore networks with varying diameters that influence rock permeability and porosity. Shale rock characterization using a combination of methods is essential for a better understanding of shale rock behavior and fluid movement within the matrix, enabling the development of efficient and sustainable extraction strategies.

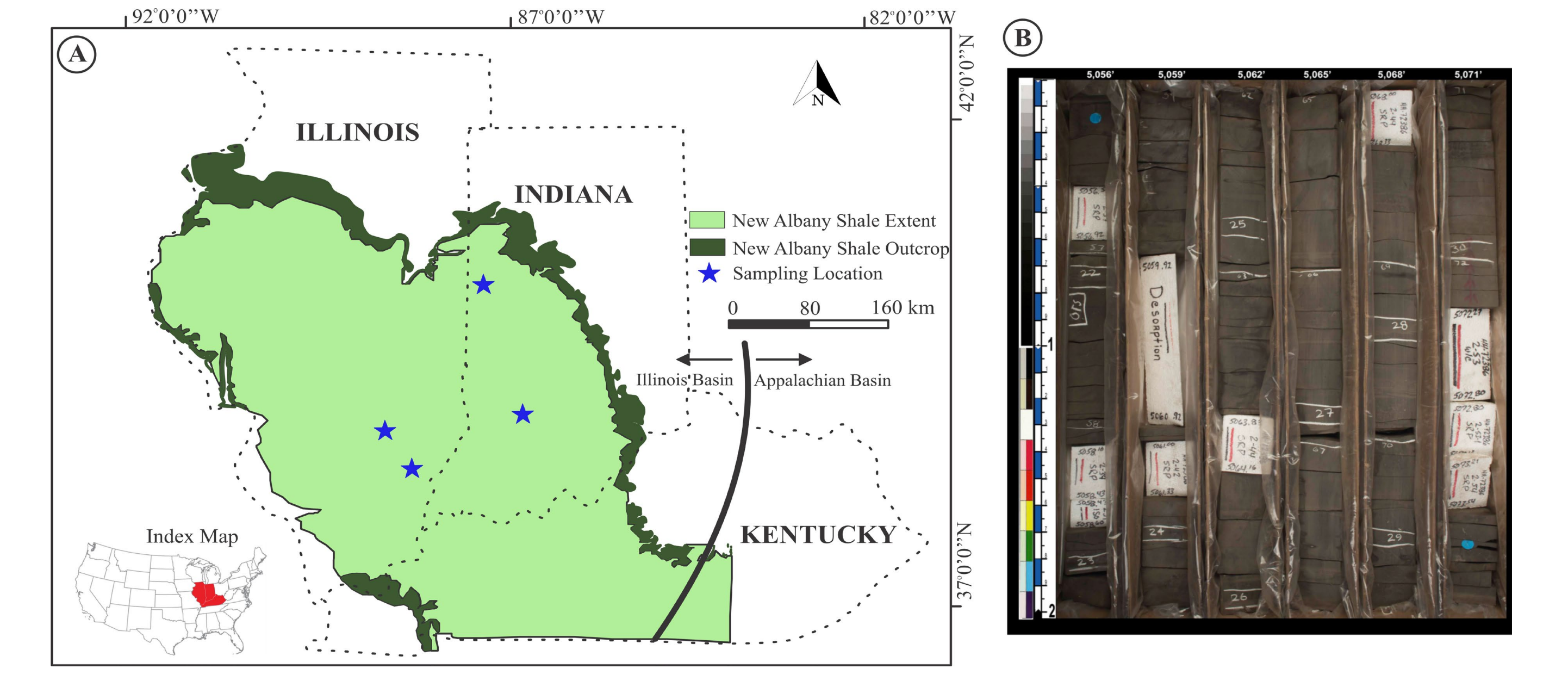
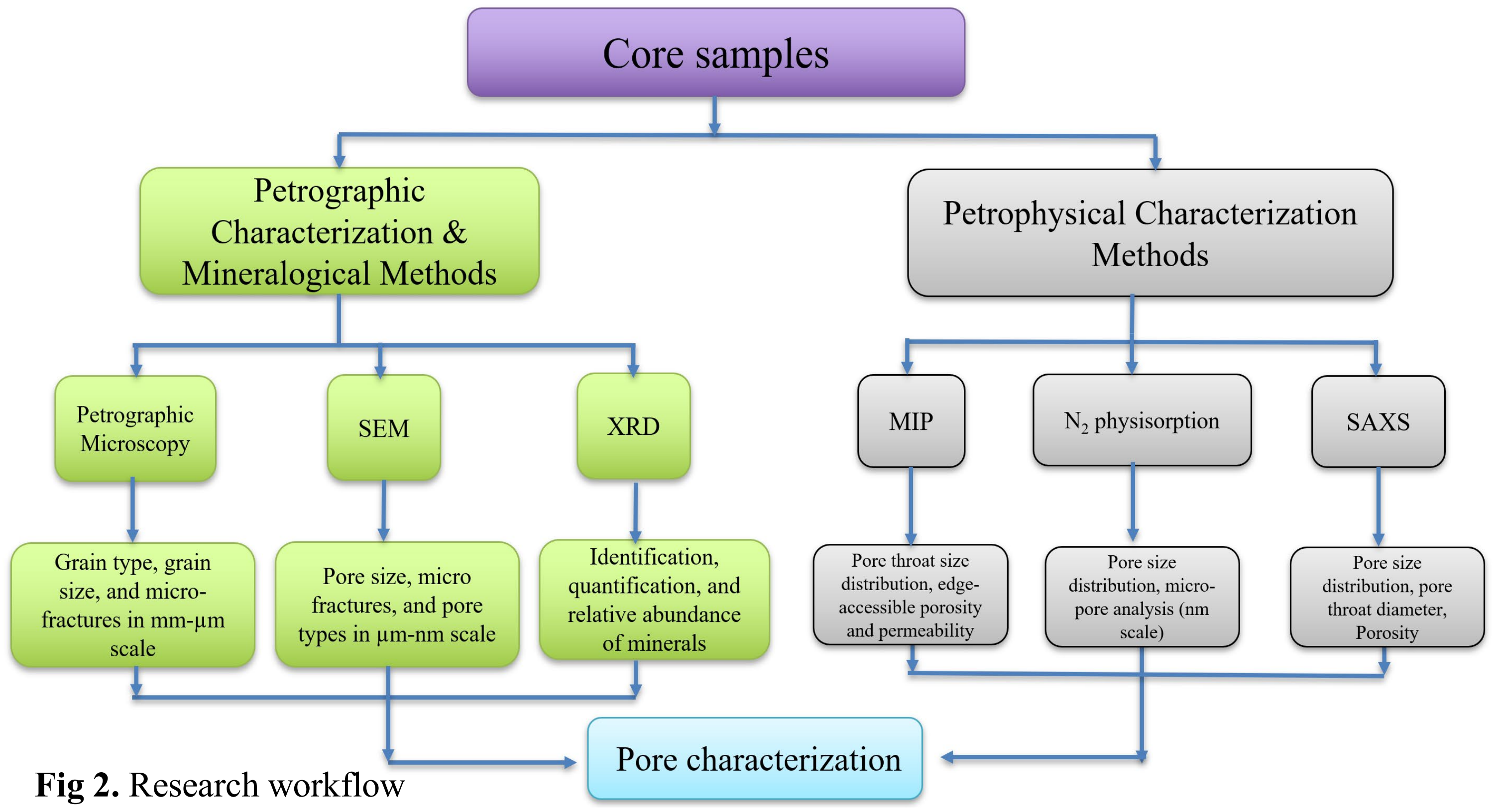


**Fig 1. (A)** Showing map of the Illinois Basin and New Albany Shale distribution and the locations of core samples utilized in this study (modified after Wimmer et al. 2011). **(B)** The cores of the corresponding wells.

## Research Methodology



Core samples

Petrographic Characterization & Mineralogical Methods
- Petrographic Microscopy → Grain type, grain size, and micro-fractures in mm-µm scale
- SEM → Pore size, micro fractures, and pore types in µm-nm scale
- XRD → Identification, quantification, and relative abundance of minerals

Petrophysical Characterization Methods
- MIP → Pore throat size distribution, edge-accessible porosity and permeability
- $N_2$ physisorption → Pore size distribution, micro-pore analysis (nm scale)
- SAXS → Pore size distribution, pore throat diameter, Porosity

Pore characterization

**Fig 2.** Research workflow

## Results

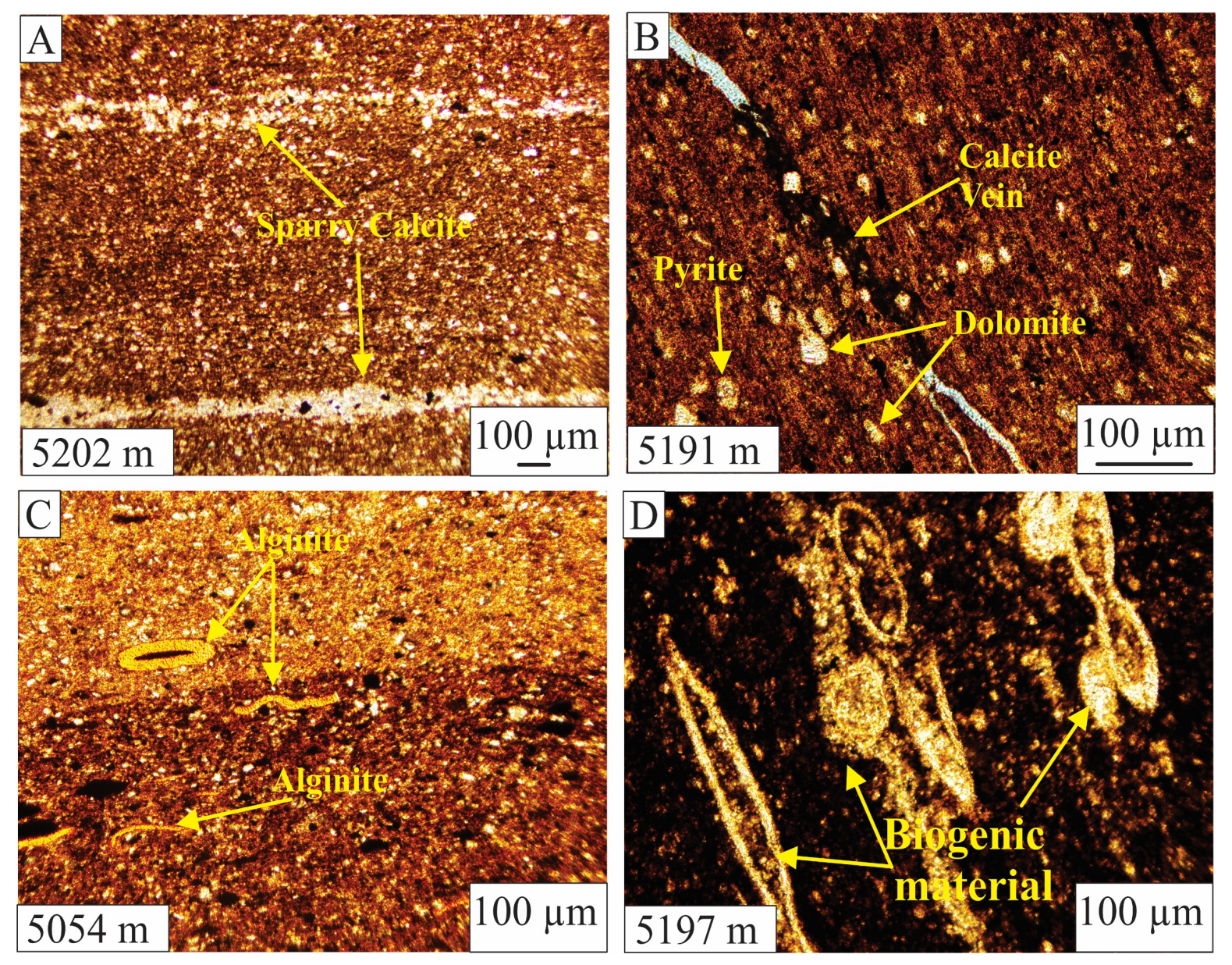


**Fig 3.** Detailed photomicrographic characterization of mineral contents in the New Albany Shale.

**(A)** Sparry calcite in shale at 5x magnification.

**(B)** Dolomite, pyrite, and organic matter interplay in calcite vein at 20x.

**(C)** Alginite and organic clay at 10x magnification.

**(D)** Biogenic content in shale at 10x.

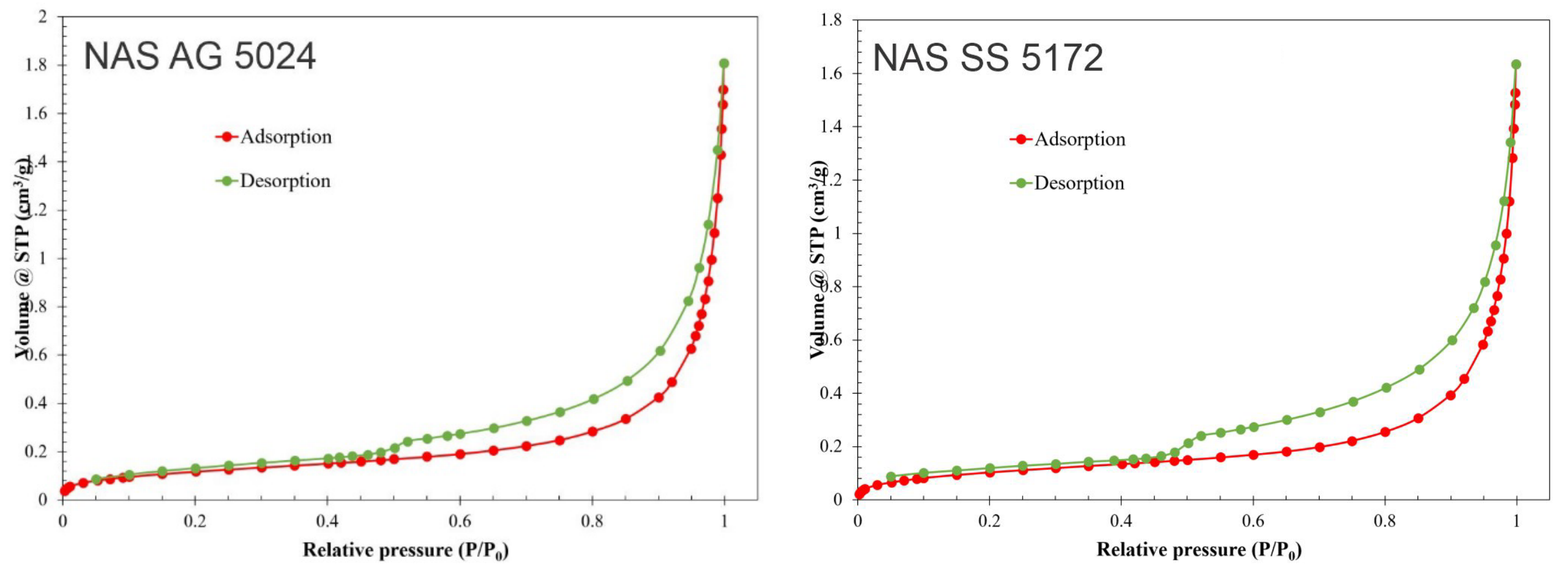


**Fig 4.** Isotherm adsorption and desorption curves, indicating pore volume and surface area characteristics across different relative pressures.

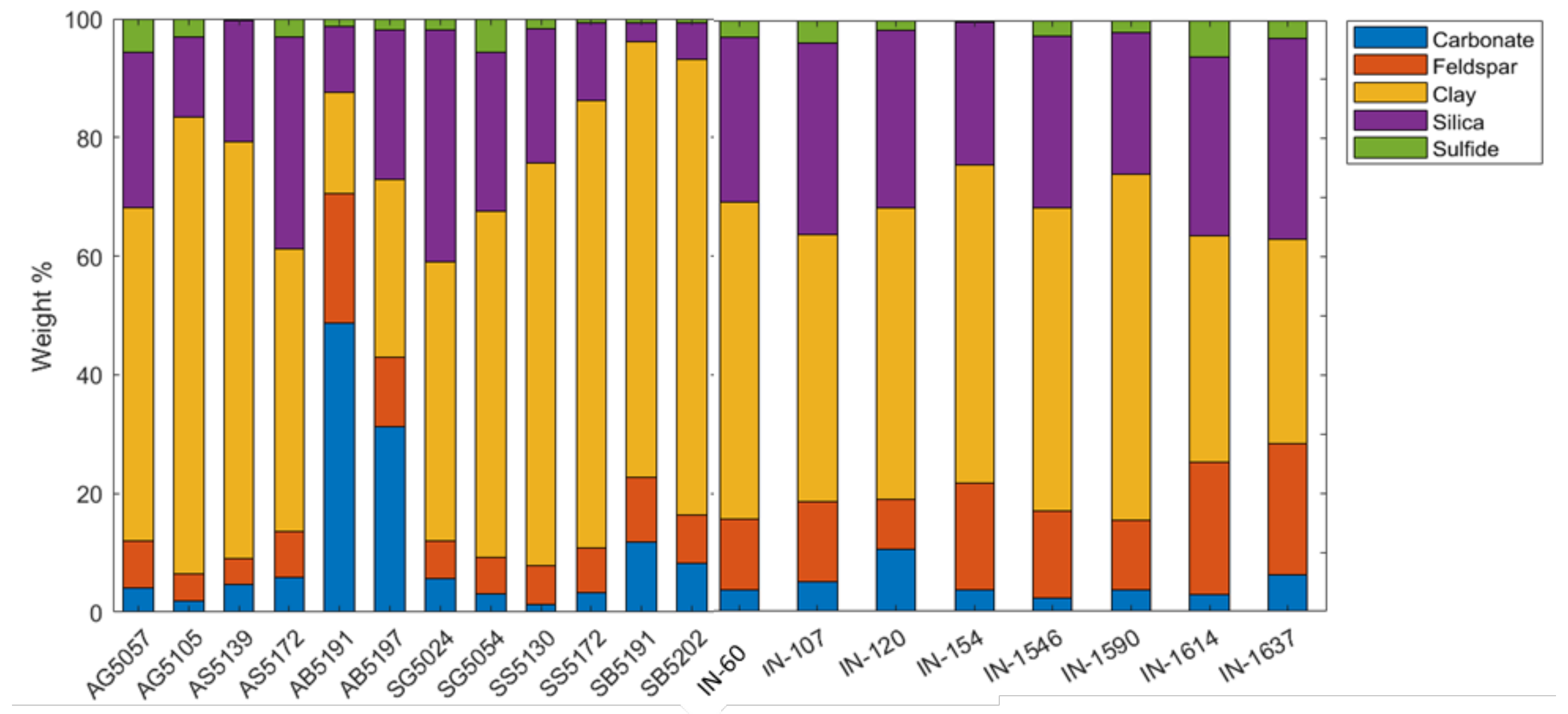


**Fig 5.** X-ray diffraction (XRD) analysis of the New Albany Shale.

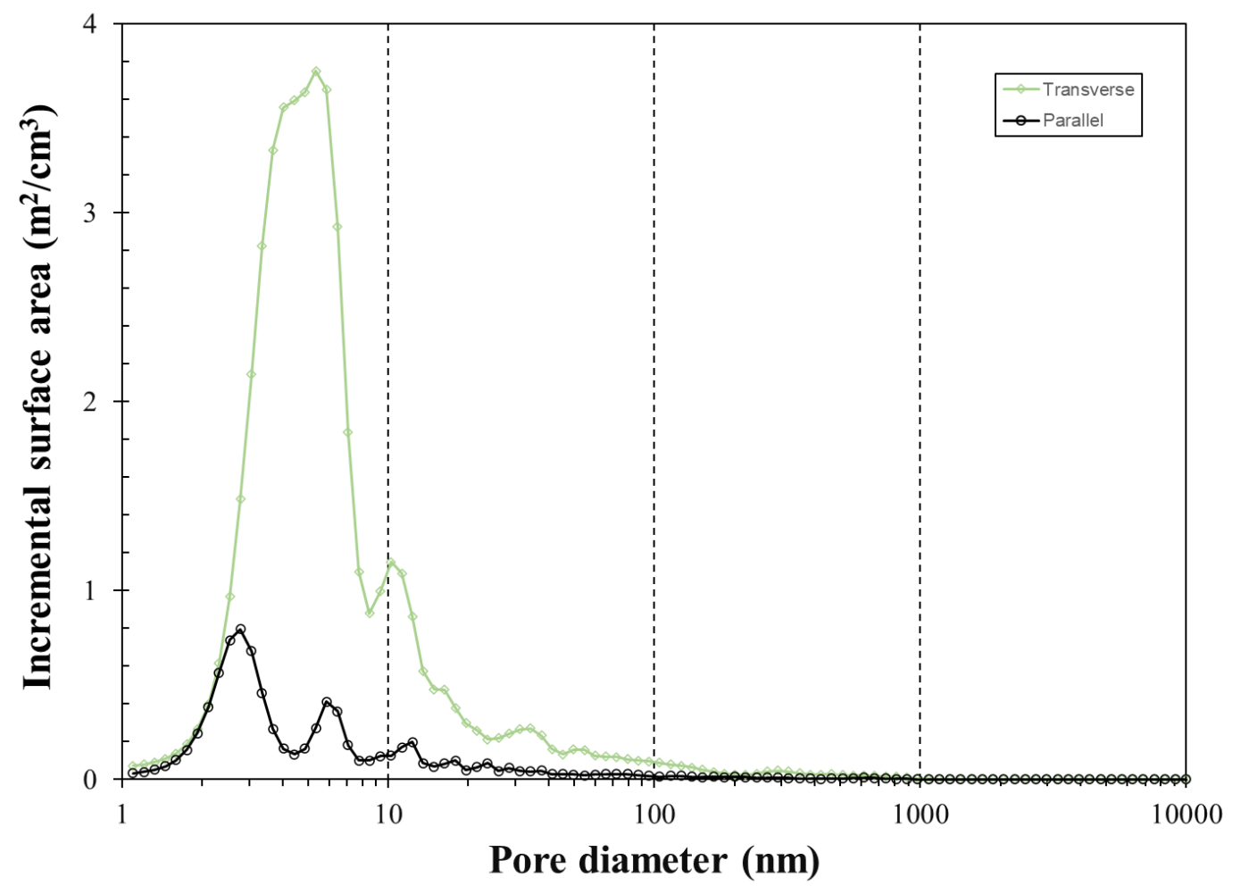


**Fig 6.** The figure shows pore surface area variations in transverse and parallel orientations, revealing significant heterogeneity in pore sizes within the sample.

**Table 1.** Showing variations in pore diameter, average specific surface area and average distribution

| Pore diameter | Avg. SSA (m²/g) | Avg. Dist (%) |
|---|---|---|
| 1-2.8 nm | 5.868 | 30.085 |
| 2.8-5 nm | 4.535 | 23.267 |
| 5-10 nm | 3.731 | 19.136 |
| 10-50 nm | 4.097 | 21.014 |
| 50-100 µm | 0.644 | 3.304 |
| 100 nm-1 um | 0.622 | 3.188 |
| 1-10 µm | 0.001 | 0.006 |
| Total SSA | 19.499 | 100 |

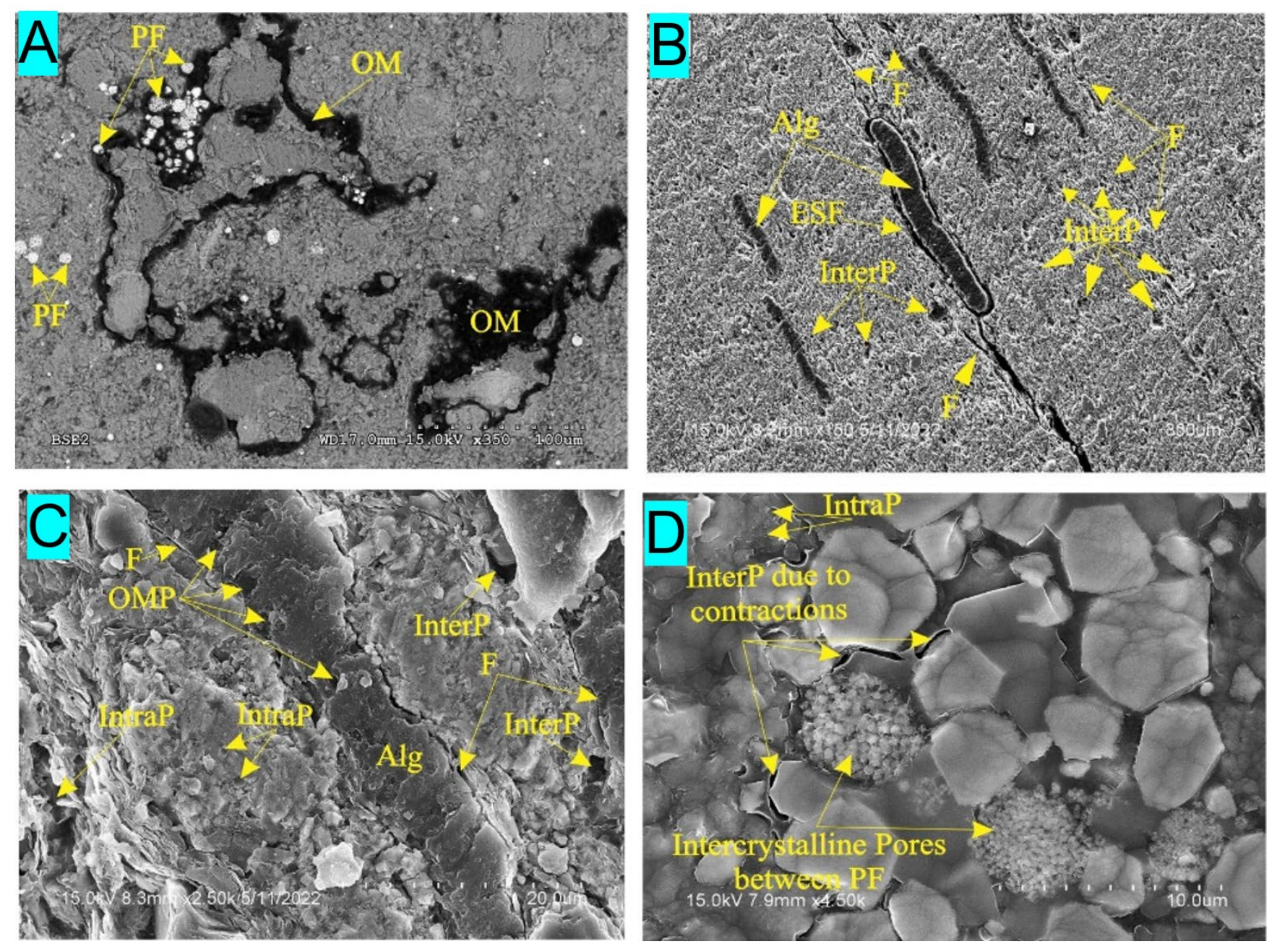


**Fig 7. (A)** Pyrite framboids (PF) are uniformly distributed in organic matter, suggesting low oxygenation/anoxic conditions during formation. **(B)** Features alginate, interparticle pores (interP), Fractures (F), and Edge seam fractures (ESF) between organic matter and crystalline phase. **(C)** Includes large intraparticle pores (IntraP). **(D)** Depicts intraparticle (IntraP) and Interparticle (InterP) pores from compaction, along with intercrystalline pores.

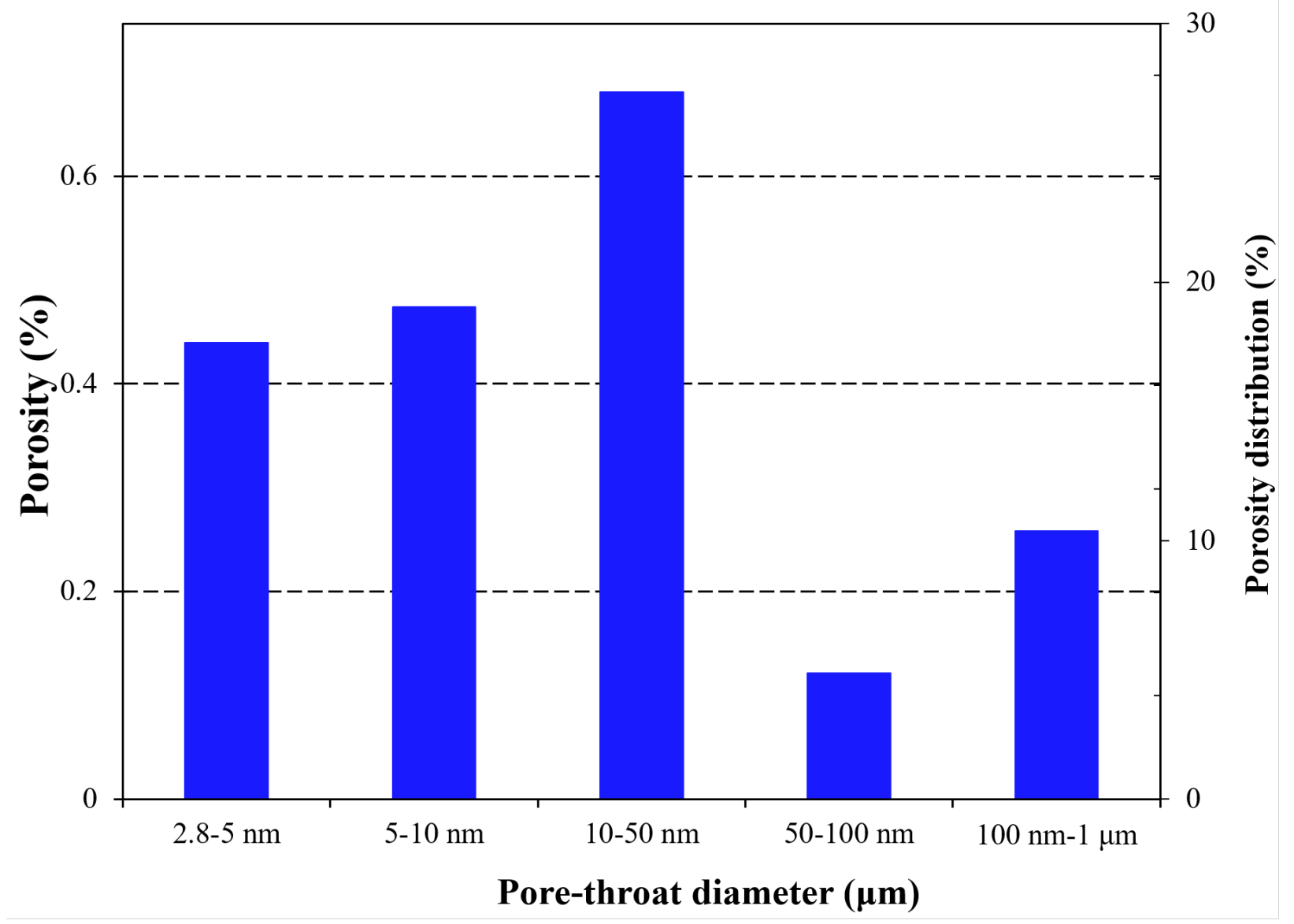


**Fig 8.** Distribution trend of pore-throat and porosity from the mercury injection capillary pressure experiment.

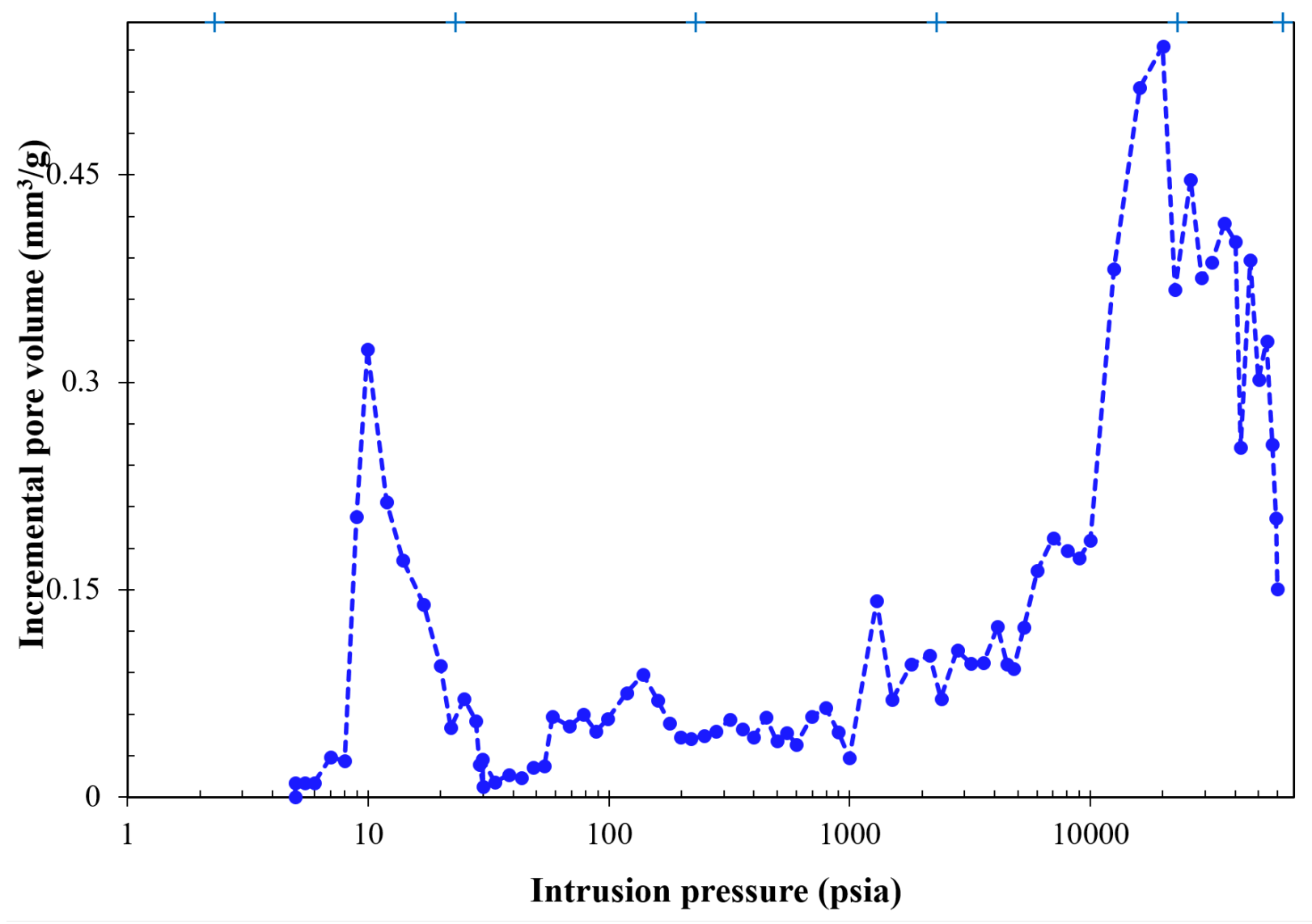


**Fig 9.** Incremental pore volume vs. intrusion pressure, displaying a mercury intrusion porosimetry curve with peaks indicating pore size distribution

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**Table 2.** Showing the pros, cons and pore size detection range of different methods (Bustin et al., 2008).

| Technique | Pros | Cons | Pore Size Detection Range |
|---|---|---|---|
| $N_2$ Physisorption | - Non-destructive<br>- Provides specific surface area and pore volume<br>- Good for micropore analysis | - Limited to pores accessible by $N_2$ at cryogenic temperatures<br>- not suitable for accurately characterizing macropores (more than 50 nm) | Micropores (0.35-2 nm)<br>Mesopores (2-50 nm) |
| SEM | - High-resolution images of pore surfaces<br>- Direct observation of pore morphology | - might miss internal pore details due to its focus on surface imaging.<br>- Surface characterization only, not bulk | Approx. 1 nm to 100 µm |
| MIP | - Provides pore size distribution<br>- Good for macropore analysis (more than 50 nm)<br>- Can measure pore throat size | - Invasive technique can alter pore structure<br>- can inaccurately represent very small pores due to mercury's high surface tension. | Approx. 3 nm to 100 µm or more |
| SAXS | - Non-destructive<br>- Provides information on pore structure in the nanometer range<br>- Applicable to a wide range of pore sizes | - Less effective for very large pores or macroscopic features.<br>- Requires high-intensity X-ray source | Approx. 1 nm to 100 nm |

## Conclusions

1. The NAS's complex pore network with abundant organic matter and various pore sizes, especially nanopores, significantly enhances its capacity for hydrocarbon adsorption.
2. Brittle minerals like quartz and feldspar in the NAS indicate potential for creating fracture networks, which could improve permeability.
3. $N_2$ physisorption results reveal the presence of meso- and macropores, indicating high storage capacity, but the pore shapes and connectivity might challenge consistent fluid flow.
4. MIP results show a predominance of micropores to mesopores, suggesting high microscale storage capacity; however, smaller pore throats could hinder fluid transport, necessitating reservoir stimulation for better production.
5. A wide range of nanopore sizes in the NAS indicates a high surface area for hydrocarbon adsorption, enhancing storage capacity. However, pore structure anisotropy could affect the direction and efficiency of fluid movement within the shale.

## References

- Wimmer, B. T., Krapac, I. G., Locke, R., & Iranmanesh, A. (2011). Applying monitoring, verification, and accounting techniques to a real-world, enhanced oil recovery operational $CO_2$ leak. Energy Procedia, 4, 3330-3337.
- Bustin, R. M., Bustin, A. M., Cui, X., Ross, D. J. K., & Pathi, V. M. (2008, November). Impact of shale properties on pore structure and storage characteristics. In SPE shale gas production conference (pp. SPE-119892). SPE.