

## An Overview of How the Petrophysical Properties of Rock Influenced After Being Exposed to Cryogenic Fluid

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### ABSTRACT

Exposure to cryogenic liquids can significantly impact the petrophysical properties of rock, affecting its density, porosity, permeability, and elastic properties. These effects can have important implications for various applications, including oil and gas production and carbon sequestration. Cryogenic liquid fracturing is a promising alternative to traditional hydraulic fracturing for exploiting unconventional oil and gas resources and geothermal energy. This technology offers several advantages over traditional hydraulic fracturing, including reduced water consumption, reduced formation damage, and a reduced risk of flow-back fluid contamination. In this study, an updated review of recent studies demonstrates how the thermal shock caused by the cryogenic liquid during the fracturing process substantially affects the rock's physical properties. Additionally, changes in permeability, porosity, and pore structure brought about by cryogenic treatments are highlighted. This work aims to draw attention to the studies that deal with the effect of thermal shock on rock petrophysical properties and establish the ideal conditions for employing cryogenic liquids in these contexts. Simulation studies, laboratory trials, and field application cases have been undertaken to assess the efficacy of cryogenic liquid fracturing technology. These investigations have provided important insights into the physical and mechanical impacts of thermal shock on rock and the performance of cryogenic liquid fracturing in real-world situations.

**Keywords:** Petrophysical Properties, Cryogenic, Porosity, Permeability, Liquid Nitrogen.

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## نظرة عامة على كيفية تأثير الخصائص البتروفيزيائية للصخور بعد تعرضها للسائل المبرد

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### الخلاصة

يمكن أن يكون للتعرض للسوائل المبردة تأثير كبير على الخصائص البتروفيزيائية للصخور ، مما يؤثر على كثافته ومساميته ونفاذيته وخصائصه المرنة. يمكن أن يكون لهذه التأثيرات آثار مهمة على تطبيقات مختلفة ، بما في ذلك إنتاج النفط والغاز ، وخصائصه المرنة. يعتبر التكسير بالسائل المبرد بديلاً واعداً للتكسير الهيدروليكي التقليدي لاستغلال موارد النفط والغاز غير التقليدية والطاقة الحرارية الأرضية. تقدم هذه التقنية العديد من المزايا مقارنة بالتكسير الهيدروليكي التقليدي ، بما في ذلك تقليل استهلاك المياه وتقليل أضرار التكوين وتقليل مخاطر تلوث السوائل المتدفقة. الصدمة الحرارية الناتجة عن السائل المبرد أثناء عملية التكسير لها تأثير كبير على الخصائص الفيزيائية للصخور. يمكن أن يتسبب الإجهاد الحراري الشديد في تكسير الصخور وإنشاء شبكة تكسير أكثر تعقيداً من تلك الناتجة عن التكسير الهيدروليكي التقليدي. يمكن أن يؤدي ذلك إلى تحسين النفاذية وإنتاج موارد النفط والغاز. الغرض من هذا العمل هو لفت الانتباه إلى الدراسات التي يجب إجراؤها لفهم هذه التأثيرات بشكل كامل وتهيئة الظروف المثالية لاستخدام السوائل المبردة في هذه السياقات. كانت هناك دراسات محاكاة وتجارب معملية وحالات تطبيق ميدانية تم إجراؤها لتقييم فعالية تقنية تكسير السوائل المبردة. قدمت هذه التحقيقات رؤى مهمة حول التأثيرات الفيزيائية والميكانيكية للصدمة الحرارية على الصخور ، بالإضافة إلى أداء تكسير السائل المبرد في مواقف العالم الحقيقي.

**الكلمات المفتاحية:** الخصائص البتروفيزيائية ، التجميد ، المسامية ، النفاذية ، النيتروجين السائل.

## 1. INTRODUCTION

Liquid nitrogen fracturing technology is a rapidly expanding field of study with enormous potential for improving the efficiency of unconventional oil and gas resources and for producing geothermal energy (JPT staff, 1998b; Beck et al., 2017; Alharith et al., 2020; Yang et al., 2021; Sun et al., 2023). Cryogenic fracturing fluids are liquids used in cryogenic liquid fracturing, an alternative to traditional hydraulic fracturing (Bai et al., 2019; Kalam et al., 2021; Alameedy et al., 2023a). Cryogenic liquid fracturing primarily aims to create fractures in rock formations by inducing a thermal shock. Some common examples of cryogenic fracturing fluids include liquid nitrogen (-196 °C) and liquid helium (-268 °C) (State, Polytechnic and Kingdom, 2010; Li et al., 2022; Qu et al., 2023).

These cryogenic liquids are known for their low temperatures, which can cause thermal shock in rock formations, leading to changes in their physical properties such as density, porosity, permeability, and elastic properties (Khalil and Emadi, 2020; Memon et al., 2020; Wang et al., 2022a; Alameedy et al., 2023b). This can result in the formation of fractures, improving the flow of fluids, such as oil and gas, in the reservoir. However, using cryogenic liquids in down-hole conditions presents technical and economic challenges that need to be addressed in future research efforts (Wang et al., 2016; Fu and Liu, 2019; Gaurina-Medimurec et al., 2021).

Compared to the most conventional hydraulic fracturing fluids like water (**Rassenfoss, 2013; Al-Ameedy and A.Alrazzaq, 2022**), liquid nitrogen usage has several advantages. This is because the rock rapidly contracts due to the low temperature of the liquid nitrogen, resulting in tensile stress and ultimately fracturing the rock. Furthermore, the low viscosity of liquid nitrogen can enable it to seep into the rock's microscopic pores and fractures, distributing the heat stress uniformly throughout the material (**Almubarak et al., 2020**). Liquid nitrogen's use as a fracturing fluid also decreases the total volume of fluid required for the process and the volume of fluid that must be recovered afterward. This lessens the likelihood of fluid contamination in the reservoir, and thus, it can reduce the costs associated with transporting and storing the fluid (**Kim et al., 2014; Yang et al., 2019**).

However, some challenges are associated with using liquid nitrogen as a fracturing fluid (**Gala et al., 2023**). The low temperature of liquid nitrogen can cause a rapid decline in temperature in the rock formation, leading to the formation of ice and other secondary cracks that can affect the fracture network. Additionally, the handling and transportation of liquid nitrogen require specialized equipment and infrastructure, which can increase the costs of the fracturing process (**Li et al., 2020**).

Due to its potential to address some of the limitations and environmental concerns associated with traditional hydraulic fracturing methods, using liquid nitrogen as a fracturing fluid has garnered significant attention in recent years (**Cong et al., 2022**). The impact of low temperatures on rock properties, heat transfer between the wellbore and the rock at the bottom hole, and the fracturing efficacy of liquid nitrogen jets as overview are topics of interest that we'll be cover in this research (**Sahu et al., 2022**). In addition to research and development in the lab, liquid nitrogen fracturing has also been applied in the field (**Grundmann et al., 1998**).

## 2. EFFECTS OF LIQUID NITROGEN COOLING ON ROCK PROPERTIES

Cryogenic fluids like liquid nitrogen can drastically reduce temperatures, leading to thermal stresses in the underlying rock (**Yao et al., 2017; Ramezani and Emadi, 2021; Longinos et al., 2022a; 2022b**). As seen in **Fig. 1**, rocks are susceptible to damage from rapid cooling, particularly macro-cracks development (**Cai et al., 2015a**). Cryogenic liquids can alter various rock properties that contribute to fracturing (**Han et al., 2023; Thiyagarajan et al., 2023; Winterfeld et al., 2023**).



**Figure 1.** A phenomenon where rocks break after being exposed to liquid Nitrogen (**Cai et al., 2015a**).



## 2.1 Modifications of Mechanical and Physical Rock Properties

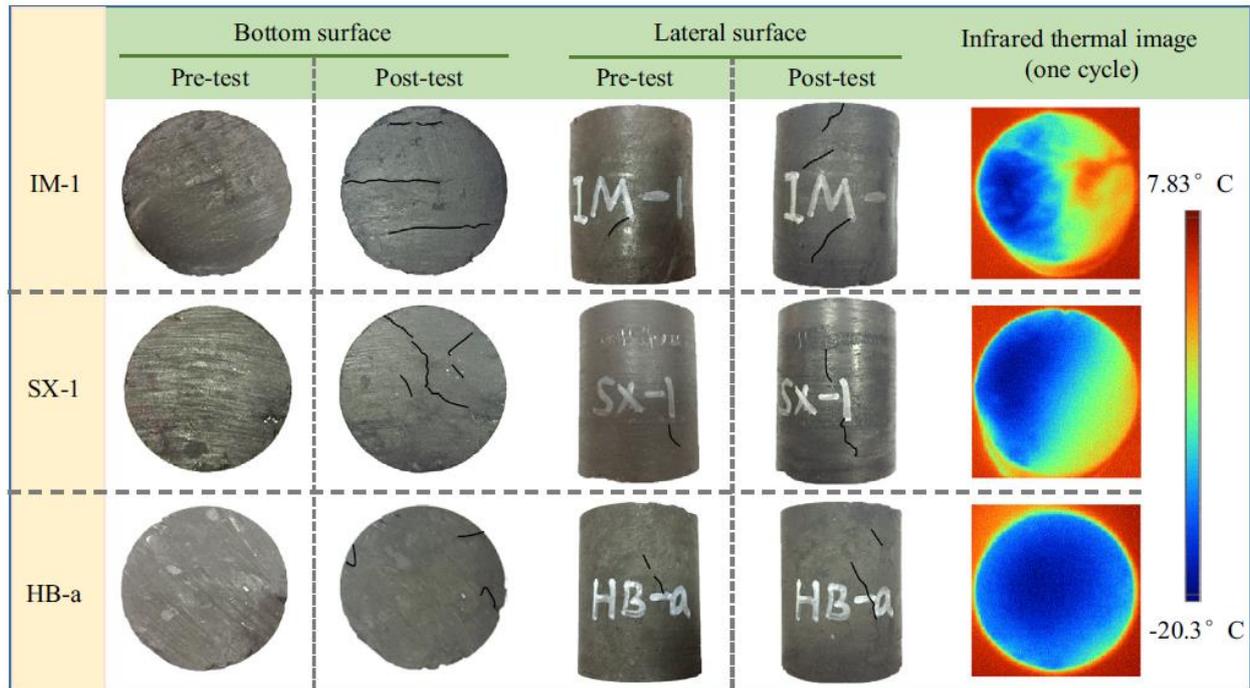
The effects of liquid nitrogen cooling on coal and shale as host rocks in unconventional reservoirs (**Beier and Sego, 2009; Huang et al., 2020**) and the results of this research (JPT staff, 1998a) showed that natural joints and beddings in these rocks act as weak planes and contribute to the start and spread of thermal fractures when they are subjected to liquid nitrogen thermal shock. Additionally, JPT staff stated that coal and shale substantially modify their physical and mechanical characteristics upon chilling in liquid nitrogen. Coal's permeability increased by 93.55 %, its P-wave velocity decreased by 10.43 %, and its uniaxial compression strength and peak strain decreased by 33.74 % and 20.61 %, respectively (**Cai et al., 2015b**). Nevertheless, shale permeability was improved from 8.01% to 74.36%, and the P-wave velocity reduced from 4.06% to 16.08% (**Jiang et al., 2018a**). Furthermore, shale cored perpendicular to the bedding planes undergoes less of a change in characteristics when cooled in liquid nitrogen, suggesting that the coring direction has a significant impact on the response of shale to liquid nitrogen freezing (**Zhou et al., 2022; Wang et al., 2023a**).

## 2.2 Rocks' Deterioration After Cooling

Thermal stress is the main culprit, and it's produced by two different factors: a temperature gradient and uneven deformation of nearby minerals (**Wang et al., 1989**). The LN2 boils when it comes into contact with the rock. This boiling heating transfer of LN2 may cause a sharp temperature gradient, which causes differential deformations in various rock areas. Due to deformation mismatches between adjoining mineral particles during the LN2 chilling process, the rock comprises several distinct mineral particles with various thermophysical and mechanical characteristics (**Li et al., 2020**). Local thermal stresses are created at the mineral borders as a result of this, and when the thermal stress is too great compared to the cementation strength, the cementation structure between the particles breaks, and intergranular fractures are produced (**Wu et al., 2019**). They also pointed out that some minerals may exhibit intragranular deformation mismatch because of various crystallographic axes' varying thermal expansion coefficients. This may result in intragranular fractures, which are less frequent and smaller than intergranular cracks.

(**Xu et al., 2017**) presented a study to alter the coalbed's permeability and increase pore volume by using the freezing-thawing process. This was accomplished by putting forward a technique known as "cyclical liquid carbon dioxide injection" which would involve regularly injecting liquid carbon dioxide into the coalbed (**Yang et al., 2023**). The study's findings implied that the various coal samples had varying degrees of deterioration due to the freezing-thawing cycles, as shown in **Fig. 2**. The temperature was not uniformly distributed due to elements such as core anisotropy and metamorphism. The fracture formation varied amongst the coal samples as well, and it seemed that petrophysical properties (**Almalichy et al., 2022**), ice extrusion, thermal stress, and axial compression were all factors in its development.

**Fig. 3** depicts the development of fractures in coal samples with increasing freeze-thaw cycles (**Qin et al., 2017**). The front and bottom of the coal sample are shown in **Fig. 3a** at various points throughout the testing (**Yang et al., 2019a**). The coal sample's surface was smooth before testing, with just a few noticeable early fractures. After many freeze-thaw cycles, a surface major fracture was created. A crisscrossing pattern of secondary fractures developed towards the top end of the primary crack after 20 freeze-thaw cycles. The fissures eventually grew and joined to create a crack network after several freeze-thaw cycles, seriously harming the coal sample. These findings demonstrate the significance of considering the cumulative effects of freeze-thaw cycles on coal and rock samples in engineering and geotechnical applications by showing that the damage to the coal samples increased with the number of freeze-thaw cycles.



**Figure 2.** Varying degrees of deterioration as a consequence of the freezing-thawing cycles (Xu et al., 2017)

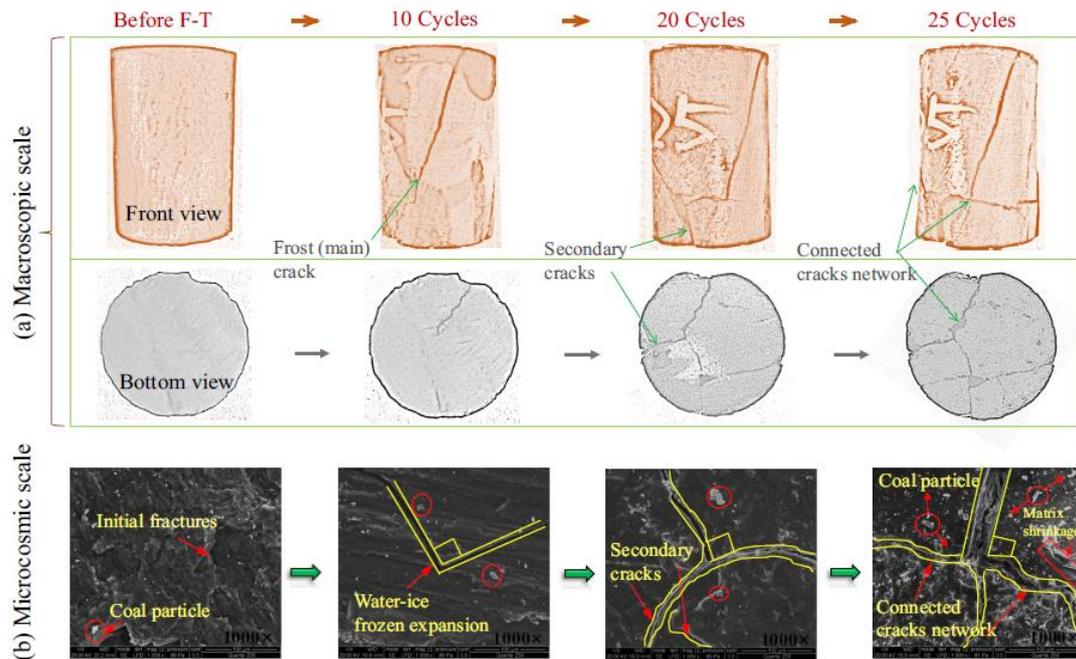
A microscopic view of the coal samples after passing through multiple freeze-thaw cycles is shown in the SEM picture in **Fig. 3b**. Before the examination, the coal sample's surface seemed smooth with a few visible particles and thin fractures. The cleats, on the other hand, developed rectangular frost-heaving fractures during the first freeze-thaw cycles. Following the second stage of freeze-thaw cycles, these fissures widened even further, with secondary cracks developing alongside the primary frost-heaving cracks. Long freeze-thaw cycles formed crack networks along the cleats, with the biggest crack being the largest. These networks included both extensional and shear fractures.

Extensional fractures were created as a result of the expansion of water during the transition between the water-ice phase and freezing and thawing processes (Jiang et al., 2018b). Tensile-shear cracks were also created due to the coal substrate material contracting as it cooled. As the number of freeze-thaw cycles grew, additional particles also spalled off the surface of the coal due to the ultralow temperature of liquid nitrogen (Han et al., 2022; Alameedy et al., 2023d). When these particles entered the fractures, they prevented them from closing. This made removing the methane from the ground simpler.

### 3. CRYOGENIC EXPERIMENTATION TECHNIQUES

The experimental procedure for cryogenic cooling with petrophysical characterization typically involves the following steps (Xu et al., 2017; Huang et al., 2020; Memon et al., 2020; Ayala et al., 2023):

- 1. Sample preparation:** The rocks or coal samples are drilled from the underground reservoirs and cut into cylindrical core samples of appropriate size. The samples should be well sealed to prevent water or air exchange (McPhee et al., 2015).



**Figure 3.** Demonstrate images of the evolution of coal cracks using LN2 (a) on the optical macroscopic scale and (b) on the SEM microcosmic scale (Qin et al., 2017).

2. **Sample characterization:** Before the cryogenic cooling experiment, the sample should be characterized to obtain its initial petrophysical properties, such as porosity, permeability, density, and mineral composition (Corbett and Potter, 2004; Alameedy, 2023). Nuclear magnetic resonance (NMR) and infrared thermal imaging (ITI) are two methods that are often used for this (Xu et al., 2017).
3. **Cryogenic cooling:** The core samples are placed in a container, and liquid nitrogen (LN2) is used to cool the samples. During the cooling process, the temperature of the sample has been monitored, and the cooling rate can be changed as needed (Huang et al., 2019). The experimental setup for LN2 fracturing shown in Fig. 4 was designed to create controlled conditions for investigating the effects of cryogenic cooling on rocks. Researchers can use this setup to study how rocks behave mechanically under different conditions, like temperature and confining pressure. They can also figure out how thermal stress and other factors affect rock damage.
4. **Crack observation:** After the cryogenic cooling process, the sample is inspected to determine the extent of damage caused by thermal stress (Song et al., 2016). The naked eye, scanning electron microscopy (SEM), and other imaging methods can detect cracks in the samples.
5. **Post-test petrophysical characterization:** After the cryogenic cooling experiment, the sample should be characterized again to determine the changes in its petrophysical properties. NMR and ITI can be used to track how pores change and how surface temperatures vary (Xu et al., 2017).
6. **Fracture analysis:** Look for cracks, fractures, and other types of mechanical damage in the cores, and use imaging techniques like X-ray computed tomography (CT)(Shirani et al., 2010; Tudisco, 2013; Butt, 2019) or optical microscopy to study where the cracks are and how they relate to other petrophysical parameters (Jin et al., 2019).

7. **Data analysis:** The data collected during the experiment is analyzed to understand the influence of cryogenic cooling on the sample's petrophysical properties. The results can be compared to the first characterization data to determine how big the changes are.

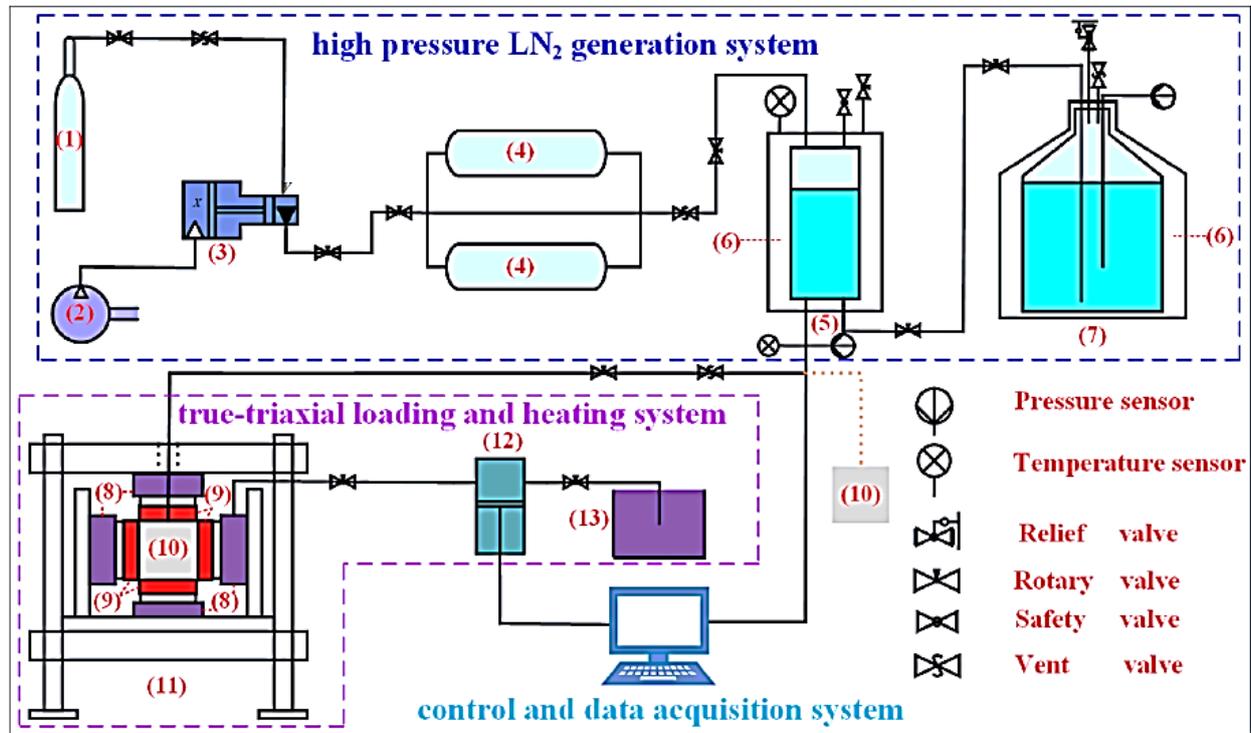
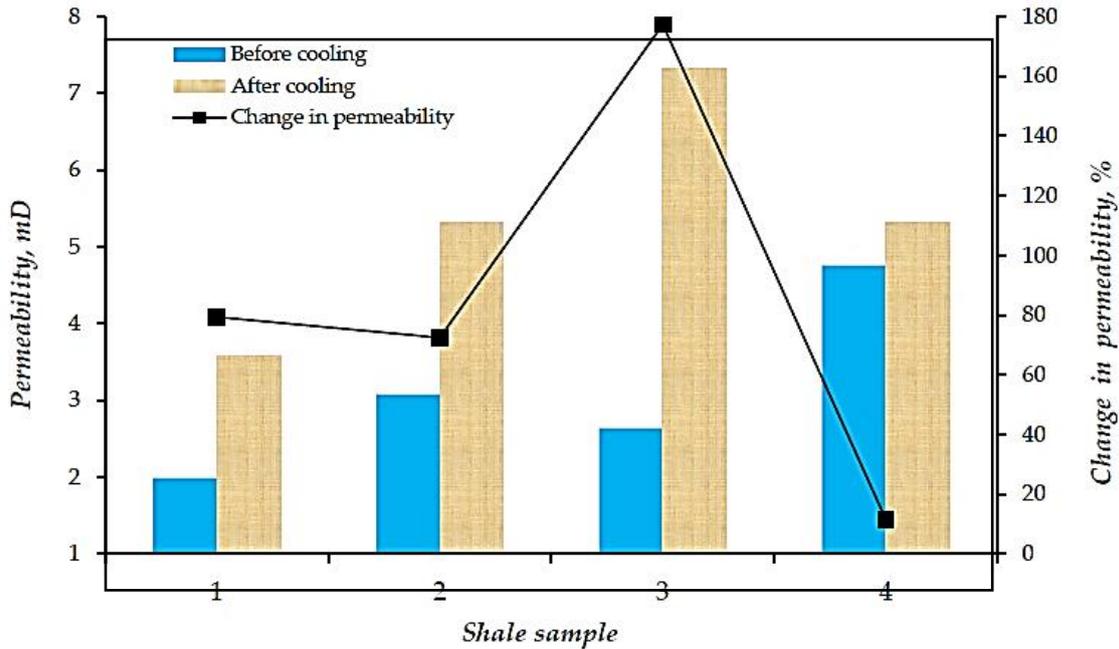


Figure 4. A conceptual illustration of the LN2F configuration (Huang et al., 2019).

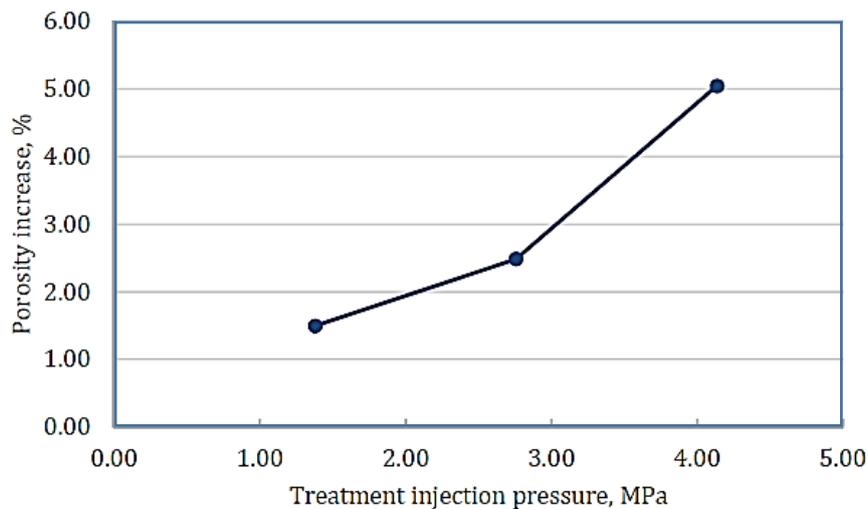
#### 4. PERMEABILITY AND POROSITY STRUCTURE CHANGES CAUSED BY CRYOGENIC TREATMENTS

The permeability test results indicate that cooling with liquid nitrogen substantially impacts the connectivity of the rock pore structure (Wang et al., 2022b; 2023b; Soykan et al., 2023). This may impact various industries, including petroleum engineering, where increased permeability may make it simpler to extract hydrocarbons from the earth (Alameedy et al., 2023c). As explained in the literature, the enhanced connectedness of the rock pore structures is shown by the higher permeability after liquid nitrogen cooling (Cai et al., 2015a). According to Fig. 5, the increase in permeability varied amongst the various samples and ranged from 11.55% to 177.27%. The thermal stress brought on by the shrinking of the rock matrix during contact with liquid nitrogen resulted in the formation of new micro-cracks and the enlargement of pre-existing cracks, which improved the connectedness of the rock pore structure. It's crucial to keep in mind that the permeability test results rely on various factors, including the kind of rock, the minerals present, the pore structure, and the cooling time (Khalil and Emadi, 2020).



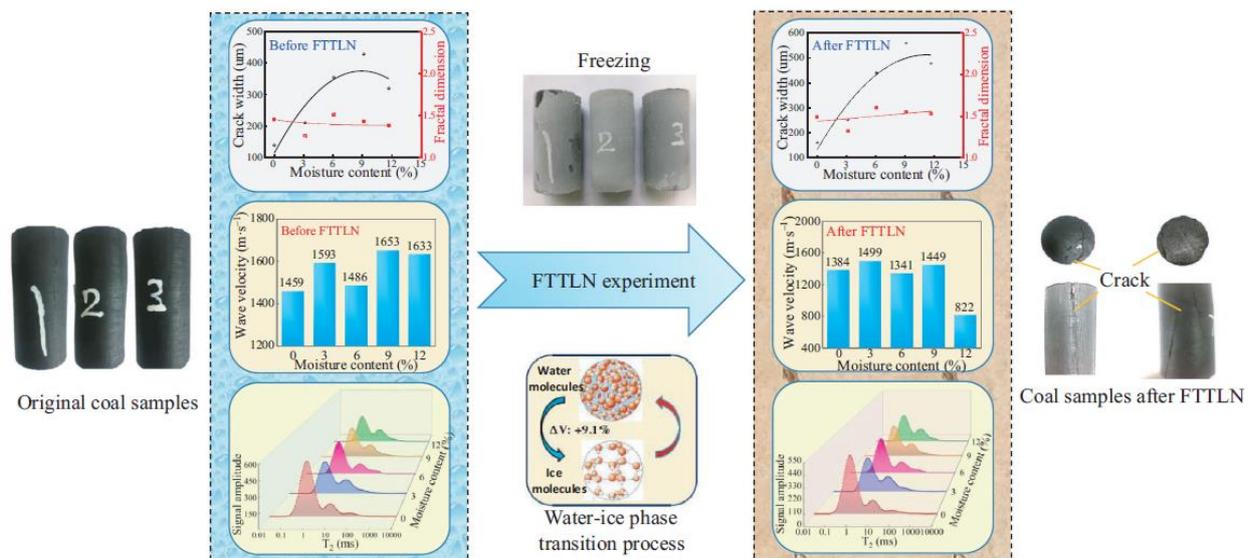
**Figure 5.** A Comparison of various permeability samples before and after being cooled in liquid nitrogen (Cai et al., 2015a).

It is commonly accepted that there is a clear correlation between increased porosity and the amount of pressurizing during cryogenic treatments. In other words, When the pressure rises, the rate of fracture propagation within the core samples also rises, increasing porosity; **Fig. 6** depiction of the direct link between the two components, lends credence to this relationship. According to the findings of (Khalil and Emadi, 2020), cryogenic treatments involving higher pressures may result in a bigger increase in porosity. However, it's crucial to remember that this connection is complex and subject to variations depending on the mechanical and thermal characteristics of the rock, the temperature of the cryogenic fluid, and the duration of the treatment (Carpenter, 2017).



**Figure 6.** Due to cryogenic treatments, the core samples' porosity has increased (Khalil and Emadi, 2020).

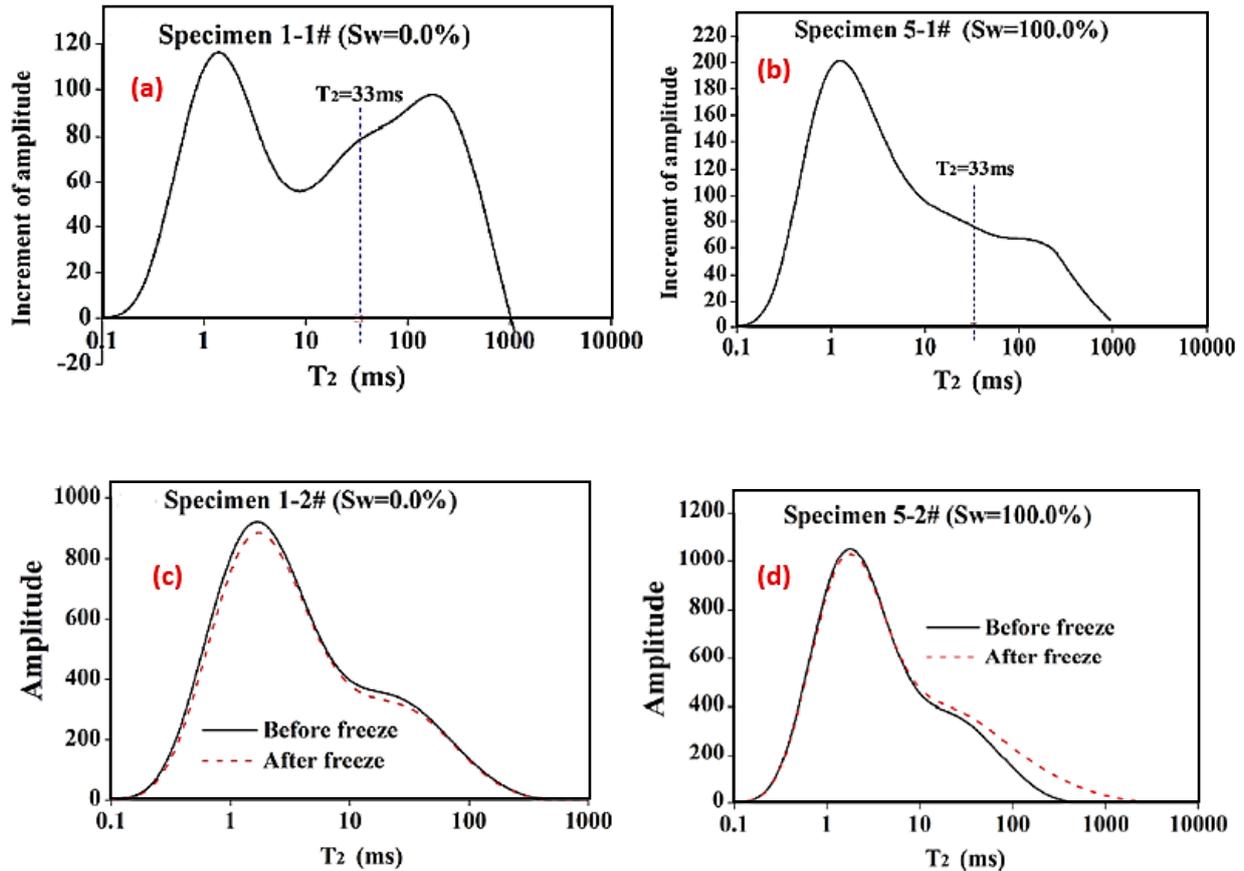
According to the investigations, the freeze-thaw treatment with liquid nitrogen (FTTLN) process significantly affected the structural changes in coal mass, as shown in **Fig. 7**. The breadth of the surface fissures widens, and the ultrasonic wave velocity decreases as the moisture content of the coal bulk rises (**Lin et al., 2020**). The coal mass's pore structure changes from micropores and tiny pores to mesopores and macropores while producing new micropores and small ones. The damage factor, which is determined by several factors including fracture width, longitudinal wave velocity, the fractal dimension of surface fractures, and porosity (**Qin et al., 2018**), was found to have a positive correlation with moisture content, suggesting that the more severely damaged the coal mass is, the higher the moisture content (**Cai et al., 2015b**).



**Figure 7.** Pore and fracture structures in coal samples frozen and thawed with liquid nitrogen changed over time (**Lin et al., 2020**).

## 5. IMPACT OF CRYOGENICS ON ROCK PORE STRUCTURE

The interesting experiments (Cai et al., 2014; 2015b) showed that the main cause of pore structure damage in sandstone and coal specimens that resulted from freezing by the liquid nitrogen for dry ( $S_w=0\%$ ) and wet ( $S_w=100\%$ ) samples was the development of micropores or micro-fissures. This was evident by the increase in  $T_2$  amplitude, which indicated the generation of new micropores or micro-fissures. The  $T_2$  amplitudes rose after freezing in the NMR test for a dry coal specimen ( $S_w = 0.0\%$ ) shown in **Fig. 8a**.  $T_2$  values larger than  $32 \mu s$  exhibited a more pronounced increase for a sample that was completely saturated with water ( $S_w = 100\%$ ), as shown in **Fig. 8b**; they climbed from  $260 \mu s$  to  $931 \mu s$ . These findings showed that freezing caused damage to the pore structure, with  $T_2 > 32 \mu s$ -corresponding pores often suffering more severe damage. It was shown in **Fig. 8c** for a dry sandstone specimen ( $S_w = 0.0\%$ ) that the amplitude of the whole  $T_2$  distribution reduced post-freezing because the sandstone's pore structure was not tightly packed and some pore space was occupied by tiny silts that had separated from other bigger grains. As demonstrated in **Fig. 8d**, the amplitudes of  $T_2$  behavior for a completely saturated ( $S_w = 100\%$ ) specimen, on the other hand, tend to grow dramatically from  $369$  to  $2,781 \mu s$ , particularly when  $T_2 > 32 \mu s$ .



**Figure 8.** T<sub>2</sub> distributions of specimens before and after freezing with liquid nitrogen: (a) coal sample with Sw=0, (b) coal sample with Sw=100, (c) sandstone sample with Sw=0, and (d) sandstone sample with Sw=100 (Cai et al., 2015b).

## 6. CONCLUSION

The significance of comprehending the alterations in rock properties caused by thermal shock is emphasized in this review, highlighting the necessity of determining the most favorable conditions for utilizing cryogenic liquids in such scenarios. Numerous simulation, laboratory, and field studies have contributed significantly to our understanding of the mechanical and physical effects of thermal shock on rocks and the practical effectiveness of cryogenic liquid fracturing technology. Cryogenic liquid fracturing is a promising technology for exploiting unconventional oil and gas resources and geothermal energy. Further research is needed to fully understand the effects of thermal shock on rock and address this technology's limitations. One major limitation of this technology is the excessive cost and the technical difficulties associated with handling and transporting cryogenic liquids. In addition, there are concerns about the environmental impact of this technology, including the release of greenhouse gases and the potential for groundwater contamination.

## NOMENC LATURE



Symbol	Description	Symbol	Description
CT	= X-ray computed tomography	LN2	=Liquid nitrogen
FTTLN	= Freeze-thaw treatment with liquid nitrogen	NMR	=Nuclear magnetic resonance
		SEM	=Scanning electron microscopy
ITI	= Infrared thermal imaging	T2	= Relaxation time ( $\mu$ s)

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