

THE IMPACT OF CARBONIC ACID ON POROSITY AND PORE STRUCTURE DURING MATRIX ACIDIZING OF CARBONATE RESERVOIRS: IMPLICATIONS FOR REDUCING CO₂ EMISSIONS

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Abstract

This study investigates the impact of carbonic acid on pore structure and porosity during matrix acidizing in carbonate formations by analysing T2 relaxation times and incremental porosity before and after treatment. Four core samples of Indiana limestone (outcrop formation) with dimensions of 2.5" in length and 1.5" in diameter, 14-15% porosity, and 3-7 mD permeability were used. Different injection rates (0.2, 0.5, and 1 cm³/min) were applied to assess the acid treatment's effectiveness under varying conditions. The carbonic acid was prepared by mixing 70% fresh water with 30% supercritical CO₂ at 1500 psi and room temperature. The results revealed a consistent pattern of incremental porosity and a shift in T2 relaxation times, indicating changes in the pore structure. Specifically, the acid treatment enhanced pore connectivity and increased pore size, resulting in elevated porosity. These findings provide valuable insights into the use of CO₂ as an alternative to conventional acids for improving well productivity.

Keywords: carbonic acid, matrix acidizing, nuclear magnetic resonance (NMR), CO₂ emission reduction

ABBREVIATIONS:

CO ₂	Carbon dioxide
NMR	Nuclear Magnetic Resonance
PVBT	Pore Volume to Breakthrough
IR	Injection Rate (cm ³ /min)
Ø	Porosity
K	Permeability (mD)
T2	Transverse Relaxation Time (NMR term)
D	Diameter of the core (cm)
L	Length of the core (cm)

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PV	Pore Volume (cm ³)
TBT	Time to Breakthrough (min)

1. INTRODUCTION

Recent advancements in CO₂ utilization have highlighted its potential in various industrial applications beyond enhanced oil recovery (EOR) and carbon capture and storage (CCS). One promising area is the use of carbonic acid, formed from CO₂ and water, as a stimulation acid for carbonate reservoirs. This innovative approach helps reduce greenhouse gas emissions and addresses tubular corrosion, often associated with hydrochloric acid (HCl) stimulation. By employing carbonic acid, industries can achieve more sustainable and environmentally friendly operations while maintaining the efficiency of well-stimulation processes [1, 2].

Matrix acidizing, a widely-used well-stimulation technique, involves injecting acid solutions below the formation's fracture pressure to enhance productivity by eliminating formation damage and creating new flow channels called "wormholes"[3, 4]. Since the 1890s, hydrochloric acid has been the dominant choice in treating oil and gas reservoirs due to its effectiveness in dissolving carbonate formations. However, HCl has several drawbacks, including high corrosivity, rapid reaction rates in high-temperature/high-pressure (HT/HP) conditions, inefficient acid penetration into deeper formations, increasing costs, and corrosion inhibitor requirements [5]. As a result, researchers are actively seeking alternative acid systems that offer the benefits of HCl with lower reaction rates and reduced corrosivity [6]

Organic acids such as methanesulfonic acid (MSA) and acetic acid are among the most promising alternatives. MSA's lower reactivity, resulting from its lower H⁺ activity coefficient and higher pK_a value than HCl, allows for deeper penetration into carbonate formations and more effective wormhole creation [7, 8]. Similarly, acetic acid and other weak acids like formic acid have shown slower reaction kinetics, facilitating deeper stimulation[9, 10].

Retarded acid systems, including gelled acids, in-situ gelled acids, and emulsified acids, have emerged as effective alternatives to control acid reactivity. Gelled acids are a prominent approach to managing the acid reaction rate by increasing the viscosity of the acid, which helps prevent fluid loss and improve acid placement. This approach is particularly practical in formations with heterogeneous permeability, where conventional acids might preferentially enter high-permeability zones, leaving low-permeability areas untreated[11]. In-situ gelled acids, which form a gel within the reservoir through pH or temperature-triggered reactions, further enhance acid placement by treating less permeable zones [12]. Emulsified acids, consisting of HCl droplets dispersed in an oil phase, slow down the acid-rock interaction, enabling deeper penetration. Surfactant-stabilized emulsions reduce acid consumption near the wellbore and improve wormhole propagation [13, 14]. Biopolymeric resin-based retarded HCl also shows promise, as polymers slow down the mobility of hydrogen ions, improving acid penetration in high-temperature environments [15].

Chelating agents like ethylenediaminetetraacetic acid (EDTA) and nitrilotriacetic acid (NTA) have also been explored for deep penetration and uniform acid distribution. These agents form stable complexes with calcium ions, controlling the reaction rate and minimizing near-wellbore spending, making them effective in high-temperature carbonate reservoirs [15–17].

Foamed acids, which incorporate nitrogen gas into HCl, present another practical approach for controlling acid reactivity. By reducing the contact between acid and rock, foamed acids slow the reaction rate, which promotes wormhole growth and reduces overall acid consumption. This system has proven particularly effective in heterogeneous formations, ensuring more uniform stimulation and deeper penetration even at low injection rates [18, 19].

Recently, many researchers have studied carbonic acid and its impact on carbonate formation. Al-Dhafeeri et al. examined the interaction of carbonated brine with Indiana limestone at three different temperatures (35, 60, and 85°C). They also discovered that increased temperatures correlated to greater wormhole volume and lowered rock strength, confirming the role of temperature in CO₂ storage and acid stimulation applications[20]. Additionally, they examined the effects of brine salinity on wormholes. Thicker wormholes were formed with lower salinity, whereas higher salinity greatly reduced rock strength. These results reinforce using low-salinity water for CO₂ storage and acid stimulation[21]. Mirzayev et al. investigated the effects of injected CO₂-saturated brine, using micro-CT scans to study wormhole formation in Indiana limestone at different pore pressures (1500–4000 psi) and 60 °C, and found that greater damage resulted from higher pore pressures, with reduced surface hardness and mechanical properties. These studies highlight the effects of temperature, salinity, and pore pressure on the efficiency and feasibility of CO₂ storage and acid stimulation[22].

In this study, we utilize carbonic acid as an environmentally friendly acid system to investigate its effect on the petrophysical properties of carbonate formations. The primary objective of this research is to explore how carbonic acid, when used in various injection rates, impacts these petrophysical properties, focusing on using nuclear magnetic resonance (NMR) as a critical evaluation tool. By analyzing the alterations in petrophysical characteristics under different conditions, this study aims to provide a comprehensive understanding of the influence of carbonic acid on the reservoir characteristics of carbonate formations, offering insights that could enhance acidizing practices in an environmentally conscious manner.

2. EXPERIMENTAL METHODS AND MATERIALS

2.1. Experimental procedure

As illustrated in Fig. 1, the experimental process starts with measuring two essential petrophysical properties: porosity, which quantifies the void space within the rock, and permeability, which assesses the rock's capacity to transmit fluids. Initially, the core sample's dimensions were determined using a caliper and weighed in a dry state. Porosity was then measured using a helium porosimeter, and permeability was roughly estimated with the Autoscan. The core sample was subjected to dry heating in an oven at 100°C overnight. It was then placed under a vacuum for three hours and saturated with fresh water at 2000 psi for 24 hours. The core flooding procedure was carried out next, as detailed in the corresponding section, during which the differential pressure (DP) and pore volume to breakthrough (PVBT) were recorded. The core samples were dried in an oven at 100°C after core flooding for 24 hours, placed under a vacuum for three hours, and saturated with fresh water at 2000 psi for 24 hours. Finally, Nuclear magnetic resonance (NMR) measured the relaxation time (T₂).

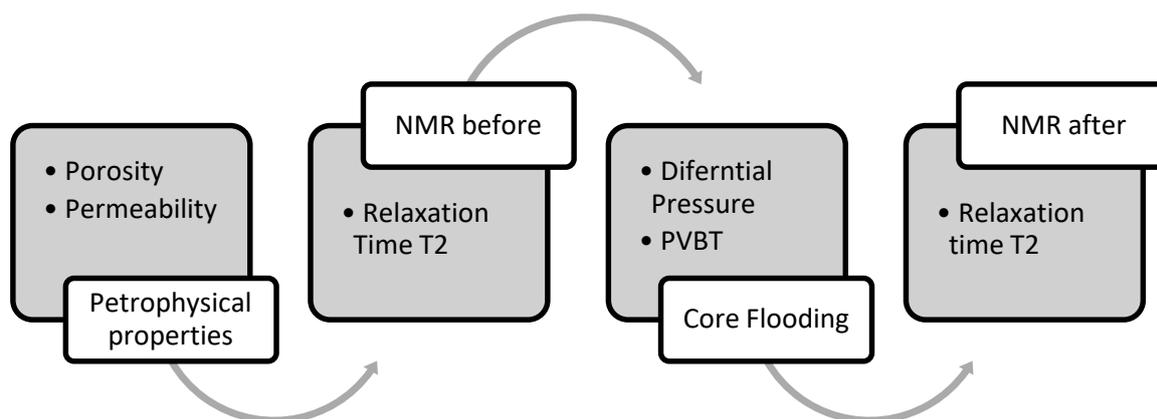


Fig.1. The experimental workflow

2.2. Samples

This study utilized three core samples from Indiana limestone (outcrop formation) measuring 2.5 inches in length and 1.5 inches in diameter. Initial porosity was assessed using helium porosimetry (AP-608); the results are presented in Table 1. Preliminary permeability estimates obtained via an auto scan indicated an initial permeability range of approximately 3 to 7 mD. XRD analysis revealed that the Indiana limestone is composed entirely of calcite. Each core was encased in a shrink tube to prevent cross-flow from the core sides.

Table 1. Core sample characteristics and properties

Core No.	D (cm)	L (cm)	IR (cm ³ /min)	Porosity (%)	Permeability (mD)
IL8	3.78	6.465	0.2	14.91	3.4
IL12	3.77	6.441	0.5	15.32	7
IL10	3.82	6.486	1	15.17	4

2.3. Core Flooding

2.3.1 Core flooding setup

Figure 2 depicts the schematic diagram of the flooding system used in Indiana limestone core flooding experiments with carbonic acid. The system operates at a pressure of 10,000 psi and a temperature of 150°C. The core holder and lines are constructed from Hastelloy, a material chosen for its high resistance to acid and high temperatures. The HP/HT Core Holder, made of Hastelloy to resist acidic conditions, houses the core sample. Before conducting the experiments, the core is loaded into the holder and subjected to a confining pressure of 3,500 psi. The core holder's inlet is connected to two accumulators: fresh water and carbonic acid. At the same time, the outlet is linked to a production line for effluent

sample collection lined with 2000 psi back pressure regulator. The ISCO pump injects carbonic acid at 0.2, 0.5, and 1 cc/min flow rates. A pressure transducer was introduced to measure the pressure differences between the inlet and outlet. Finally, the fraction collector gathers the effluent samples for analysis. This setup ensures precise control and monitoring of the flooding experiments, allowing for accurate data collection on the behavior of Indiana limestone core plugs under these conditions.

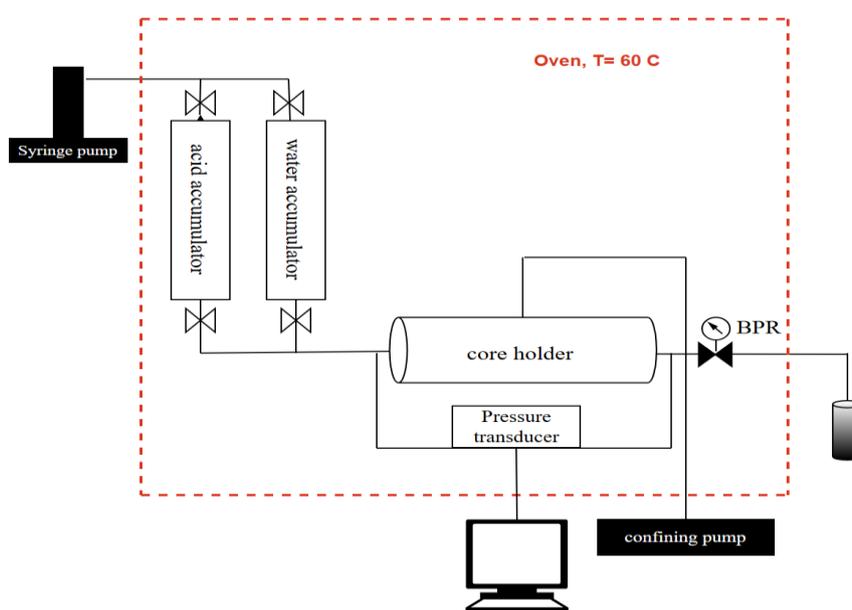


Fig. 2. Schematic diagram of single core flooding system

2.3.2 Core flooding procedure

This research prepared carbonic acid for testing by mixing 30% CO₂ with 70% fresh water using a high-pressure/high-temperature accumulator. Core flooding experiments commenced with the core sample being sealed with a shrink tube, loaded into the core holder, and then placed in an oven. A confining pressure of 3500 psi and a back pressure of 2000 psi were applied. Before each experiment, fresh water was injected at flow rates of 1, 2, 4, and 8 cm³/min, and the resulting differential pressure was measured to determine the water permeability of the core sample before flooding experiments began. Injection tests were conducted using an HP/HT esco pump at four different rates (0.2, 0.5, and 1 cm³/min). The criteria was ceased injection upon breakthrough.

2.4. Nuclear magnetic resonance (NMR)

NMR is a powerful and non-destructive tool widely used in laboratory and field scales as a wireline logging tool [23]. NMR measurement detects the existence of hydrogen protons. Throughout NMR analysis, it is possible to detect the amounts and characteristics of the fluids present and the diameters of the pores that hold these fluids [24]. NMR measurements were conducted before and after the acidizing to measure the relaxation time T₂ of the core sample and the diffusion tortuosity to study the effect of the carbonated water on the pore size distribution. Before the NMR measurement, the cores

were dried in the oven for 24 hours at 60°C, vacuumed for three hours, and then saturated with de-ionized water at 2000 psi for 24 hrs.

3. EXPERIMENTAL RESULTS

3.1. Pressure curve

Three core flooding experiments using carbonic acid were conducted to investigate the impact of injection rate on the petrophysical properties of carbonate formations. The details of each experiment are summarized in Table 2. In the first experiment, performed at a rate of 0.2 cm³/min, the pressure drop across the core sample ceased after 12.7 hours (762 minutes), with a breakthrough pore volume (PVBT) of 14.1 cc, as shown in Fig. 3. The second experiment, conducted at a rate of 0.5 cm³/min, is illustrated in Fig. 4. In this case, the pressure drop reached zero after 8.4 hours (504 minutes), with a PVBT of 22.8 cc. The third experiment, using a flow rate of 1 cm³/min, is depicted in Fig. 5. Here, the pressure decrease across the core sample ceased after 7 hours (459 minutes), with a corresponding PVBT of 40.5 cc.

The pressure drop graph for the IL8 sample at an injection rate of 0.2 cm³/min initially exhibits fluctuations, indicative of dynamic changes in the pore structure. These fluctuations occur as smaller pores dissolve and merge. Following this phase, the graph shows a gradual decline, signifying stabilization in the system. This steady decline highlights the formation of larger, interconnected pores, leading to enhanced permeability and improved connectivity within the sample.

The PVBT is a key indicator of pore structure modifications. An increase in NMR porosity post-treatment, evidenced by a shift toward shorter T₂ relaxation times, suggests the presence of smaller, interconnected pores. Lower PVBT values observed at reduced injection rates enable a more gradual and controlled dissolution process, resulting in uniform pore enlargement and enhanced porosity within the sample.

Table 2. The carbonic acid volume consumed at different injection rates

Core No	IR (cm ³ /min)	TBT (min)	PV	Volume (cm ³)	PVBT
IL8	0.2	762	10.80	152	14.1
IL12	0.5	504	11.03	252	22.8
IL10	1	459	11.32	459	40.5

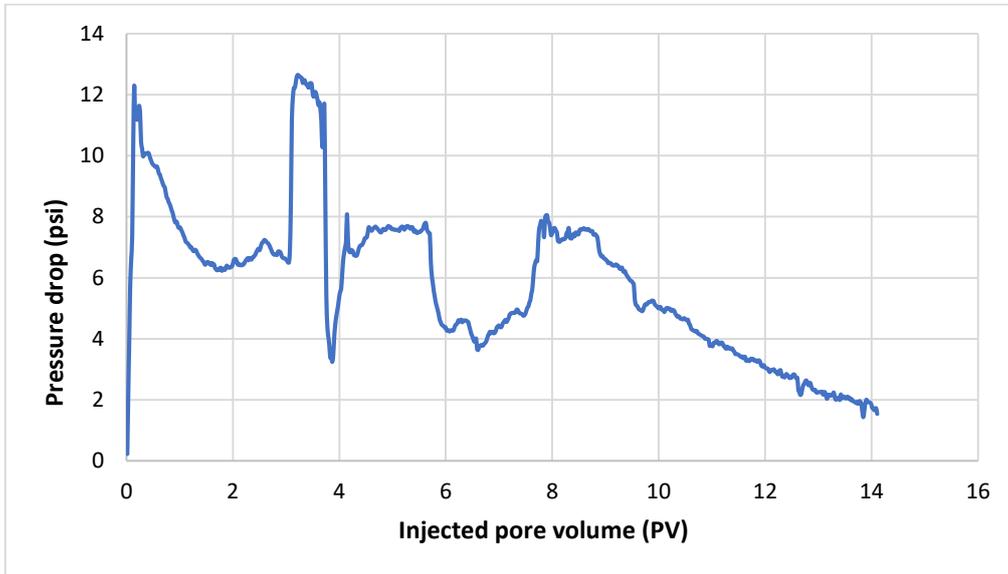


Fig. 3. Pressure drop across IL8 sample with 0.2 (cm³/min) injection rate

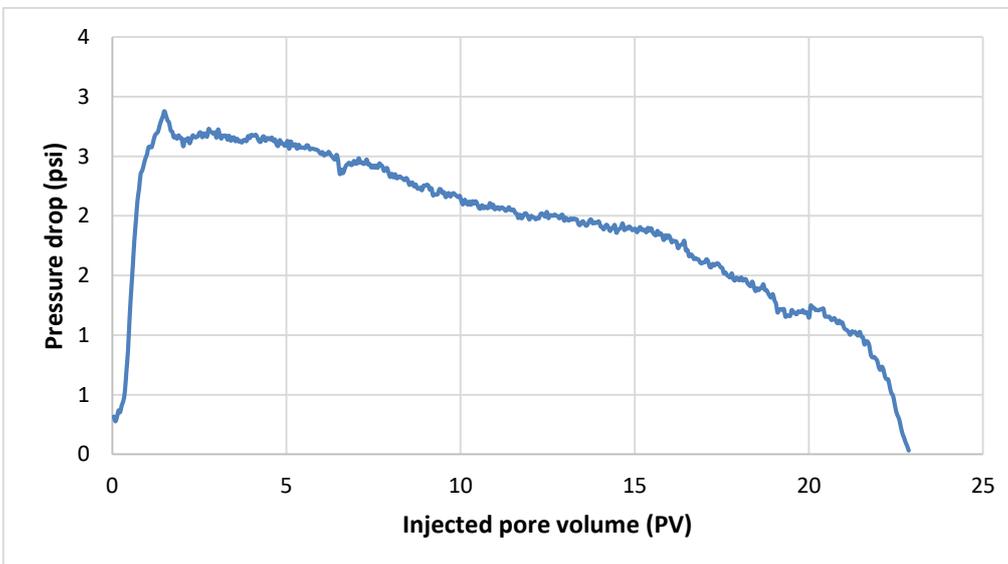


Fig. 4. Pressure drop across IL12 sample with 0.5 (cm³/min) injection rate

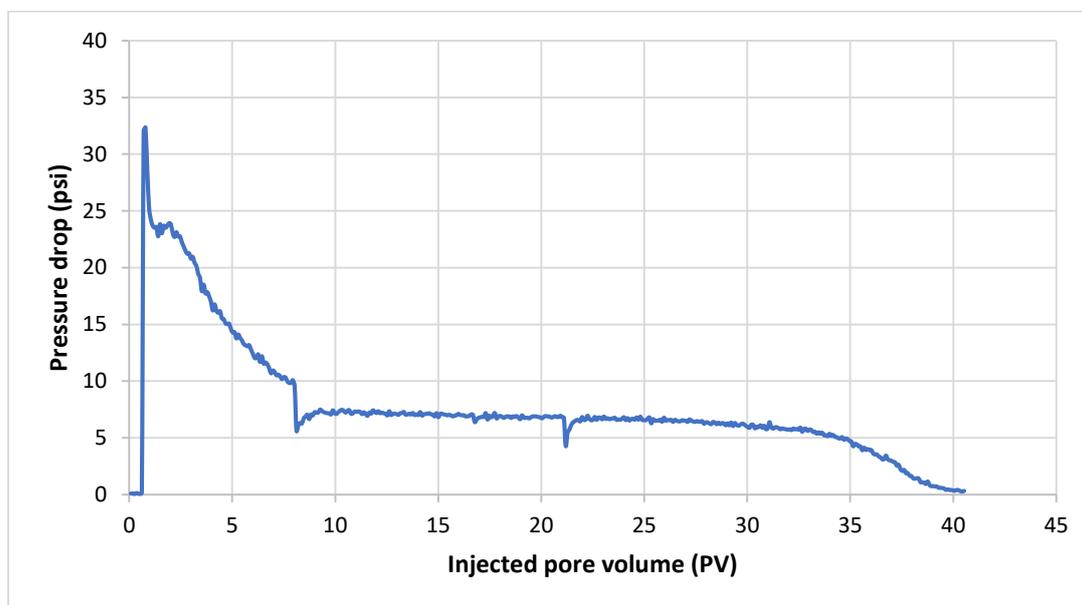


Fig. 5. Pressure drop across IL8 sample with 1 (cm³/min) injection rate

3.2. Pore size distribution

The impact of carbonic acid treatment on the pore size distribution of carbonate cores was assessed by analyzing the pressure drop data for Core Nos. 8, 10, and 12, each subjected to different injection rates. The post-treatment of the cores results in significant changes in the pore size distribution, directly impacting the flow characteristics and permeability.

3.2.1 Core No. 8 (Injection Rate: 0.2 cc/min)

For Core No. 8, the pressure drop graph shows significant initial fluctuations followed by a gradual decline, indicating dynamic alterations in the pore structure with smaller pores dissolving and merging into larger ones. The final steady decrease in pressure suggests a more stable pore network post-treatment. Correspondingly, the T₂ relaxation times for Core No. 8 post-treatment shift towards shorter relaxation times, indicating an increase in smaller, interconnected pores. However, the broadening of the T₂ peak reflects the creation of a range of pore sizes from small to medium-large. This suggests that the carbonic acid treatment has enhanced pore connectivity and increased the overall porosity by generating new pore spaces of varying sizes, which is particularly beneficial for improving the overall permeability of the rock.

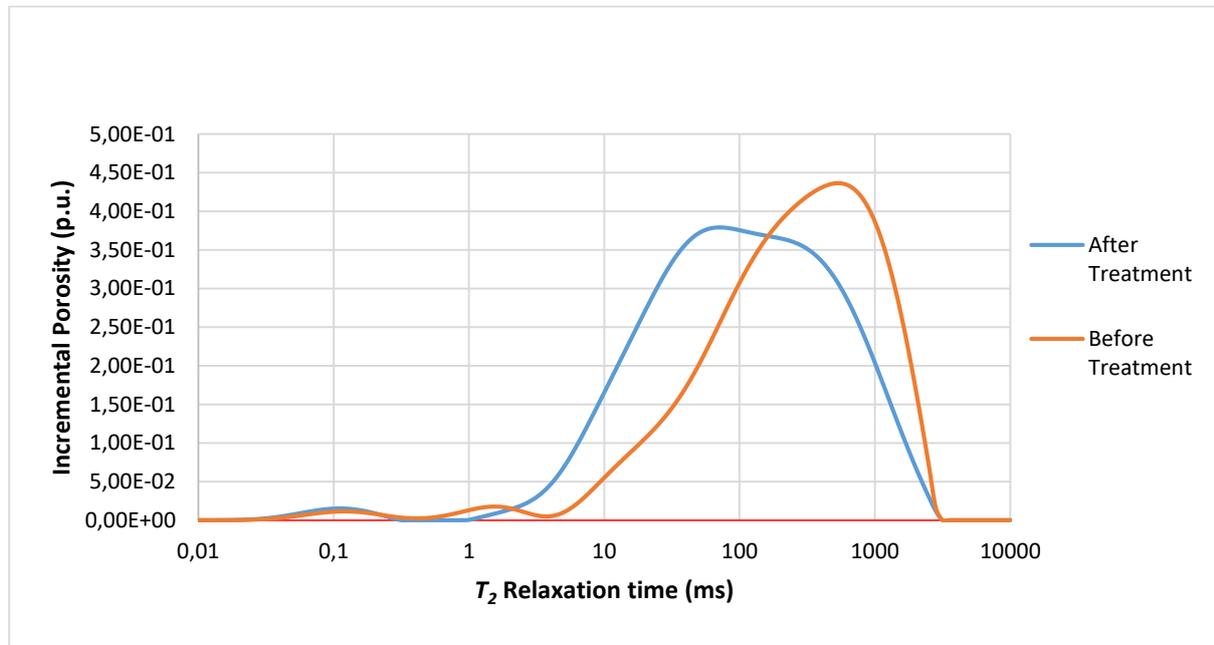
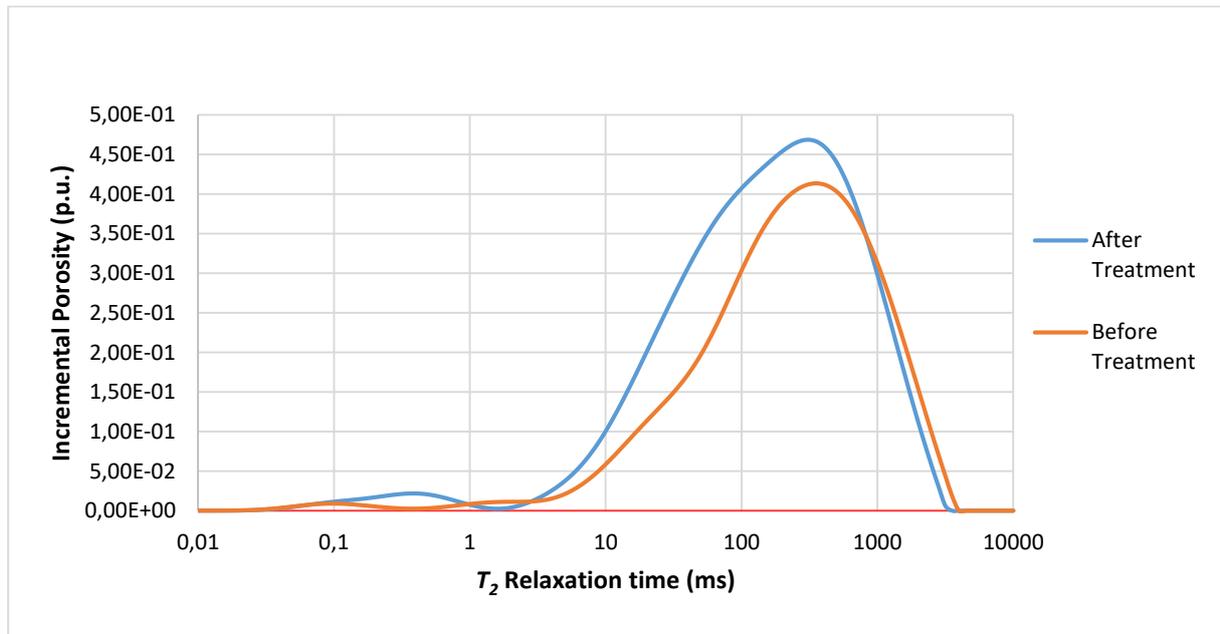


Fig. 6. T₂ relaxation time distribution of IL8

3.2.2 Core No. 12 (Injection Rate: 0.5 cc/min)

For Core No. 12, the pressure drop data shows an initial peak followed by a gradual decline with less pronounced oscillations than Core No. 8, suggesting a more uniform and gradual enlargement of pore spaces. This results in stable improvements in the pore network. The T₂ relaxation times for Core No. 12 post-treatment exhibit a moderate shift towards shorter relaxation times, with a broad peak indicating a diverse range of pore sizes. The changes are less dramatic than in Core No. 10 but more controlled than in Core No. 8, reflecting a balanced enhancement of pore sizes. This moderate injection rate allows for a controlled reaction between the acid and the carbonate matrix, resulting in a well-distributed network of small to medium pores. The balanced approach in Core No. 12 demonstrates effective pore enhancement without the extremes seen in other cores.

Fig. 7. T₂ relaxation time distribution of IL12

3.2.3 Core No. 10 (Injection Rate: 1 cc/min)

Core No. 10 demonstrates a rapid initial pressure drop followed by stabilization and a steady decline, indicating a uniform enhancement of the pore network due to the higher injection rate, accelerating the acid-rock interaction. This results in more immediate and extensive dissolution of the carbonate material. The smooth pressure curve suggests a more homogeneous modification. The T₂ distribution for Core No. 10 post-treatment shows a pronounced shift towards shorter relaxation times, indicative of an increased number of smaller pores. Compared to Core No. 8, the narrower peak suggests a more consistent distribution of pore sizes, enhancing the permeability significantly by creating a uniform network of medium-sized pores. This uniform enhancement at a higher injection rate proves highly effective in improving the rock's overall porosity and permeability.

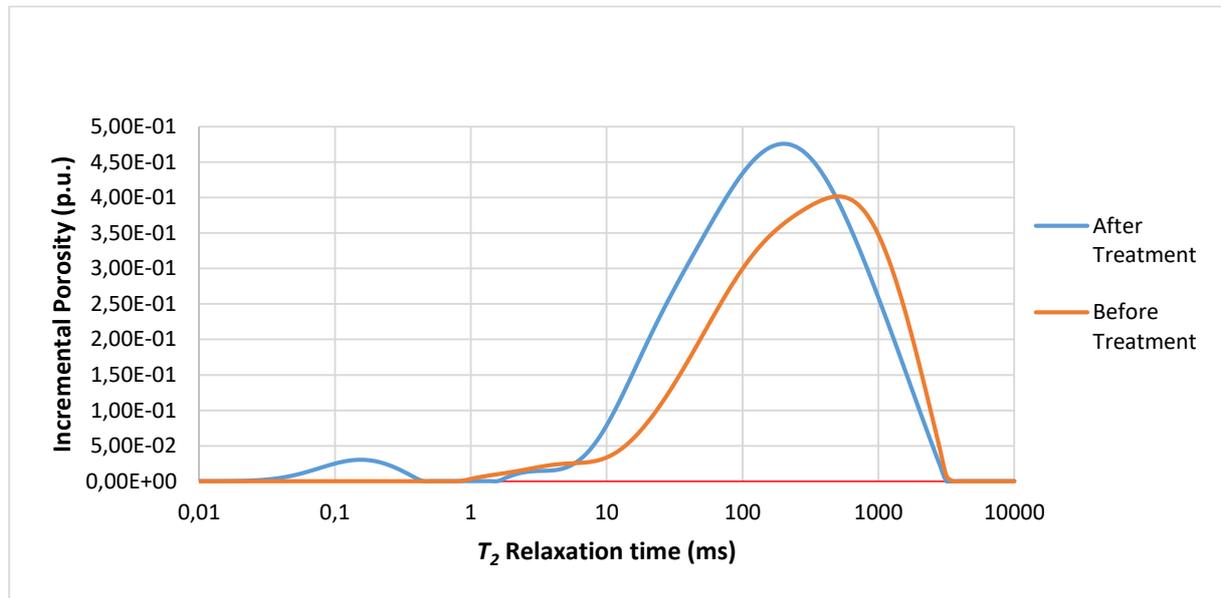


Fig. 8. T2 relaxation time distribution of IL10

3.3. Porosity and Permeability Analysis

The reaction between carbonic acid and limestone resulted in notable changes to the petrophysical properties, particularly porosity, while permeability became infinite following wormhole formation. Porosity was measured before and after flooding using helium porosimetry (AP-608), and permeability was assessed using a liquid permeameter (LP-100A). As shown in Table 3, the porosity changes were similar for injection rates of 0.2 and 1 cm³/min, while the change was less pronounced for the 0.5 cm³/min rate. The moderate flow rate of 0.5 cm³/min is less effective in increasing porosity because it may not provide sufficient time for the acid to react with the limestone fully. This results in less extensive dissolution compared to the more thorough reaction at 0.2 cm³/min and the more aggressive dissolution at 1 cm³/min.

Table 3. Porosity and permeability results

Core No	IR (cm ³ /min)	Initial ϕ %	Final ϕ %	ϕ change %	Initial K mD	Final K mD
IL8	0.2	14.91	16.01	7.4	3.4	infinity
IL12	0.5	15.32	16.05	4.8	7	infinity
IL10	1	15.17	16.27	7.3	4	infinity

4. CONCLUSION

This study comprehensively investigated the impact of carbonic acid on the porosity and pore structure of carbonate reservoirs, using Indiana limestone as a representative sample. By analyzing T₂ relaxation times and incremental porosity before and after treatment, the study aimed to understand the implications of using carbonic acid in matrix acidizing to reduce CO₂ emissions. Based on the outcomes and findings of this research, the following conclusions were drawn:

1. Applying carbonic acid treatment led to a consistent enhancement in porosity at all examined injection rates (0.2, 0.5, and 1 cm³/min). This improvement is attributed to the chemical reaction between carbonic acid and calcite in the limestone, which causes the rock to dissolve and generates extra pore spaces. The reaction also expands preexisting pores, contributing to an overall augmentation in porosity and enhanced pore connectivity.
2. The injection rate substantially impacted the degree of alteration in porosity and pore structure. Reducing the injection rates to 0.2 cm³/min facilitated a gradual and stable reaction, leading to a controlled improvement of the pore network. Increasing the injection rates to 1 cm³/min resulted in a faster and more extensive acid-rock interaction, causing the dissolution process to occur rapidly. This led to the formation of a more uniform network of larger pores. The moderate rate of 0.5 cm³/min provided a well-balanced reaction, resulting in increased pore sizes without the extreme variations at lower and higher rates.
3. The permeability of the core samples experienced a substantial increase following treatment, resulting in an effectively infinite permeability due to the formation of wormholes. These channels were created by the preferential dissolution paths of carbonic acid, which enhanced fluid flow through the rock.
4. Carbonic acid, derived from combining CO₂ and water, provides an environmentally friendly substitute for hydrochloric acid. This approach effectively decreases greenhouse gas release and minimizes tubular corrosion, offering a sustainable solution for well-stimulation.

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