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RESEARCH ARTICLE OPEN ACCESS

BEST LABORATORY PRACTICES FOR REDUCING UNCERTAINTY IN THE PRESSURE DIFFERENTIAL (ΔP) MEASUREMENTS IN CORE-FLOODING TESTS FOR THE PETROPHYSICAL CHARACTERIZATION OF RESERVOIRS

Jorge Alberto Rojas P.[1], Jenny Paola Rueda M.*[2], Jorge Alejandro Sarmiento G.[3], Pedro Juan Rojas M.[4], Jose Luis Mendoza[5], Cesar Augusto Romero R.[6] and Carlos Humberto Amaya A.[7]

[1, 2] Ecopetrol, Technology and Innovation Center, ICP, Colombia.
[3, 4, 5, 6] PSL, Proanálisis, Colombia.
[7] Industrial University of Santander, Colombia.

[1] http://orcid.org/0000-0003-2084-2611, [2] http://orcid.org/0000-0002-4243-2288, [3] http://orcid.org/0000-0003-3595-5272, [4] http://orcid.org/0000-0002-0712-268X, [5] http://orcid.org/0000-0003-0373-9786, [6] http://orcid.org/0000-0001-7775-3527, [7] http://orcid.org/0000-0002-9557-7800

Email: jorgeal.rojas@ecopetrol.com.co, *jennypa.rueda@ecopetrol.com.co, alejosarmientogalindo@gmail.com, pedro.rojas67@hotmail.com, jlmendoza.33@gmail.com, carr1214@yahoo.com, carlosh.amaya@correo.uis.edu.co

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ABSTRACT

Special Core Analysis (SCAL) as a tool for characterization of conventional reservoirs in the laboratory reduces the petrophysical uncertainty and optimizes the resolution and precision of rock-fluid petrophysical models to refine reserve estimates, calculate oil in place, and select the best oil-field development mode. Within the SCAL program, measurements of fluid flow capacity through porous media (permeability [Darcy]) are used to characterize geological formations of interest, with the fluctuation of "pressure differential" values being decisive in the calculation of this property. This research focuses on determining the sources that generate these fluctuations and implements a significant reduction by varying certain parameters in the laboratory. Through detailed analysis of the experimental procedure and equipment operation, as well as a statistical analysis, the sources impacting the quality of the measurements were identified. It was found that the precision and accuracy of pressure differential values were considerably low and directly related to: (1) the pressure pulses created in the backpressure system; (2) the high dead volume in the signal conduction lines (piping); and (3) the influence of temperature variation of the measurement environment. Modifications were implemented both in the experimental protocol and the core-flooding equipment, and tests were conducted under the same conditions to observe the changes produced. It was possible to significantly reduce the standard deviation in the measurements of the pressure differential value from 7.08% to 1.45% with respect to the mean. Consequently, these modifications reduced data dispersion, obtaining more-accurate readings of the stability behaviour of the pressure differential value and, thus, generating more-reliable results. The relevance of this work is demonstrated by the improvement in quality and reliability of petrophysical measurements, in addition to the optimization of turnaround times considering that, by reducing dispersion of pressure differentials values, flow stability state in the porous medium is reached in less time, which is a critical condition for permeability evaluations.

I. INTRODUCTION

This study responds to the need to ensure the quality and reliability of the measurements in the coreflooding tests carried out in the laboratories of Ecopetrol's Innovation and Technology Center ICP, likewise due to the representativeness of the results it is suggested if this is the case to apply in external laboratories where *Coreflooding* tests are conducted.

Previous observations [1-2] revealed parameters in the measurements that impact the accuracy and precision of the coreflooding tests and, consequently, the reliability of the results. Therefore, through the development of numerous studies that have been carried out in the laboratory, the causes of uncertainty were identified, solutions were sought to reduce it, and protocols for *coreflooding* tests were adjusted and implemented.

II. EXPERIMENTAL UNCERTAINTY SOURCES

According to statistical studies, no physical quantity can be measured with perfect certainty; there are always errors in any measurement, e.g. [3,4,5]. This means that if you measure a quantity and then repeat the measurement, you will surely get a different value the second time. However, if extreme care is taken in the measurements and more refined experimental methods are applied, errors can be reduced and therefore greater confidence or certainty in the measurements can be obtained.

II.1 ERROR, ACCURACY AND PRECISION

According to ***ASTM E177-20*** *(Use of the Terms Precision and Bias in ASTM Test Methods)* [6] *and* ***ASTM E456*** **13a-2022** (*Standard Terminology Relating to Quality and Statistics)* [7], the **error** of result, is a test result minus the accepted reference value of the characteristic, where ***accepted reference value***, is a value that serves as an agreed-upon reference for comparison, and which is derived as: (1) a theoretical or established value, based on scientific principles, (2) an assigned or certified value, based on experimental work of some national or international organization, or (3) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group.

The experimental error itself is quantified by its accuracy and by its precision, where ***accuracy*** is the closeness of agreement between a test result and an accepted reference value and involves a combination of a random component and of a common systematic error or bias component, ***precision*** is the closeness of agreement between independent test results obtained under stipulated conditions and depends on random errors and does not relate to the accepted reference value [6].

Precision is also known as repeatability or reproducibility. A measurement with a high repeatability tends to give values that are very close to each other.

II.2 TYPES AND SOURCES OF EXPERIMENTAL ERROR

An estimate of measurement uncertainty would be required in a case, in which the uncertainty is an indication of the magnitude of error associated with a value that considers both systematic errors and random errors associated with the measurement or test process [7].

As indicated by Taylor 1997 [3]., experimental uncertainties that can be revealed by repeating the measurements are called **random errors**; those that cannot be revealed in this way are called ***systematic***. In practice, since a "***true***" or "***correct***" value cannot be absolutely determined, an accepted **reference value** can be used instead for the comparison [8]. In the Figure 1 is explained the terminology from the typical example (e.g., [3], [8]) which use the pattern shots on a target where *the center of the target represents the true value of a measurand*, and results of repeated measurements are compared to shots at the target. Where **Trueness** (the closeness of agreement between the population mean of the measurements or test results and the accepted reference value [7]) improves with the positive direction of the y-axis as it moves up, moving the average results towards the center of the target) [8].

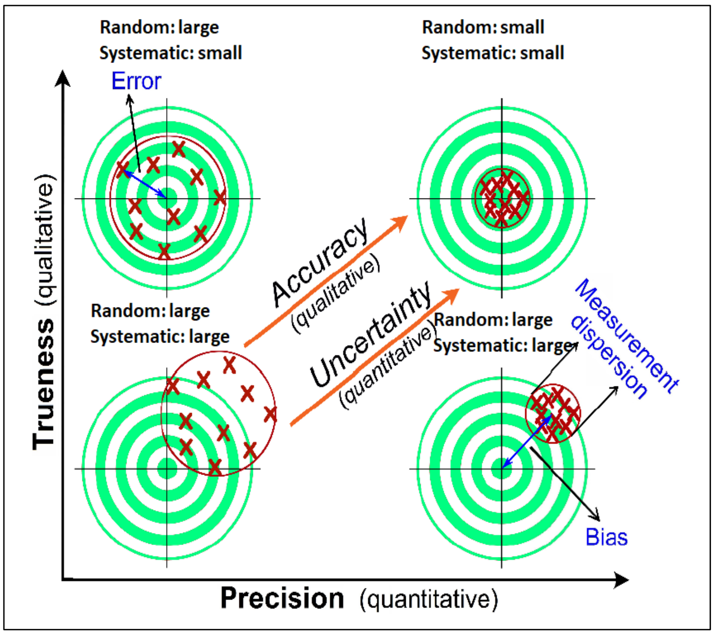

Figure 1: Accuracy and Precision, Systematic and Random Error. With increasing trueness and increasing precision, there is an increasing accuracy and a decreasing uncertainty.
Source: Modified [3, 8].

When the results are offset from the center in the same area of the target, as in the lower quadrants, there is a significant *systematic error*, or ***bias*** *(the difference between the expectation of the test results and an accepted reference value)*, which is shown in the lower right quadrant as the difference between average results and the center of the target. The upper left quadrant demonstrates a number of results exhibiting no significant bias for their average result but large random error for individual trials, and the error of a result is shown as the difference between a single result and the center of the target. The lower left quadrant shows a set of results having both large systematic and random error. In contrast, the upper right quadrant demonstrates a number of results having small random error and no significant bias [8].

Random error of result, is a component of the error that, in the course of a number of test results for the same characteristic, varies in an unpredictable way while systematic error of result,is a component of the error that, in the course of a number of test results for the same characteristic, remains constant or varies in a predictable way [6].

Systematic errors [5] have a non-random character and distort the result of a measurement. They result from erroneous calibration or just from a lack of proper calibration of a measuring instrument, from careless measurements (uncorrected parallax, uncorrected zero-point deviations, time measurements uncorrected for reaction time, etc.), from impurities in materials, or from causes the experimenter is not aware of. The latter are certainly the most dangerous type of error; such errors are likely to show up when results are compared to those of other experimentalists at other laboratories. Therefore independent corroboration of experimental results is required before a critical experiment (e.g. one that overthrows an accepted theory) can be trusted. These types of errors are corrected or minimized by predicting the behavior of the experimental process. [4].

Random errors [5] are unpredictable by their very nature. They can be caused by the limited precision of instrumental readings, but are ultimately due to physical noise, i.e. by natural fluctuations due to thermal motions or to the random timing of single events. Since such errors are unavoidable and unpredictable, the word "error" does not convey the proper meaning and its prefer to use the term uncertainty for the possible random deviation of a measured result from its true value. Statistical methods are applied to determine its magnitude. [4].

If a measurement is repeated many times, the results will show a certain spread around an average value, from which the estimated inaccuracy in the average can be determined. The probability distribution, from which the measured values are random samples, is supposed to obey certain statistical relations, from which rules to process the uncertainties can be derived. In the case of a single measurement one should estimate the uncertainty, based on knowledge of the measuring instrument [5].

II.3 CALCULATION OF THE EXPERIMENTAL ERROR

An error of theoretical importance [4], is the basis for the development of the theory of errors and their propagation, is the real uncertainty or real error, Ex, of a x number –result of a measurement or a calculation–, defined as: the difference between the values: real, X, and approximate or measured, x, that is: (1).

$$Ex = X - x \qquad (1)$$

where: X = numerical data obtained through a measurement or calculation, but which remains constant during the measurement process (wherefore the name real value), and x = measured value or numerical value obtained through a calculation.

II.4 THE MEAN AND THE STANDARD DEVIATION

Mean or ***Arithmetic Average,*** of a population, **μ**, average or expected value of a characteristic in a population – of a sample, $\bar{x}$, sum of the observed values in the sample divided by the sample size. The mean is a measure of centrality or central tendency of a distribution of observations. It is most appropriate for symmetric distributions and is affected by distribution nonsymmetry (shape) and extreme values [9, 10]. The calculation of the mean is the sum of the n sample values divided by the number of values, n. This equation is (2):

$$\bar{x} = \frac{1}{N}\sum_{i=1}^{N} x_i = \frac{1}{N}(x_1 + x_2 + \cdots + x_{N-1} + x_n) \qquad (2)$$

Where x_i is the i-th value of x measured.

Standard deviation—of a population, **σ**, the square root of the average or expected value of the squared deviation of a variable from its mean; —of a sample, **s**, the square root of the sum of the squared deviations of the observed values in the sample from their mean divided by the sample size minus 1 [10].

$$\sigma_x = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(x_i - < x >)^2} \qquad (3)$$

The meaning of the standard deviation is observed in figure 2, which shows data with a mean **μ**, as it is observed, the greater the standard deviation, the greater the dispersion of the data around the mean. The normal distribution is a symmetrical, bell-shaped curve and is completely determined by its mean, μ, and its standard deviation, σ. The parameter μ locates the center, or peak, of the distribution, and the parameter σ determines its spread. The distance from the mean to the inflection point of the curve (maximum slope point) is σ.

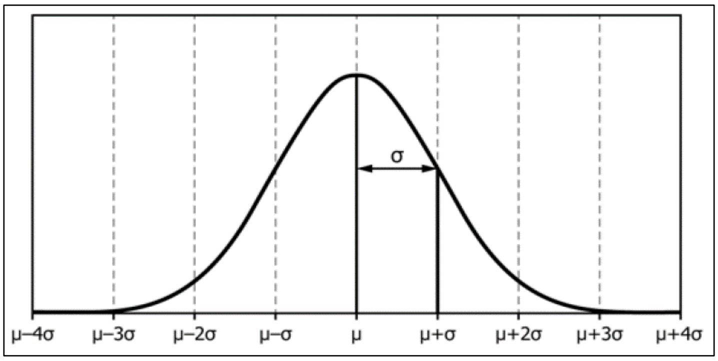

Figure 2: Normal Distribution and Relationship to Parameters μ ($\bar{x}$) representing the "center" and σ (s) representing the "spread".
Source: [10].

For measurements with only random errors, as seen in Figure 3, 68% of the data values are in an interval around mean ± 1 standard deviation; 95% of the data values are in an interval around mean ±2 standard deviations, 99.7% of the data values are in an interval around mean ±3 standard deviations.

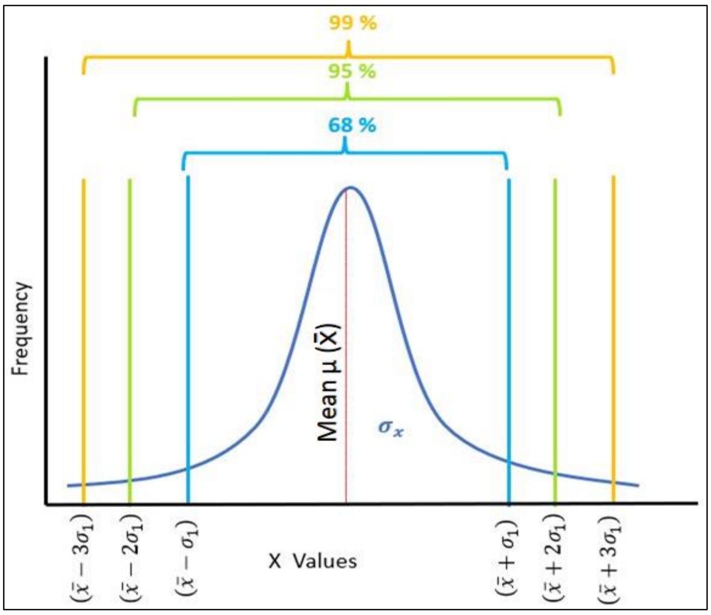

Figure 3: Probabilities in the normal distribution. 99%, 95% y 68%.
Source: Authors based on [9].

II.5 REPORT OF RESULTS OF EXPERIMENTAL MEASUREMENTS

When the result of the experimental measurement of a quantity x is reported, the result should be reported in two parts. First, the best estimate of the measurement is reported. The best estimate of a set of measurements is usually reported as the mean μ ($\bar{x}$) of the measurements. The variation in measurements is usually reported as the standard deviation of the measurements. The measured quantity may then be presented in the following ways:

- $\bar{x} \pm (\sigma_x)$ for 68% confidence
- $\bar{x} \pm (2 * \sigma_x)$ for 95% confidence
- $\bar{x} \pm (3 * \sigma_x)$ for 99% confidence

The point of using a statistic is to summarize the data set and estimate a corresponding population characteristic or parameter, or to test a hypothesis [10].

III. MATERIALS AND METHODS

The laboratories where rock-fluid interaction is evaluated base their experiments on the forced flow of fluids through a porous medium. This porous medium is usually a rock sample taken from a core or sometimes the sample is a sand pack. These laboratory tests are usually known as *coreflooding.*

In the laboratory, coreflooding tests are made to carry out the petrophysical characterization of a Formation of interest, through a formation water sensitivity analysis, critical rates, relative permeabilities; coreflooding tests are also carried out for recovery tests, optimization, and productivity of wells. Therefore, in this report, the term *coreflooding* is used to refer to any of these experiments.

The purpose of this work is to evaluate the quality of the measurements during a coreflooding. The most determining measurement in coreflooding is the pressure differential or pressure drop (ΔP) across the sample. The pressure differential measurements is used to calculate the permeability of the rock using Darcy's law (4), Fundamental law governing the flow of fluids through porous media, formulated by civil engineer Henry Darcy in 1856 [11, 12, 13] based on his experiments in filtration of water in vertical sand beds.

$$Q = \frac{-KA}{\mu}\frac{(P_b - P_a)}{L} \quad (4)$$

Where **Q** is the volumetric flow rate [m^3/s], **K** is the permeability of the porous medium [mD] **μ** is the viscosity of the fluid [Pa·s], **A** is the cross-sectional area of the rock [m^2], **L** is the length of the sample [m], and the pressure drop through the medium (**Pb-Pa**) or pressure differential **ΔP** [Pa].

III.1 DESCRIPTION OF THE RESULTS

A history of the results obtained in the experimental area of Special Core Analysis of the Production Optimization and improved recovery laboratory at the Ecopetrol Innovation and Technology Center, ICP, was reviewed; as an example, the results obtained in a sensitivity test of the Formation to the injection of production water in a sample of a well from the Acacías field located in the Llanos Orientales basin in Colombia are shown.

The data in Figure 4 shows the behavior of the pressure differential during the injection of production water at a flow rate of 1 cc/min in a stable flow period.

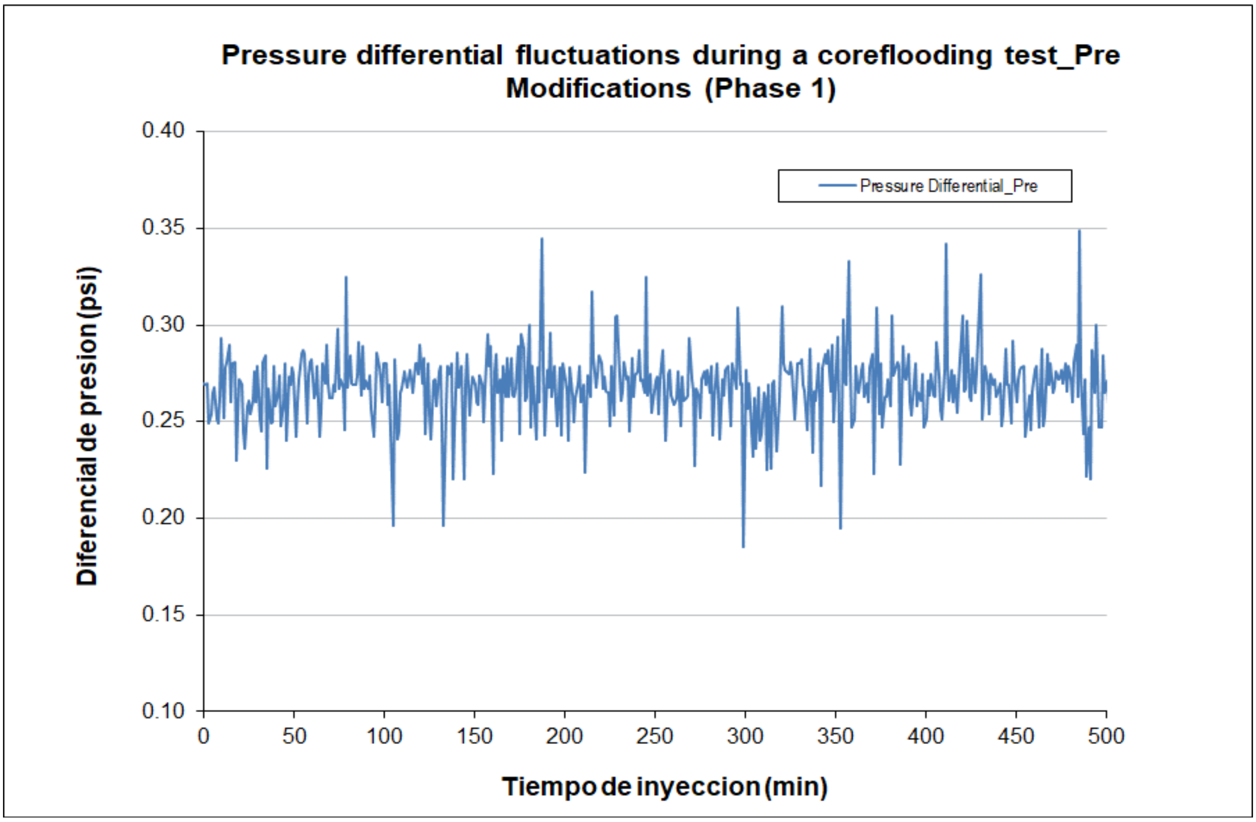

Figure 4: Pressure differential fluctuations during a coreflooding test.
Source: Authors, (2022).

As observed in Figure 4, there are fluctuations in the pressure differential in the range of 0.18 - 0.35 psi. Considering that water is being injected at a constant flow, there is an incompressible single-phase flow at a steady state and therefore the pressure differential would be expected to remain constant.

In order to objectively evaluate these fluctuations, it is necessary to perform a statistical analysis and determine the standard deviation of the data. Table 1 presents a basic statistical analysis for a sample of pressure differential data during a sensitivity test.

Table 1: Initial statistical analysis (Phase 1).

variable	valor
Mean	0.2678
Typical error	0.0008
Median	0.2690
Mode	0.2800
Standard deviation	0.0190
Sample variance	0.0004
Kurtosis	3.6620
Asymmetry coefficient	-0.0048
Range	0.1640
Minimum	0.1850
Maximum	0.3490
Addition	139.8100
Account	522.0000
Confidence level (95.0%)	0.0016
Confidence interval 95%	0.0379
Standard deviation in percentage	7.0810
Data outside CI (95%)	29.0000
Data outside CI %	5.5556

Source: Authors, (2022).

The data evaluated follow a normal distribution as shown in figure 5, with a mean of 0.2678 psi, standard deviation of 0.019 psi, taking then that the 95% confidence interval will be ± 0.038 as seen in (5):

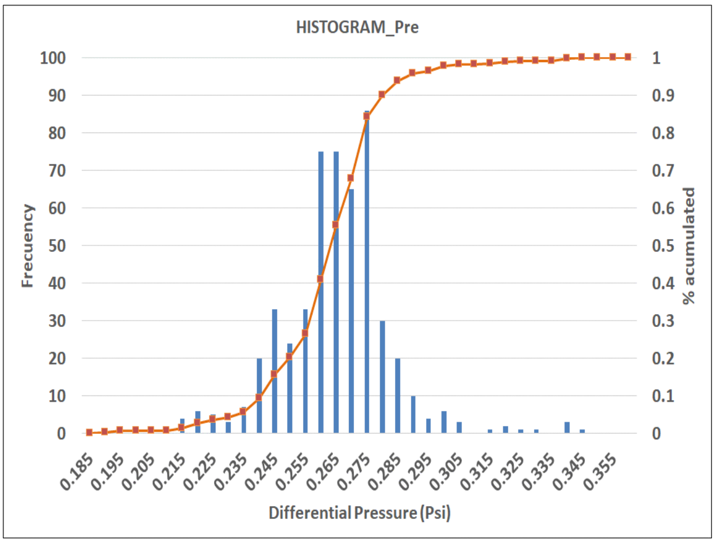

Figure 5: Distribution of pressure differential values (Phase 1). Source: Authors, (2022).

$$\bar{x} \pm (2 * \sigma_x) = 0.2678 \pm (2 * 0.019) = 0.2678 \pm 0.038\, psi \quad (5)$$

In figure 6, the moving mean for groups of 14 data was calculated and the confidence interval (95%) was estimated. For practical purposes, the confidence interval on the graph means that it's 95% sure that the measurement will fall in that range, between the two green lines. If we express the standard deviation as a percentage, we observe that the uncertainty of the data in the experiment is 7.08%, which considering the absolute error of the SMAR (pressure differentials sensor) measurement instrument with a precision of ±0.006 psi or 0.001% is substantially high.

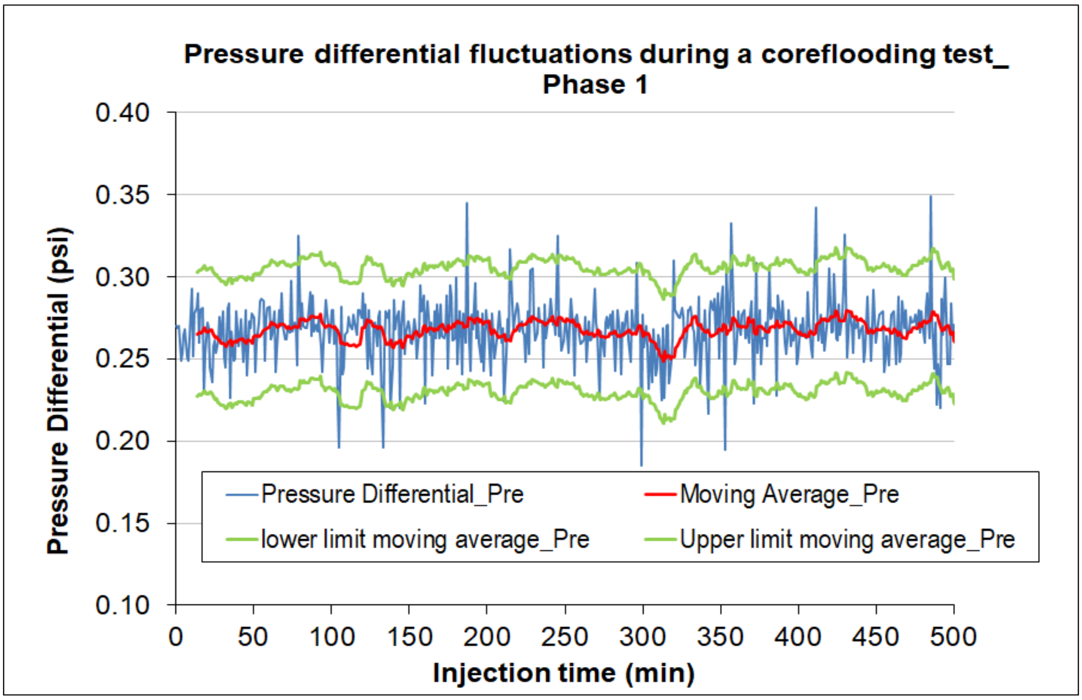

Figure 6: Confidence level (95%), for the pressure differential measurements. Source: Authors, (2022).

In order to reduce the uncertainty in the measurements, the causes were investigated by reviewing the procedures and experimental configuration of the coreflooding equipment.

III.2 IDENTIFICATION OF THE CAUSES OF DATA DEVIATION

Figure 7 schematically presents the configuration of the coreflooding equipment where the production water sensitivity test was carried out. Basically, the equipment consists of a pump that injects the fluid into the confined rock sample in the coreholder, with a pressure established in the backpressure (counterpressure or Pore Pressure). The coreholder and the cylinder containing the injected fluid are inside the furnace at the test temperature. The injection rate, the backpressure or pore pressure, the confinement pressure of the sample and the temperature of the test are established according to the case study and the conditions that this requires, generally they are the reservoir conditions or very similar to these.

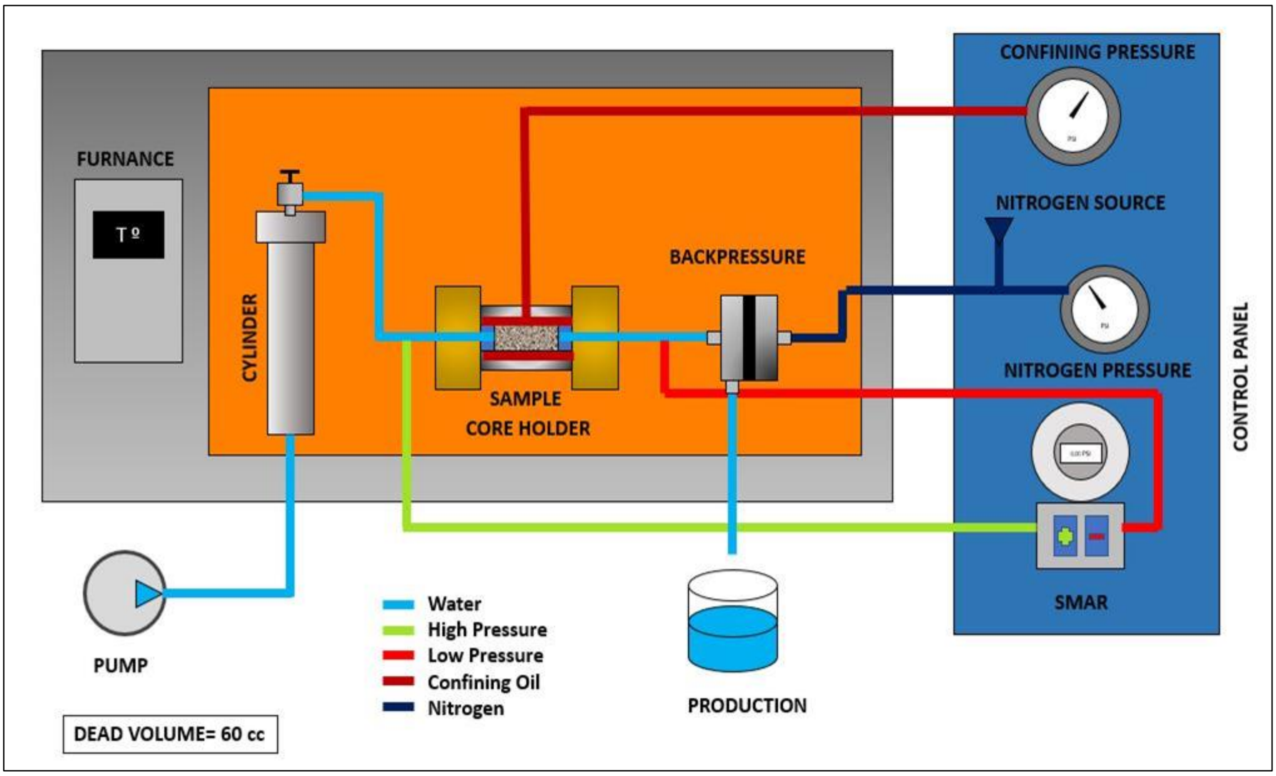

Figure 7: Schematic representation of the permeameter.
Source: Authors, (2022).

The first observation was related to the backpressure (Pore Pressure) used, those used in some tests did not have the characteristic of the gas dome type. The gas dome type has a pressurized nitrogen chamber at working pressure that cushions the fluctuations typical of a backpressure valve. Without this damping, the pressure pulses produced in the backpressure travel in countercurrent and are reflected in the pressure differential measurement.

A second observation was identified in the excess dead volume of the ducts that carry the signal from the sample to the instrumentation. The pressure sensors, both differential and backpressure and confinement, are located in a control panel attached to the equipment. Therefore, the analog signal is transmitted with mineral oil as the medium, from the coreholder to the control panel through 1/8" steel lines. This situation makes the thermal expansion of the fluid representative and is probably influencing the measurement.

A third observation is related to the room temperature or temperature at which the laboratory is located at the time of carrying out the tests. These types of tests are of long duration and must be carried out without interruption. Therefore, the laboratory temperature must also remain constant during the test. However, the air conditioning it is turned off at night and due to changes in temperature, although the furnace temperature remains constant, fluctuations in the confining pressure and changes in the mean value of the pressure differential are observed.

III.2 IMPLEMENTED IMPROVEMENTS

Figure 8 shows a scheme of the equipment with the modifications made by the laboratory personnel to reduce possible sources of error. The changes made in accordance with the recommendations were:

1. Installation of a cylinder with pressurized nitrogen at working pressure in line with production before backpressure. In this way the pressure pulse generated in the backpressure valve is damped inside the cylinder and prevents it from affecting the pressure differential.

2. The length and diameter of the signal lines to the control panel were reduced. The Smar was installed directly on the furnace wall, the flow lines were optimized thus reducing the dead volume of the equipment considerably.

3. To the third effect it was tested 1. the steel lines that lead to the signal of pressure differential were replaced by Teflon lines, since Teflon is a thermal insulator, the effect of room temperature variations on the pressure differential is substantially reduced. It must be taken into account that the maximum pressure that Teflon resists is 800 psi; consequently, it is necessary to prevent injection or displacement pressure from exceeding this limit. 2. Given the

cases in which higher pressures are required, an independent air conditioning system was structured for the laboratory to control the environmental conditions and eliminate this effect. In laboratories where it is not possible to control the air conditioning, it is recommended to cover the steel lines with thread sealant tape or Teflon tape.

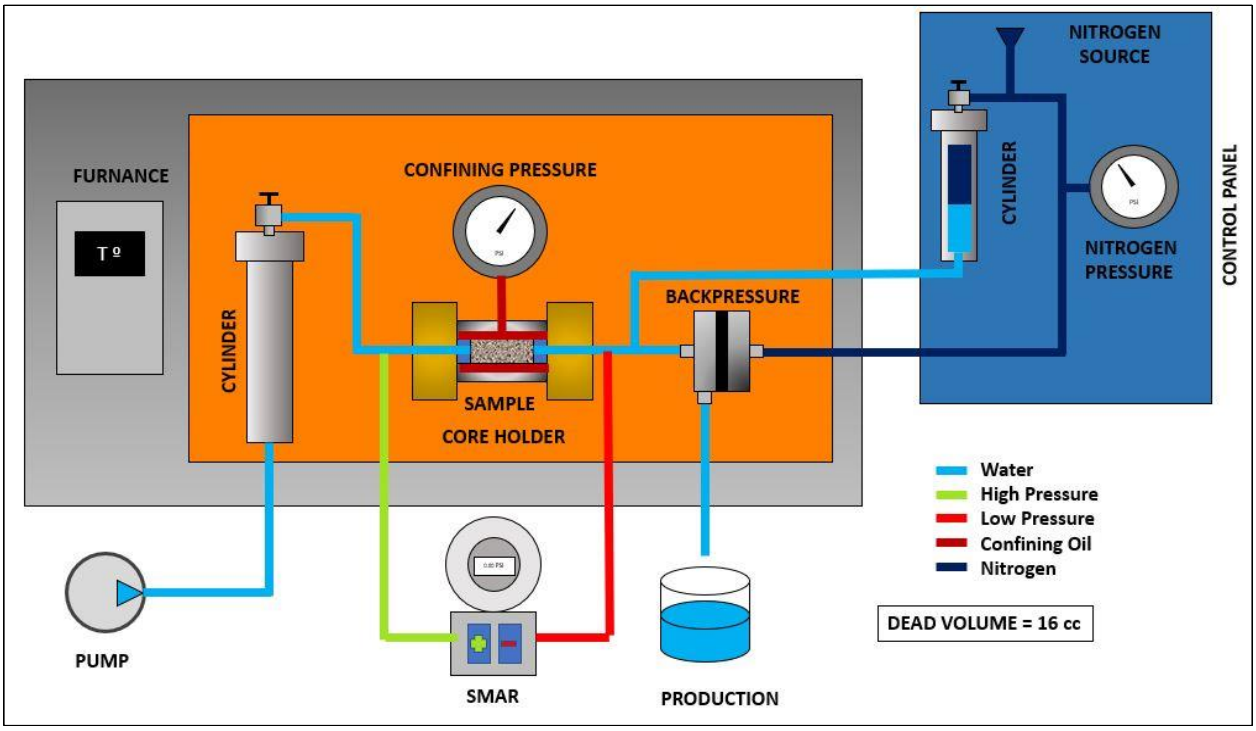

Figure 8: Diagram of the permeameter after modifications.
Source: Authors, (2022).

IV. RESULTS AND DISCUSSIONS

Once the modifications to the different permeameters were implemented, the experiment was resumed and the test continued under the same conditions. Figure 9 shows the new behavior of the pressure differential.

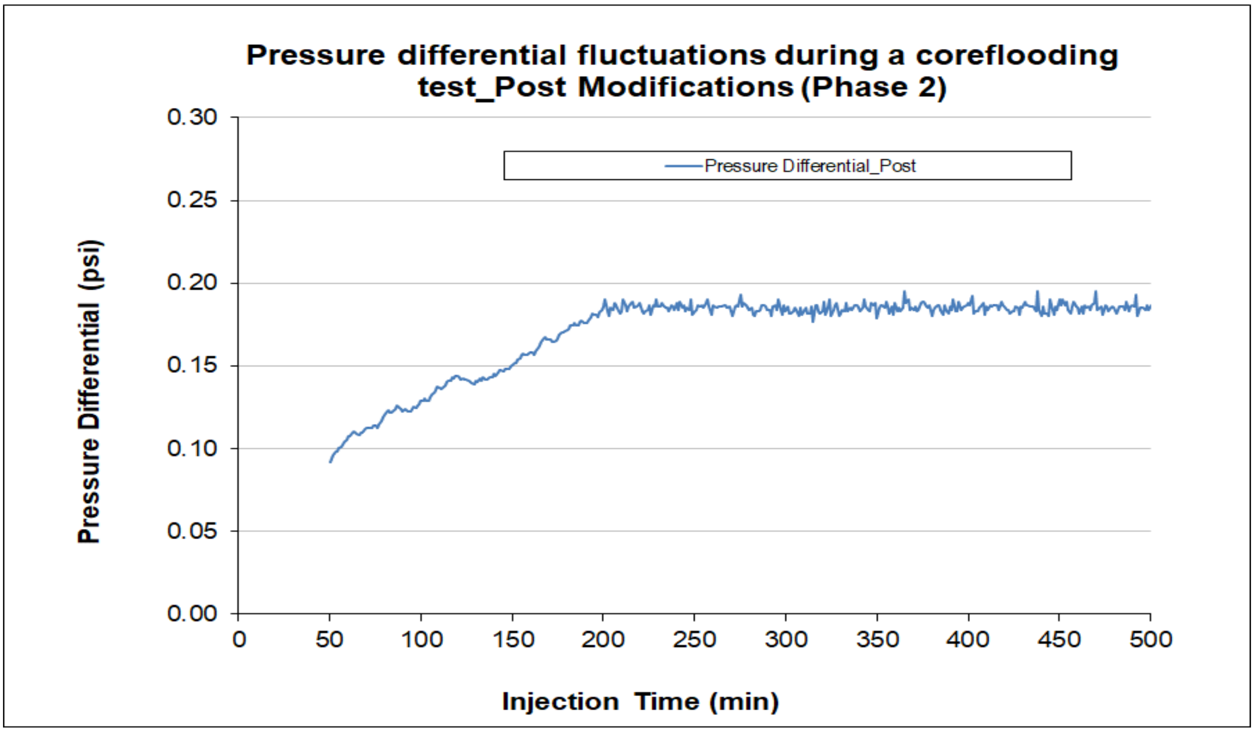

Figure 9: Fluctuations in the pressure differential measurements during a coreflooding test, after making the modifications in the permeameter.
Source: Authors, (2022).

Table 2 shows a statistical summary of the data taken after making the modifications to the equipment. It was identified that the value of the **mean** changed, showing that **systematic errors in the experiment were corrected**. Values such as the standard deviation and typical error decreased, which shows that the modifications made considerably reduced the number of errors when performing a coreflooding test in the permeameters used.

Table 2: Final statistical analysis (Phase 2).

Variable	Worth
Mean	0.1853
Typical error	0.0002
Median	0.1855
Mode	0.1860
Standard deviation	0.0027
Sample variance	0.0000
kurtosis	1.0715
Asymmetry coefficient	0.3001
Range	0.0180
Minimum	0.1770
Maximum	0.1950
Addition	59.6670
Account	322.0000
Confidence level (95.0%)	0.0003
Confidence interval 95%	0.0054
Standard deviation in percentage	1.4564
Data outside IC	19.0000
Data outside IC %	9.0062

Source: Authors, (2022).

Figure 10 shows the histogram of the pressure differential data after making the modifications, which allows us to conclude that the modifications decreased the dispersion of the data, allowing better readings of the differential behavior, and thus giving more reliable results.

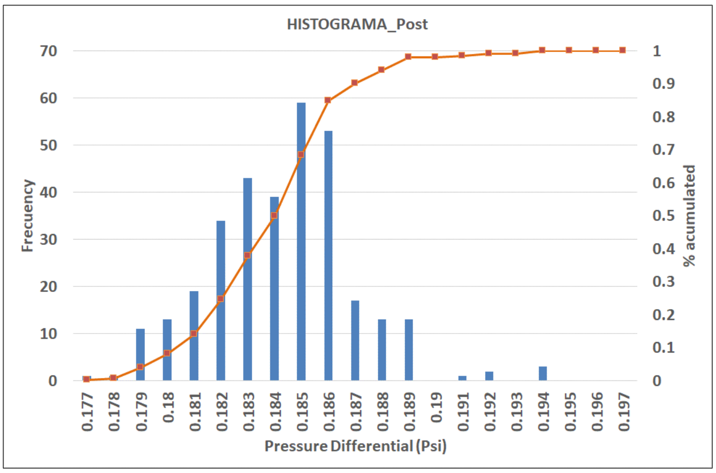

Figure 10: Histogram of the pressure differentials after the modifications made (Phase 2).
Source: Authors, (2022).

In Figure 11, the moving mean for groups of 14 data was calculated and the confidence interval (95%) was estimated. If the standard deviation is expressed as a percentage, it is observed that the uncertainty of the data in the test after making the modifications in the equipment is 1.45%. The evaluated data follow a normal distribution as shown in figure 5, with a mean of 0.1853 psi, standard deviation of 0.0027 psi, taking then that the 95% confidence interval will be ± 0.0054 as seen in (6):

$$\bar{x} \pm (2 * \sigma_x) = 0.1853 \pm (2 * 0.0027) = 0.1853 \pm 0.0054\, psi \quad (6)$$

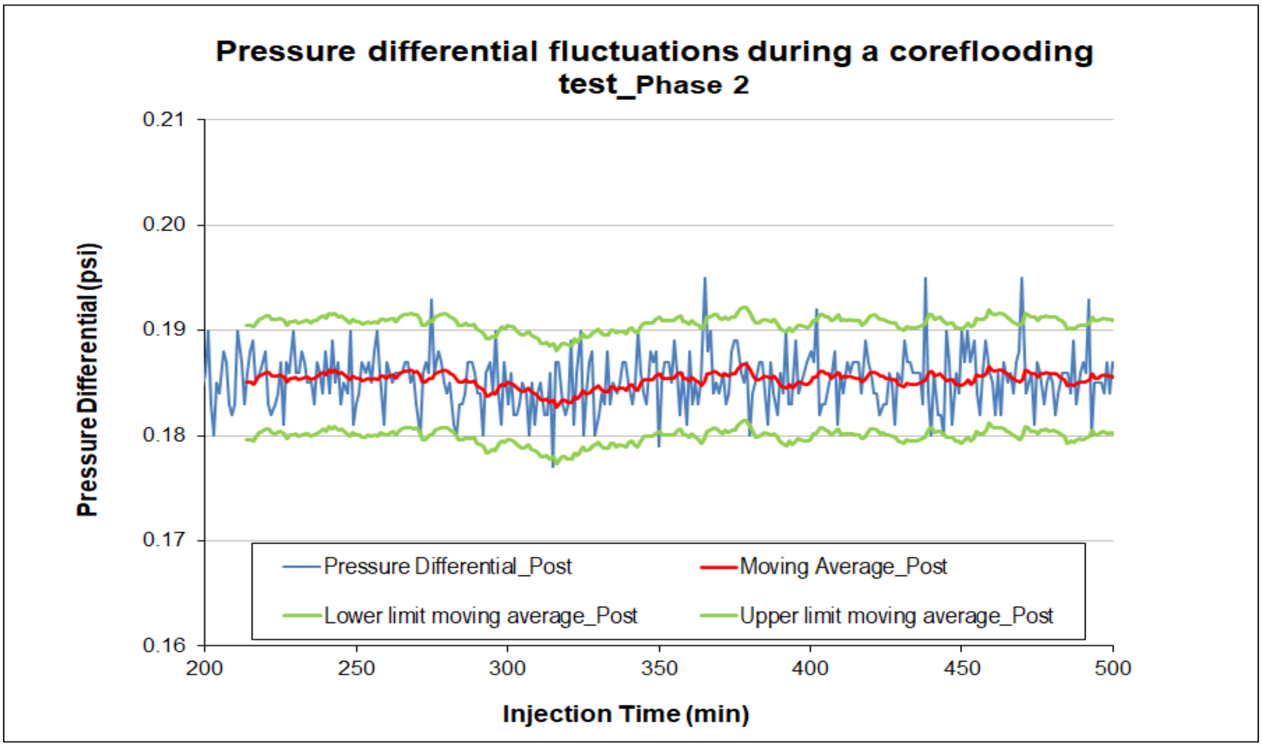

Figure 11: Fluctuations in the pressure differential data after making the modifications in the experimental protocol (Phase 2).
Source: Authors, (2022).

Figure 12 shows a comparison between the results obtained Pre and Post (phase 1 and phase 2) adjustment in the protocol of the permeameter equipment. The initial results presented a standard deviation of 0.019 psi, with a mean of 0.2678 psi and a 95% confidence interval of 0.0379 psi. While the results taken after the modifications in the equipment have a standard deviation of 0.0027 psi, with a mean of 0.1853 psi and a confidence interval of 95% of 0.0054 psi. Expressing the standard deviation as a percentage, it can be seen that the uncertainty of the measurements was reduced from 7.08% to 1.45%.

The relevance of this work is given by the improvement in the quality and reliability of the measurements, in addition to the optimization of the time of the experiments, since by reducing the dispersion of the pressure differentials data, it will be achieved in less time the stable flow state in the porous medium, a critical condition for permeability evaluations.

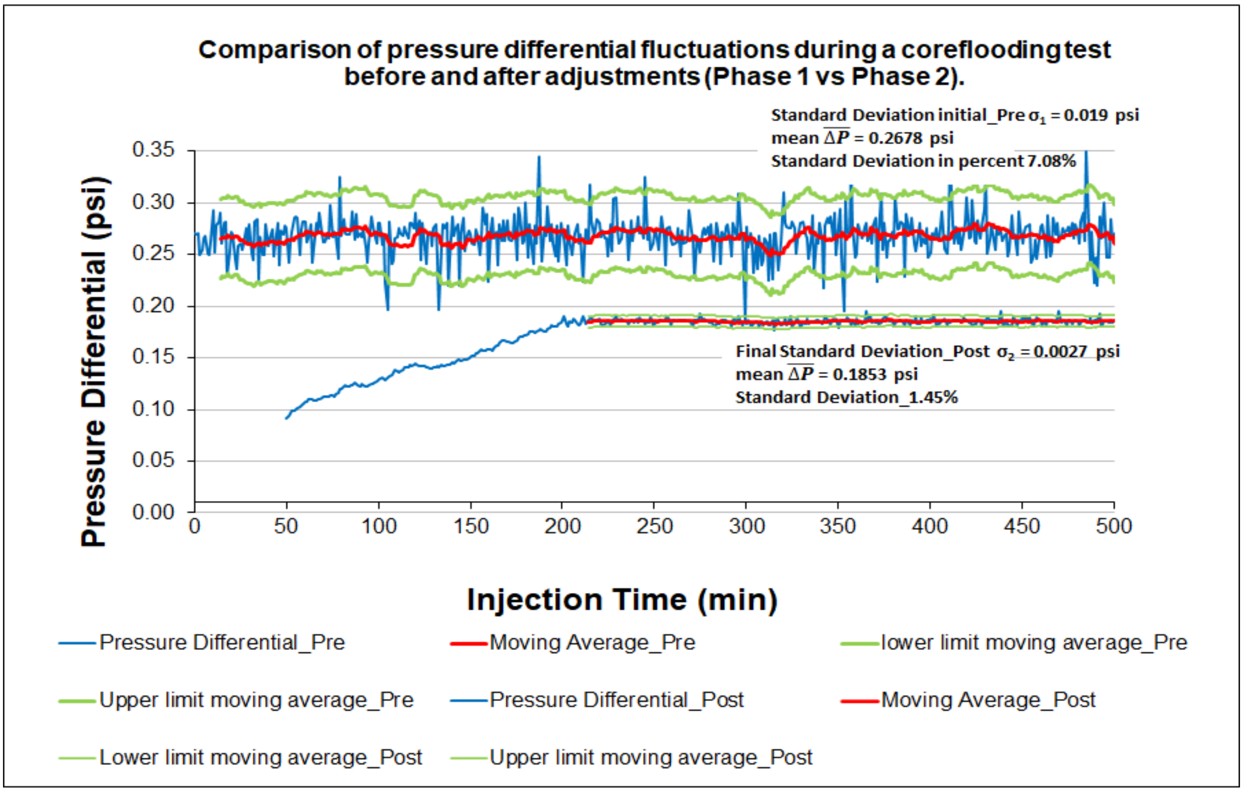

Figure 12: Comparison of pressure differentials data during coreflooding, before and after making the modifications in the permeameter protocols.
Source: Authors, (2022).

V. CONCLUSIONS

In the process of evaluating the quality of the data resulting from the coreflooding, parameters were identified in the pressure differential measurements, which present fluctuations that impact the stabilization time. A deviation of 7.08% was observed with respect to the mean of the measurements, which is considered high and affects the execution time and results of the tests.

Through detailed analysis of the experimental procedure and equipment operation, the sources that were impacting the quality of the measurements were identified and modifications to the equipment were recommended to eliminate them., The precision and accuracy of the pressure differentials displayed on the permeameter were found to be considerably low, and were directly related to the pressure pulses created in the backpressure, the high dead volume in the signal lines, and the influence of the variation of the room temperature.

With the laboratory staff, the proposed modifications were implemented, and new tests were run under the same conditions to observe the changes produced. It was possible to substantially reduce the standard deviation of the measurements to 1.45%, with respect to the mean. Consequently, the stabilization time of the pressure differential was considerably improved and, therefore, the total duration of the tests.

VI. AUTHOR'S CONTRIBUTION

Conceptualization: Jorge Alberto Rojas P., Jenny Paola Rueda M., Jorge Alejandro Sarmiento G., Pedro Juan Rojas M., Jose Luis Mendoza, Cesar Augusto Romero R. and Carlos Humberto Amaya A.

Methodology: Jorge Alberto Rojas P and Carlos Humberto Amaya A.

Investigation: Jorge Alberto Rojas P., Jenny Paola Rueda M., Jorge Alejandro Sarmiento G., Pedro Juan Rojas M., Jose Luis Mendoza, Cesar Augusto Romero R. and Carlos Humberto Amaya A.

Discussion of results: Jorge Alberto Rojas P., Jenny Paola Rueda M. and Jorge Alejandro Sarmiento G.

Writing – Original Draft: Jorge Alberto Rojas P and Carlos Humberto Amaya A.

Writing – Review and Editing: Jorge Alberto Rojas P., Jenny Paola Rueda M. and Jorge Alejandro Sarmiento G and Carlos Humberto Amaya A.

Resources: Jorge Alberto Rojas P., Jenny Paola Rueda M., Jorge Alejandro Sarmiento G., Pedro Juan Rojas M., Jose Luis Mendoza, Cesar Augusto Romero R. and Carlos Humberto Amaya A.

Supervision: Jorge Alberto Rojas P., Jenny Paola Rueda M. and Jorge Alejandro Sarmiento G.

Approval of the final text: Jorge Alberto Rojas P., Jenny Paola Rueda M. and Jorge Alejandro Sarmiento G.

VII. ACKNOWLEDGMENTS

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VIII. REFERENCES

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