



Saturn Publications

Frontiers in Engineering Science and Technology 1(2022) 2235-2241



www.saturnpublications/fest

Effect of Matrix Acidizing on Different Core Samples at Different Temperatures: An Experimental Study

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Received 15 June 2022; Accepted 23 September 2022

Abstract

The economic production of hydrocarbons from reservoirs is reliant on the natural capability of the reservoir to produce fluids (HCs) economically. However, a decrease in porosity, permeability, or both, can lead to damage near the wellbore face or deep into the rock, resulting in positive skin and resistance to fluid flow. This resistance causes a loss in production, which can be overcome by removing the impairments near the wellbore using stimulation methods. Matrix acidizing is a type of stimulation method that involves injecting an acid/solvent into the formation to dissolve/disperse materials that impair well production in sandstone reservoirs or to create new, unimpaired flow channels in carbonate reservoirs. The main aim of this study is to investigate and compare the effect of matrix acidizing on different rock formations at different temperatures, as well as to measure rock physical porosities (porosity, permeability) before and after acidizing. For this purpose, laboratory experiments were conducted on limestone (Tiyon formation) and sandstone (Gaj formation) of Thano Bulla Khan, Sindh, Pakistan. The experiments were conducted in two phases: pre-investigation of core samples and post-investigation of core samples. In the pre-investigation phase, experiments were performed before acidizing. These experiments included porosity, permeability, XRD, SEM, and EDS testing. In the post-investigation phase, experiments were performed after acidizing, except for XRD and EDS testing, which was only used for acid selection. The purpose of the post-investigation phase was to compare the results with the pre-investigation phase. Three samples of each formation were used to compare the results after injecting an acid combination (30-50ml) with a ratio of 15%HCL: 9% HCOOH, 13% CH₃COOH at 60°C, 90°C, and 120°C. The results of the study showed that the effect of matrix acidizing was greater on limestone rock samples than on sandstone rock samples. Limestone rock is composed of calcium carbonate, which reacts vigorously with acid, while sandstone forms fewer bubbles. The petrophysical properties of the rock were successfully restored and recovered by doing the matrix acidizing job. In limestone rock samples, porosity values were recovered to 20.27% -23.18%, and permeability values were recovered to 19.26 md to 24.16, 25.02, and 27.98 millidarcies at temperatures of 60°C, 90°C, and 120°C, respectively. In sandstone rock samples, porosity values were recovered to 7.5.

Keywords: Matric Acidizing; Rock Formations; Limestone Rock; Sandstone Rock; Carbonate Reservoirs.

1. Introduction

The oil and gas industries are heading towards the enhancement of recovery from low-permeable hydrocarbon reservoirs. The drilling, completion, and production activities of hydrocarbon reservoirs are the major reasons for formation damage. Due to this, damaging loss in production occurs. This can be achieved by stimulation methods by removing any disfigurement from the near wellbore. A well injection known as "well stimulation" is carried out on an oil or gas well to improve the flow of hydrocarbons from the drainage area into the well bore, increasing production. A matrix treatment increases production in both sandstone and carbonate wells by improving permeability by reducing damage near the wellbore. Although sandstone and carbonate use different acid systems, the same procedures apply to both.

In the absence of damage, the significant amount of acid needed to raise permeability around the wellbore may not be warranted the little incremental improvement in output, particularly in sandstone. Hydrochloric acid tends to generate wormholes in carbonate rock, enlarging the wellbore or causing damage to be avoided. The increase in permeability is significantly greater in carbonate than in sandstone.

Well Stimulation

Stimulation is used to boost or recover a well's production. Sometimes a well initially shows limited permeability, in which case stimulation is used to start production from the reservoir. Sometimes stimulation is utilized to increase permeability and flow further from a well that already exists but has started to produce poorly.

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The condition of the region surrounding the wellbore is one of the major constraints on establishing well productivity. By the value of "skin," all modifications to the initial construction are identified. The creation of the volume of rock with a reduced permeability in the vicinity of the wellbore region is correlated with formation damage. There are several causes for this decrease in permeability, but in every instance, it will lower "natural" production since there will be greater pressure decreases as the fluid gets closer to the wellbore.

A well's productivity can be increased by limiting the impact of formation damage in the vicinity of the wellbore or by putting a highly conductive structure on the formation. The two of the most generally used methods of well stimulation are:

- Acidizing
- Hydraulic Fracturing

Acidizing

The treatment of acidizing, a type of stimulation procedure, is carried out at a lower pressure than the reservoir fracture pressure in order to restore the naturally occurring permeability of the reservoir rock. Calcite, limestone, and dolomite cement are distributed throughout the sedimentary particles of the reservoir rocks by pumping acid into the well to produce well acidization. The use of matrix acidizing, and fracture acidizing are the two forms of acid treatment that are used.

Pumping acid into the well and the formation's pore throat enacts a matrix acidizing procedure. In this process of acidizing, the acids dissolve the mud particles and sediments while also enhancing the flow of hydrocarbons, decreasing the formation's permeability, and widening its pore throats. Fracture acidizing requires injecting high-pressure acid into the well, physically fracturing the reservoir rock, and dissolving the permeability blocking sediments. Matrix acidizing is carried out at a low enough pressure to prevent fracturing the reservoir rock. This kind of acid work creates routes for the hydrocarbons to move through.

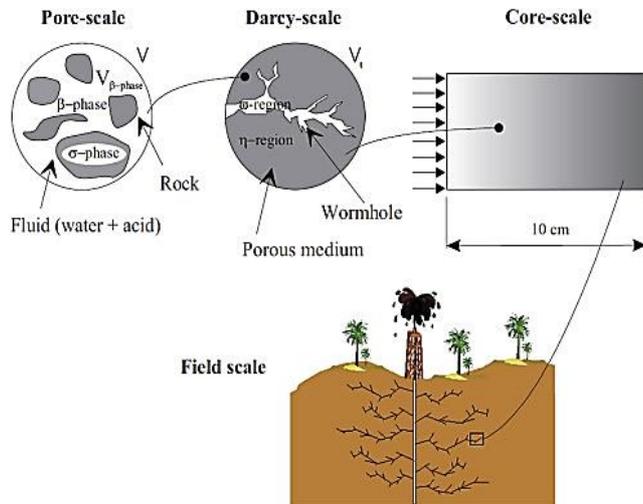


Fig. 1. Matrix Acidizing

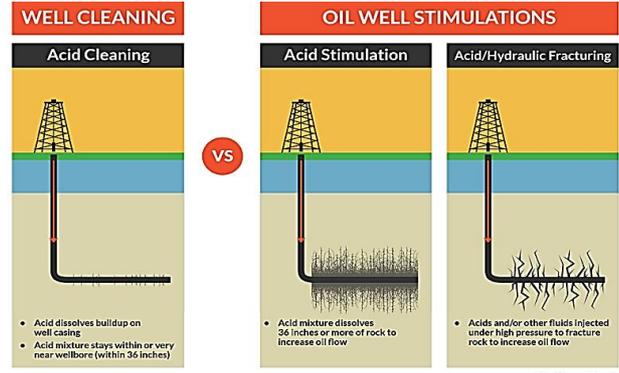


Fig. 2. Acidizing

Hydraulic Fracturing

Hydraulic fracturing is the stimulation method used by the petroleum industry the most frequently nowadays. Though educated estimations may reveal a discrepancy, the anemic claim that more than 70% of wells in North America have been fractured seems unconvincing. More than 3000 treatments have been given each month since 1955, on average. Hydraulic fracturing, which uses either acids or proppants to keep the crack open, is now the go-to method for the prompt. Acids or prop-pants fracturing is a good way to increase output in various areas. Many of the looming requirements for fracturing stimulation are formation-related, such as the uncertainty of whether the crack will spread over many formations and may interact with unwelcome water.

The four steps of the hydraulic fracturing process are as follows:

- A well is bored vertically to the desired depth, then is twisted at an angle, and continues parallel to the ground for thousands of feet into the formation thought to contain the trapped natural gas or oil.
- Natural gas or oil is released via the cracks and is brought back up the well to the surface after being pushed through a high-pressure mixture of water, sand, and chemicals into the well in order to form fissures in the shale rock.
- Following the fracturing procedure, wastewater, often known as "flow back water" or "generated water," rises to the surface.
- The oil or natural gas is gathered at the surface and is processed, refined, and shipped to the market.

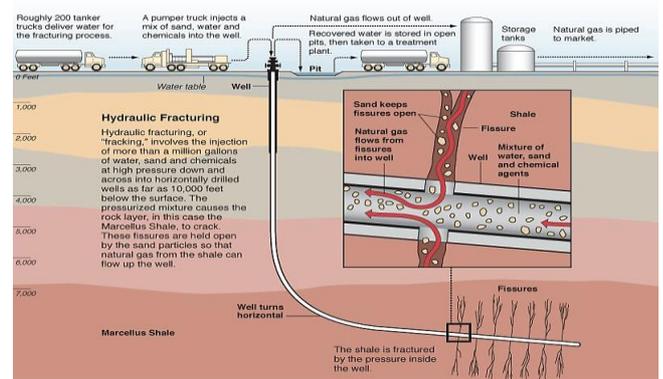


Fig. 3. Hydraulic Fracturing

Matrix Stimulation

To dissolve or scatter contaminants that hinder well production in sandstone reservoirs or to establish new, unhindered flow channels in carbonate reservoirs, matrix stimulation involves pumping an acid or solvent into the formation at a pressure lower than the formation's fracture pressure. Since mineral acids are most typically utilized in matrix stimulation, this process is also sometimes referred to as "matrix acidizing." When matrix acidizing, all the acid reacts in carbonates within a few to maybe as much as 10 feet of the wellbore. For oil wells, the lower permeability limit for matrix acidization is 10mD, and for gas wells, it is 1mD. Only a small portion of the matrix in sandstone is soluble, therefore the permeability-damaging minerals are dissolved by relatively slow-acting acid.

A/c to the thumb's rule, matrix acidizing is only used when a well has a significant skin impact that cannot be attributable to operational, surface or mechanical issues. Before attempting to remove damage by matrix acidizing, the kind (or Cause) and location of the damage must be determined. The damage identification process involves:

- Examining the well records to identify operations that might have resulted in formation damage
- Carrying out specific laboratory testing, such as a reservoir core flushing, to determine if the identified operations did indeed lead to core damage for the particular combination of the fluids in question and the reservoir formation
- Examining the damaged core with sophisticated analytical techniques such as the scanning electron microscope to confirm the damage type and location and develop ideas on how to remove it.

2. Problem Statement

When a reduction occurs in the natural capability of a reservoir to produce its fluids (HCs) economically, such as a decrease in porosity or permeability, or both, damages can occur near the wellbore face or deep into the rock. It shows that skin is damaged, either it is positive or negative, which causes the resistance to fluid flow around the wellbore or in the reservoir. Most of the hydrocarbon generating formations has been damaged, whether by drilling, completion, and production activities. This can be achieved by removing any impairment from close to the wellbore by the stimulation methods. This damage is caused by several mechanisms:

- Physical plugging of pores by mud solids
- Alteration of reservoir rock wettability
- Precipitation of insoluble materials in pore spaces
- Clay swelling in pore spaces.
- Migration of fines into pore throats
- Introduction of an immobile phase
- Emulsion formation and blockage

To remove damage near the well bore, matrix acidizing is done to eradicate this, but due to the temperature of the reservoir, it causes an unstable effect of acid in the form of precipitation, reaction with minerals, or corrosion problems.

3. Objectives of the Study

The objectives of the project are:

1. Study the effect of matrix acidizing before and after acidizing on different rock samples at different temperatures.
2. Evaluate porosity and permeability before and after acidizing.
3. Compare the results of each other before and after acidizing to differentiate the effect of matrix acidizing on them.

4. Scope of the Study

By removing damaged or creating new channels, acidizing sandstone, and limestone reservoirs is a crucial step to ensuring high output. Numerous studies have been conducted to date emphasizing the value of acidizing sandstone formations. Many researchers developed different acid combinations, and applied different chelating agents to get the best results related to

- Permeability
- Porosity
- Precipitation

Due to the limitations of the current acid combinations at high-temperature wells and the limited research on these combinations on various sandstone and limestone formations, new acid combinations are needed for future sandstone and limestone acidizing aspects, and more research is required on the current technology.

Future research should focus on creating acid mixtures that may be used effectively to reduce the problem of precipitation reaction at high temperatures. Future research on the effects of acidizing on the topology, morphology, and wettability of rock samples would greatly benefit from pore-scale imaging.

5. Literature Review

In order to properly select stimulation candidate wells, it is necessary to first have an in-depth understanding of the concepts of formation damage and well stimulation. A lot of research conducted on formation damage and well stimulation methods can be found in the literatures. Well stimulation is considered a major key to proper reservoir management; hence several authors made valid contributions.

Guo et al. (2020) took four core samples from the Eagle Ford shale formation, measuring 3.81 cm in diameter and 15.24 cm in length, and were experimentally evaluated to determine how matrix acidizing affected them. The average mineralogical compositions of the core samples were constituted by calcite (48.8%), siderite (18.1%), and dolomite (11.9%). At room temperature and atmospheric pressure, the samples were in 15% HCl for 24 hours. According to the research, the acid dissolved an average of 38% of the core samples, increasing their true porosities.

Sheng et al. (2019) using a 15% HCl and 3% KCl acid combination for 10 hours, researchers in China examined the effects of acidizing treatments on two different types of core samples from the Longmaxi marine and Yanchang

continental shale formations. Compared to the clay contents, which were 27% and 26% for the core samples from Longmaxi and Yanchang, respectively, the carbonate concentrations were 18% and 4%. Each core sample was dipped in 5 cm³ of the acid mixture for two hours before being left to dry naturally for 24 hours at room temperature (26 °C). According to the observations, carbonate minerals were dissolved by the acid, which allowed cracks to form and the pore spaces to connect. These minerals include carbonates, anhydrite, and clay, and as a result of this process, the produced tensile stress and increased the porosities of the samples.

Weldu Teklu et al. (2019) examined the effects of 1 and 3% HCl dissolved in 10% KCl (potassium chloride) brine on the porosity and permeability of 48 Canadian shale core samples from the Otter Park (14 samples), Muskwa (23 samples), and Evie (11 samples) formations. The top and bottom surfaces of the core samples were in contact with 1 or 3% HCl acid (three times the rock mass) at room temperature for four days after being covered with tape. According to the findings, HCl improved the core samples' porosity and permeability.

Wu and Sharma (2017) Studied the matrix acidizing on a single undamaged Bakken shale core. According to the XRD test results, 24% of the core sample was quartz and 27% was calcite. 50 mL of 3% HCl and 3% KCl were used to dissolve around 35% of the Bakken powder sample. The shale sample's microstructure, pore structure, and material properties were all altered by the acid. Minerals that are structurally stable in acidizing processes include clay, quartz, and organic materials. However, if the near carbonates break down in HCl, they can be removed. Acidification produced macro pores with a 120 μ(m) diameter that decreased the hardness of the shale fracture surface by 30–70% and improved fracture conductivity. The outcome was an increase in permeability and porosity, which may increase the hydrocarbon flow.

According to Khalil et al. (2017); and Khalil et al. (2020), the reservoir rock is far less permeable than the conductive flow channels (wormholes) made by matrix acidizing. As a result, near-wellbore damage and poor permeability zones can be easily overcome by fluid movement through these wormholes from the formation into the wellbore. This experimental investigation showed that performing matrix acidizing with 15% HCl on Eagle Ford core samples (a carbonate-rich shale formation) significantly increases the permeability of the core samples (from nanodarcies to microdarcies).

Zhou et al. (2016) performed core flooding tests at 25 and 65 °C with 12% HBF₄ and 12% HCl. The outcomes were contrasted with those of standard 3% HF and 12% HCl. According to the findings, the fluoroboric acid combination exhibits a 40% greater permeability enhancement than the typical mud acid. Additionally, at 65 °C, permeability enhancement is larger than at 25 °C. The research solely examined the differences between 25 °C and 65 °C.

Schmid et al. (2016) asserted that an engineer must consider all relevant data during a matrix acidizing operation, including well history, laboratory test results,

and previous operation experience, in order to choose the acidizing treatment fluid. A thorough reservoir characterization workflow method is necessary to guarantee the success of a matrix acidizing treatment. Abdelmoneim & Nasr-El-Din (2015) found the ideal HF concentration for formations of high-temperature sandstone. The core flooding tests on the Grey Berea core and Bandera core were carried out at high temperatures of 280 and 325 °F. The association between the ideal HF concentration and mineralogy was established as an inverse relationship as a result of the findings.

Similarly, Tripathi and Pournik (2014) experimentally examined the impact of matrix acidizing on four core samples from the Eagle Ford shale formation, measuring 3.81 cm in diameter and 15.24 cm in length. The majority of the core samples' average mineralogical compositions were calcite (48.8%), siderite (18.1%), and dolomite (11.9%). The samples were placed in 15% HCl for 24 hours at ambient temperature and atmospheric pressure. The research indicates that an average of 38% of the core samples were dissolved by the acid, increasing their actual porosities.

Morsy et al. (2013) gathered shale samples from Eagle Ford, Mancos, Barnett, and Marcellus. Shale formations were examined for the effects of hydrochloric acid (HCl) on porosity, sudden solubilization, and fracture spread. In that investigation, several acid concentrations of 4, 15, and 20% were examined. The results showed that the best acid concentration for increasing porosity and oil recovery without destroying the structures of the core samples was 4% HCl. Additionally, they suggested using low-concentration HCl inside the shale formation as well as around the wellbore as part of the hydraulic fracturing procedure.

Gomaa et al. (2013) stated that the change in permeability of the sandstone core matrix at a temperature of 180 °F was examined in relation to the ratio of mud acid content. There are four different mud acid concentration ratio mixtures: 1.9% HF+15% HCl, 2.3% HF+10% HCl, 2.6% HF+5% HCl, and 2.8% HF+3% HCl, respectively. The core flooding technique was used to examine each of them utilizing a core sample. According to the experimental findings, the permeability of the core sample can be positively increased by all four acid mixing ratios. However, it was found that as the HF-HCl ratio rises, the permeability result increases.

Parkinson et al. (2010) applied a different strategy to enhance the Pinda formation's production zone, which is in West Africa. The Pinda formation contained many carbonate layers. This formation's BHST (bottom hole static temperature) was 300 °F. During the main flush stage, a PH 4 HEDTA chelant was used to stimulate the six production wells from the formation zone. The outcome revealed that all six wells are now producing at a rate that has doubled since the stimulation, demonstrating a strong economic return from the high temperature stimulating acid.

Whereas. Jaramillo et al. (2010) indicated that by combining organic acid and HBF₄ to create a new acid system known as organic clay acid, it was possible to further develop the use of HBF₄ acid in the acidification of

sandstone (OCA). Many wells had been treated in low-temperature reservoirs at less than 140 °F after being stimulated with OCA. The efficiency of OCA in particle management and clay stability was demonstrated by actual field findings. It had been noted that the initial production rise on the wells stimulated with OCA was greater than the initial production increase on the wells treated with organic mud acid. This demonstrated that OCA had successfully stopped the fines migration problem produced by organic mud acid.

In a study, Ali et al. (2008), using an experimental technique, discovered that sodium hydroxyethylethylenediaminetriacetic acid (Na3HEDTA), a low-pH solution, can promote the development of high-temperature fluids in West Africa. The outcome showed that the chelating fluid is effective in raising the high-temperature well's permeability. Further, Frenier et al. (2004) found that hydrox-ethylaminocarboxylic acid (HACA)-based chelate was created and tested on Berea sandstone. The outcomes showed that the high-temperature sandstone reservoir could employ this HACA chelate.

Thomas et al. (2001) performed core flooding on Jauf core samples using HCl and acetic acid in the pre-flush at 150 °C. The channels made by the pre-flush acid, which was utilized before the mud acid, were important since they showed up in the results. The permeability had been greatly improved by channeling effects during the main acid application.

Similarly, Van Domelen and Jennings (1995) ascertain that due to the fact that it produces no insoluble products from interactions with minerals, HCl plays an important role in mud acid. Along with being advantageous in terms of cost, HCl has been used extensively for sandstone stimulation. Additionally, and especially in high temperature, high-pressure (HTHP) formation environments, HCl is extremely corrosive and dangerous to the well.

Ayorinde et al. (1992) demonstrated the benefit of HBF4 in treating an oil well in Nigeria that had had serious fines migration-related problems brought on by traditional mud acid. HBF4 had indicated compatibility with fines migration stabilizing. 850 barrels of liquid are produced by the oil well per day after it has been acidified with mud acid (BLPD). However, because to fines migration, production decreased to almost nothing. Following a successful HBF4 treatment, production rose to 2500 BLPD and remained constant at 220 barrels of oil per day (BOPD) even after a year.

6. Methods

Area of Study

Our research study is based on rocks of two different formations, limestone is from the Tiyon formation, and sandstone is from the Gaj formation. Both formations belong to Thano Bulla Khan. It is located at 25°24'34.9"N 67°46'27.1"E Jamshoro, Sindh, Pakistan. The main terrain of the region is made up of hills and valleys that alternate, with their long axes running north to south. The region's

geology is made up of marine-derived sedimentary formations as well as certain volcanic sills, which are injected with igneous bodies horizontally. With geological ages ranging from recent to Cretaceous, lithology materials such as sand, clay, silt, gravel, limestone, conglomerate, sandstone, and shale are discovered. The area is complicated structurally because beds exhibit severe dips, main structure limbs either have opposing inclinations or show dipping in the direction of one another, and thrust faulting, synclines, and anticlines are all clearly apparent. The valley is covered with substantial amounts of alluvium, which includes sand, silt, clay, and gravel. In the display, the region's geology is described.

Collection and Cutting of Rock

We have collected two surface rock samples (limestone and sandstone) from Thano Bulla Khan Jamshoro, Sindh, Pakistan. Limestone from the Tiyon formation and sandstone from the Gaj formation. After collecting rock samples, Cut the rock samples into a finely cylindrical shape by using a drill press (3 samples of each rock with the core length of 1.5 inches and diameter of 1 inch).

Preparation of Core Samples

First of all, we arranged two different rock samples, i.e., limestone and sandstone, for the matrix acidizing job and then cut them into small core samples in a fine cylindrical shape by using the drill press to perform acidizing experiment on it. Cut in a way the size of samples should be equal. We took all samples to the laboratory and ensured samples were clean and under the same conditions at room temperature and pressure. We use three small core samples (1.5 inches) of limestone and three small (1.5 inches) core samples of sandstone.

Pre-Investigation of Core Samples

After cutting rock samples, the core samples are further treated in the laboratory for pre-investigation of core plugs. In this phase, we did five tests for all given samples.

- i. XRD (X-ray Diffraction)
- ii. SEM (Scanning Electron Microscopy)
- iii. EDS (Energy Dispersive X-ray Spectrometry)
- iv. Porosity measurement
- v. Permeability measurement

Post Investigation of Core Samples

After Acid Preparation, the core samples are further treated in the laboratory for Post investigation of core plugs/samples. In this phase, we inject a combination of acids into core samples by using a syringe at different temperatures (60°C,90°C,120°C), and then we go for post-investigation and do three tests for all given samples.

- i. SEM (Scanning Electron Microscopy)
- ii. Porosity measurement
- iii. Permeability measurement

7. Results and Comparison

Throughout the project, we have done many experiments and laboratory testing to investigate and compare the effect

of matrix acidizing before and after acidizing on different core samples at different temperatures and found the following results.

Name of samples	Before Acidizing		After Acidizing			
	Porosity (%)	Permeability (md)	Porosity (%)	Permeability (md)		
				60°C	90°C	120°C
Limestone	20.27	19.26	23.18	24.16	25.02	27.98
Sandstone	7.5	14.82	9.47	19.68	19.45	20.22

Table 1. Comparative Chart of Porosity and Permeability of Sandstone before and After Acidizing

Our basic objectives were to increase porosity and permeability. We prepared acid combination by analyzing XRD and EDS testing and found the following:

- Limestone is mostly composed of calcite and magnesium minerals with a percentage of 69.29%, and the shape of the crystal is rhombohedral.
- And sandstone is mostly composed of calcite magnesium (74%) and dolomite (24%) minerals, and the shape of the crystal is rhombohedral.

After we performed the matrix acidizing job on samples and found the expected results as

- The initial average porosity of limestone and sandstone was 20.27% and 7.5%, respectively, and after acidizing job, it increased with the percentage of 3% and 2%, respectively, compared to its initial value, which is quite better.
- The initial permeability of the limestone was 19.26 md, and after acidizing job, we found that permeability increases as temperature increases. final permeability of limestone at 60°C, 90°C, and 120°C is 24.16, 25.05, and 27.98 md respectively.
- Similarly, the initial permeability of sandstone was 14.82, whereas the final permeability was 19.68, 19.45, and 20.22 md at temperatures 60°C, 90°C, & 120°C, respectively.
- Comparative graphs of porosity and permeability are given below.

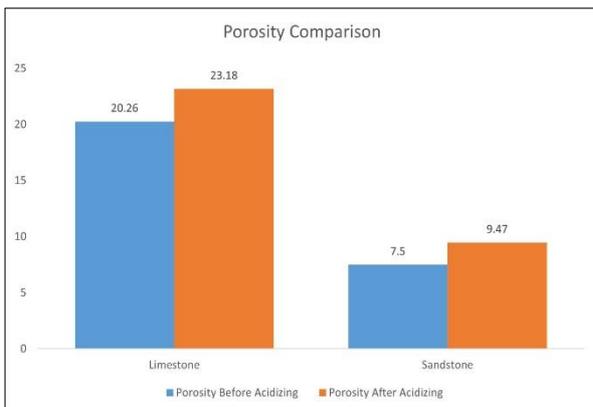


Fig. 4. Comparative Graph for Porosity

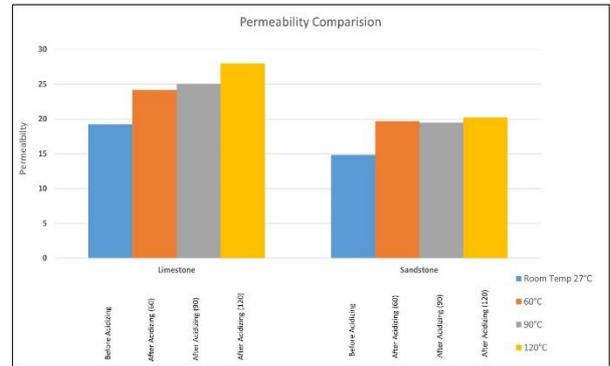


Fig. 5. Comparative Graph Permeability

8. Conclusion

Our main objectives were to increase the porosity and permeability of the reservoir after acidizing on different core samples at different temperatures.

- It is concluded that this acid combination gives the best results in limestone formation at high temperatures as it increases porosity and permeability up to 25% and 30%, respectively.
- The result is clearly seen that acid has reacted more effectively with limestone rock samples than with sandstone.
- The acid dissolves minerals, and no corrosion problems occur in limestone.
- While this acid combination is not recommended for sandstone formation due to precipitation which can block perforations and pore throat, this research provides strong evidence and complement. The methodology we use will help for the modern application in production engineering as well as it helps to explore a greatly enriched area in hydrocarbons.
- We must enhance the fieldwork for development, which is heavily focused on observing and interpreting the geological information present in the outcrop of Thano Bulla Khan.
- This project recommends upcoming changes and challenges in the domain of matrix acidizing and their performance for porosity and permeability increase (wormhole effect).

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