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Effect of mineral and organic composition on petrophysics of black shales

Efeito da composição mineral e orgânica na petrofísica de folhelhos negros

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Abstract: The exploration of unconventional resources derived from rocks whose petrophysical characteristics do not allow extraction through traditional methods has been an important aspect of a new global energy matrix. Among these rocks, black shales stand out as reservoirs with high hydrocarbon generation potential, although they exhibit complex properties. This study evaluates the black shales of the Araripe Basin (northeast of Brazil) through basic and advanced petrophysical tests, as well as material characterization techniques, to understand the effect of mineral and organic composition on the petrophysical properties of this type of rock. The results indicate that these shales are predominantly composed of minerals such as calcite and feldspar — a mineralogical composition that favors hydraulic fracturing, an essential technique for enabling hydrocarbon production in unconventional systems — and also contain high levels of organic matter. The samples exhibit low permeability but considerable porosity, although much of it is formed by micropores, mainly present in the organic matter and clay minerals. The analysis of the relationship between porosity and elastic wave propagation velocity revealed the important role of microporosity, which is mainly responsible for the elastic impedance contrast between the unconventional reservoir and host rocks in that basin, favoring the application of the seismic method for mapping this important type of hydrocarbon reservoir.

Keywords: Black shales; Petrophysics; Fracking.

Resumo: Um novo cenário para a expansão da matriz energética global tem se delineado com a exploração de recursos não convencionais, oriundos de rochas cujas características petrofísicas não permitem extração por métodos tradicionais. Entre essas rochas, destacam-se os folhelhos negros que atuam como reservatórios com alto potencial gerador de hidrocarbonetos, embora apresentem propriedades complexas. Este trabalho avalia os folhelhos negros da Bacia do Araripe (nordeste do Brasil) por meio de ensaios petrofísicos básicos e especiais, além de técnicas de caracterização de materiais, para entender o efeito da composição mineral e orgânica sobre as propriedades petrofísicas desse tipo de rocha. Os resultados indicam que esses folhelhos são compostos predominantemente por minerais como calcita e feldspato – composição mineralógica que favorece o fraturamento hidráulico, técnica essencial para viabilizar a produção de hidrocarbonetos em sistemas não convencionais, além de conter teores elevados de matéria orgânica. As amostras apresentam baixa permeabilidade, mas porosidade considerável, embora grande parte dela seja formada por microporos, presentes principalmente na matéria orgânica e nos argilominerais. A análise da relação entre porosidade e velocidade de propagação de ondas elásticas revelou o importante papel da microporosidade, principal responsável pelo contraste de impedância elástica entre o reservatório não convencional e as rochas encaixantes presentes naquela bacia, favorecendo a aplicação do método sísmico para o mapeamento desse importante tipo de reservatório de hidrocarbonetos.

Palavras-chave: Folhelhos negros; Petrofísica; Fraturamento hidráulico.

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1. Introduction

Due to the growing global energy consumption and the continuous dependence on conventional energy sources, such as oil, coal, and natural gas, the international energy sector has directed significant efforts toward the development of alternatives, including solar, wind, hydroelectric, and geothermal energy, which are technically and economically competitive in relation to traditional sources (MIRANDA, 2023). Starting in 2010, the United States of America substantially increased its domestic hydrocarbon production through the adoption of exploitation techniques such as hydraulic fracturing and horizontal drilling in unconventional reservoirs, which made it possible to harness this resource, previously not considered economically viable due to technological limitations. For this reason, production jumped from about 5 million barrels of oil per day in 2010 to 13.2 million barrels of oil per day in 2024. During the same period, natural gas production doubled (EIA, 2025). Conventional oil and natural gas reservoirs are part of petroleum systems composed of mature source rocks, accumulation structures (traps), reservoir rocks with high permeability - generally sandstones or carbonates - and sealing rocks. In contrast, unconventional reservoirs are characterized by their occurrence in low-permeability lithologies, such as shale, in which all the elements of the petroleum system are present within a single geological unit. In this case, the source rock itself also works as a reservoir and seal (ROSS & BUSTIN, 2007).

Among the main types of unconventional reservoirs is black shale (oil shale), which consists of shale rich in organic matter, with potential for generating and accumulating economically significant volumes of hydrocarbons (SLATT, 2012). Shales pose a challenge to geological and petrophysical characterization, mainly due to their heterogeneous composition, fine grain size, and, often, the presence of amorphous mineral and organic phases (RODRIGUES, 2018). Figure 1 shows a block of black shale extracted from the Araripe Basin. These reservoirs are marked by the presence of pores and microfractures ranging from micro- to nanoscale, with approximately 80% of the porosity consisting of micropores. Furthermore, they differ from conventional reservoirs by presenting high concentrations of organic matter, predominance of clay minerals, low macroporosity and permeability - characteristics that imply nanometer-scale pore throats, high mineral surface area, and a complex diagenetic history (ZOU, 2017).

Figure 1 – Outcrop of black shale in the Araripe Basin.
Source: Authors (2025).

Brazil presents considerable potential for the research and exploration of unconventional reservoirs. Among the sites where such material occurs is the Araripe Basin, located across the states of Pernambuco, Ceará, and Piauí. It is the largest inland basin in Northeastern Brazil and contains multiple units of black shales outcropping in the Ipubi Formation, with laminated limestones and gypsum as host rocks (ASSINE, 2007).

Petrophysics, a branch of geosciences that studies the physical properties of rocks and their relationship with fluid movement in pores, considers these properties to be intrinsically associated with mineralogical composition and the presence of fluids. Among these properties are porosity, permeability, and density (RODRIGUES, 2018). Porosity, in particular, is an essential parameter in reservoir engineering, as it is directly related to the fluid storage capacity. In shale lithologies, however, porosity evaluation is complex due to the microscopic nature of their pores (ROSA, 2006). Permeability, defined as the ease with which a fluid moves through a porous medium, is important for the recovery of fluids contained within it (SEABRA, 2005). However, as highlighted by Sakhaee-Pour and Bryant (2012), understanding permeability in shales remains limited, mainly due to the difficulty of modeling fluid flow in nanometer-scale pores. The propagation of elastic waves allows for the elastodynamic characterization of rocks and forms the basis of the seismic method, widely applied in hydrocarbon reservoir prospecting.

For mineralogical characterization, analytical techniques such as X-ray diffraction (XRD) and X-ray fluorescence (XRF) spectrometry are commonly employed. XRD is a technique used for the identification and quantification of crystalline phases, based on the fact that each crystalline compound has a unique diffractometric pattern (MELLO, 2006). XRF, in turn, allows for multielemental determination of the chemical composition of geological materials, such as rocks, soils, and sediments, and is widely applied for this purpose (DUTRA & GOMES, 1984).

The presence of organic matter makes the physical properties of unconventional reservoirs more complex, being an important factor in the development of microporosity within the rock (SHI *et al.*, 2015). Nanometer-scale pore systems in shales play a relevant role in controlling hydrocarbon storage capacity (CHALMERS *et al.*, 2012). The identification of shales with hydrocarbon production potential involves characterization studies. Among the analyses used for this purpose are those focused on organic matter, mineralogy, gas content in rocks, gamma-ray spectrometry, porosity, and permeability (ALEXANDER *et al.*, 2011).

Thus, the present study aims to characterize the black shales of the Araripe Basin, with emphasis on their petrophysical, mineralogical, and organic properties, through laboratory analyses, and to understand the effect of mineral and organic composition of black shales on their more relevant petrophysical properties.

2. Methodology

The study area is located on the border between the municipalities of Nova Olinda and Santana do Cariri, in the state of Ceará, where rock samples were collected during field campaigns carried out at the Pedra Branca Mine. In this location, representative outcrops of black shales from Araripe Basin occur (Figure 2).

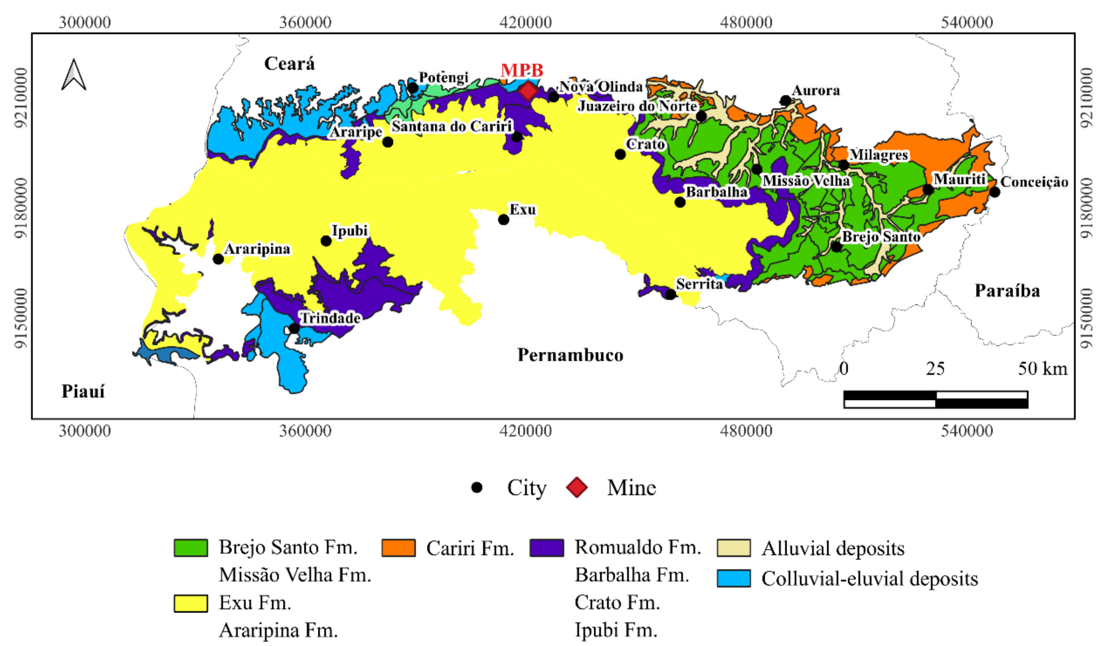

Figure 2 – Location map of the study area.
Source: Adapted and modified from Assine (2007) and Medeiros (2004).

The basic petrophysical tests carried out in this research comprised the determination of porosity, permeability, bulk density, and grain density. The measurement of elastic wave propagation velocities is considered a special petrophysical method. For these procedures, the samples were cut and their surfaces polished. Due to the essentially fissile nature of the shales, these samples were tested at their natural saturation, without the traditional drying in an oven at 90 °C for 24 hours, which would have resulted in breakage and consequent loss of the samples. The basic petrophysical tests were performed using the Ultra-Poro/Perm 500 porosimeter-permeameter, with nitrogen as the saturating gas, while the special petrophysical tests were carried out using the Autolab 500 system.

X-ray fluorescence was employed as a complementary technique to X-ray diffraction, providing information about the elemental composition of the materials. The analyses were conducted by energy-dispersive spectrometry, enabling the quantification of chemical elements present in the samples. XRD analyses were fundamental for the identification of crystalline mineral phases, based on the interaction of X-rays with the ordered atomic structure of minerals, producing specific diffraction patterns. The experiments were performed on a diffractometer with Cu-Kα radiation, operating at 40 kV and 30 mA, with an angular scan range of 5° to 60° (2θ) and a step size of 2°/min. Quantitative analysis of the mineral phases was carried out using the Rietveld refinement method, allowing precise determination of the proportions of the identified phases.

Ultrasonic velocity tests were performed with simultaneous acquisition of three waveforms: the compressional wave (P-wave), which propagates with polarization parallel to the longitudinal axis of the sample, and two shear waves (S_1 and S_2), with mutually orthogonal polarization directions and perpendicular to the sample axis.

Thermogravimetric analysis (TGA) was used to determine the organic matter present in the samples. The following parameters were employed: initial temperature of 30 °C, final temperature of 900 °C, heating rate of 15 °C/min, and nitrogen as the carrier gas.

3. Results and discussion

Shales are fine-grained sedimentary rocks, classified as composite materials at the microscopic scale, formed by particles with diameters ranging from a few nanometers to 100 micrometers. These rocks exhibit significant mineralogical variability and may contain clay minerals, phyllosilicates, quartz, feldspar, carbonates, organic matter, and pyrite (CHARLTON et al., 2021). Due to their compact structure and fine grain size, shales display low macroporosity and permeability, as their porosity is mostly composed of micropores hosted in clay and organic matter. This makes them excellent seals in petroleum systems and an important factor in studies of geological CO_2 storage or contaminant containment (BJØRLYKKE, 2010). Table 1 shows the results obtained from the petrophysical tests performed on samples collected at the Pedra Branca Mine.

Table 1 – Results of the petrophysical tests performed on samples from the MPB mine.

Sample	Porosity (%)	Permeability (mD)	Grain density (g/cm³)	Bulk density (g/cm³)
MPB_1A	27.4	<1	2.223	1.614
MPB_1B	26.8	1.50	2.313	1.693
MPB_1C	28.0	<1	2.233	1.608
MPB_1D	27.6	<1	2.259	1.635
MPB_1E	25.6	3.97	2.314	1.723
MPB_1F	27.6	2.07	2.252	1.631
MPB_1G	23.4	3.31	2.372	1.816
MPB_1H	26.5	-	2.303	1.693
MPB_1I	25.8	1.89	2.398	1.780
MPB_1J	28.2	-	2.274	1.632
MPB-P002	21.9	-	2.491	1.944
MPB-P003	20.6	2.06	2.437	1.935
MPB-P005	21.2	<1	2.470	1.945
MPB-P006	31.3	-	2.324	1.596
MPB-P007A	24.6	<1	2.360	1.777

MPB-P008	18.9	<1	2.460	1.996
MPB-P009	22.4	-	2.002	1.553
MPB_P07B	14.5	<1	2.411	2.061
MPB01-01	4.6	-	1.942	1.853
MPB01-02	4.0	-	1.986	1.907
MPB01-03	9.9	-	2.102	1.894
MPB01-04	2.3	-	1.926	1.882
MPB01-05	5.1	-	2.064	1.959
MPB01A-01	10.9	-	2.121	1.890
MPB01A-02	10.4	-	2.113	1.893
MPB01A-03	7.4	-	2.002	1.854
MPB01A-04	6.9	-	2.010	1.871
MPB02-01	7.9	-	2.041	1.880
MPB02-03	14.8	-	2.142	1.825
MPB02-05	4.2	-	1.912	1.832
MPB02-06	10.2	-	2.080	1.868
MPB03-01	8.0	-	2.056	1.892
MPB03-03	13.7	-	2.210	1.907
MPB02-04	9.4	-	2.084	1.888

Source: Authors (2025).

The results of the petrophysical tests indicated low permeability values. In some cases, permeability could not be measured because the values were below the sensitivity limit of the equipment (1 mD). According to Amyx *et al.* (1960), permeabilities below 1 mD are considered low. Since all obtained results fall within the same order of magnitude, it is concluded that permeability is low in all samples, which is consistent with the type of rock analyzed. For comparison, conventional compacted sandstone reservoirs exhibit permeabilities ranging from 0.5 mD to 20 mD (KING, 2012). The low permeability of shales results from the limited interconnection between their macropores, as observed by Law & Curtis (2002). However, even when well interconnected, micropores do not substantially contribute to shale permeability due to their high capillary pressure.

Porosity values measured in laboratory tests ranged from 2.3% to 31.3%. According to Chapman (1976), conventional reservoir rocks, typically detrital in composition with grain sizes between sand and gravel, have porosity sufficient to store and subsequently allow the extraction of petroleum. The most common porosity values in such rocks range between 5% and 35%, with higher concentrations between 15% and 30%. Although the analyzed samples are shales, the results obtained from basic petrophysical tests revealed relatively high porosity values. It is noteworthy that these values do not include porosity associated with micropores, as the equipment used does not have sufficient sensitivity to characterize them. Therefore, the total porosity of the samples may be even higher than that recorded in the tests.

According to the standards established by the International Union of Pure and Applied Chemistry (IUPAC), pores can be classified into three categories: macropores (>50 nm), mesopores (2–50 nm), and micropores (<2 nm). In shale reservoirs, pores are significantly smaller than those found in conventional reservoirs, often reaching the nanometer scale (NELSON, 2009; ROSS & BUSTIN, 2009). Laboratory porosity tests, performed with the equipment used in this study, predominantly characterize macropores and mesopores. Thus, microporosity—usually associated with organic matter—may not be fully captured by this methodology (SHI et al., 2015).

Although mesopores and macropores are also present, micropores dominate in black shale systems (BU *et al.,* 2015; YANG et al., 2016). For this reason, the laboratory-measured porosity does not represent the total porosity of this type of rock, as a large portion is associated with the presence of organic matter and clay minerals in the sample. In the shales analyzed in this study, there are indications that organic matter predominantly contributes to the development of micropores, showing a direct relationship between organic matter content and microporosity.

XRD analyses indicate that the predominant mineral phases in the investigated samples are calcite and orthoclase (feldspar). X-ray fluorescence tests revealed average proportions of 60% CaO and 17% SiO_2. Other mineral phases were identified in lower concentrations. Figure 3 presents some of the generated diffractograms. The mineralogical composition of shales plays a key role in the feasibility of gas and oil production in reservoirs, as it directly influences the mechanical behavior of the rock during hydraulic fracturing, an essential operation in the exploitation of unconventional deposits (SLATT, 2012). Shales with higher quartz and calcite content, for example, tend to respond better to fracturing due to the brittleness of these minerals, which favors fracture generation (GALE *et* al., 2007; JARVIE *et al.,* 2007). On the other hand, a significant presence of clay minerals can reduce both porosity and permeability, hindering production. In the samples analyzed in this study, the most abundant mineral fractions were calcite, muscovite, and orthoclase. Considering the composition of the analyzed samples, it is possible to infer that the rocks from the study area are likely to exhibit a good response to hydraulic fracturing.

The Rietveld method allows for the quantitative analysis of mineral phases identified through XRD tests. Six samples were subjected to this technique, and the results are presented in Table 2.

Table 3 shows the organic matter content of the samples subjected to thermogravimetric analysis (TGA). The organic matter content of the evaluated samples ranged from 9.8% to 30.4%, which is consistent with the results reported by Castro (2015), where total organic carbon (TOC) values obtained for shales of the Ipubi Formation indicated exceptionally high organic matter abundance, well above the minimum threshold of 1% required for a rock to be considered potentially hydrocarbon-generating. With an average of approximately 22.5%, the shales of the Ipubi Formation demonstrate exceptional hydrocarbon generation potential. Despite having relatively low clay mineral content, their high organic matter content contributes to elevated microporosity.

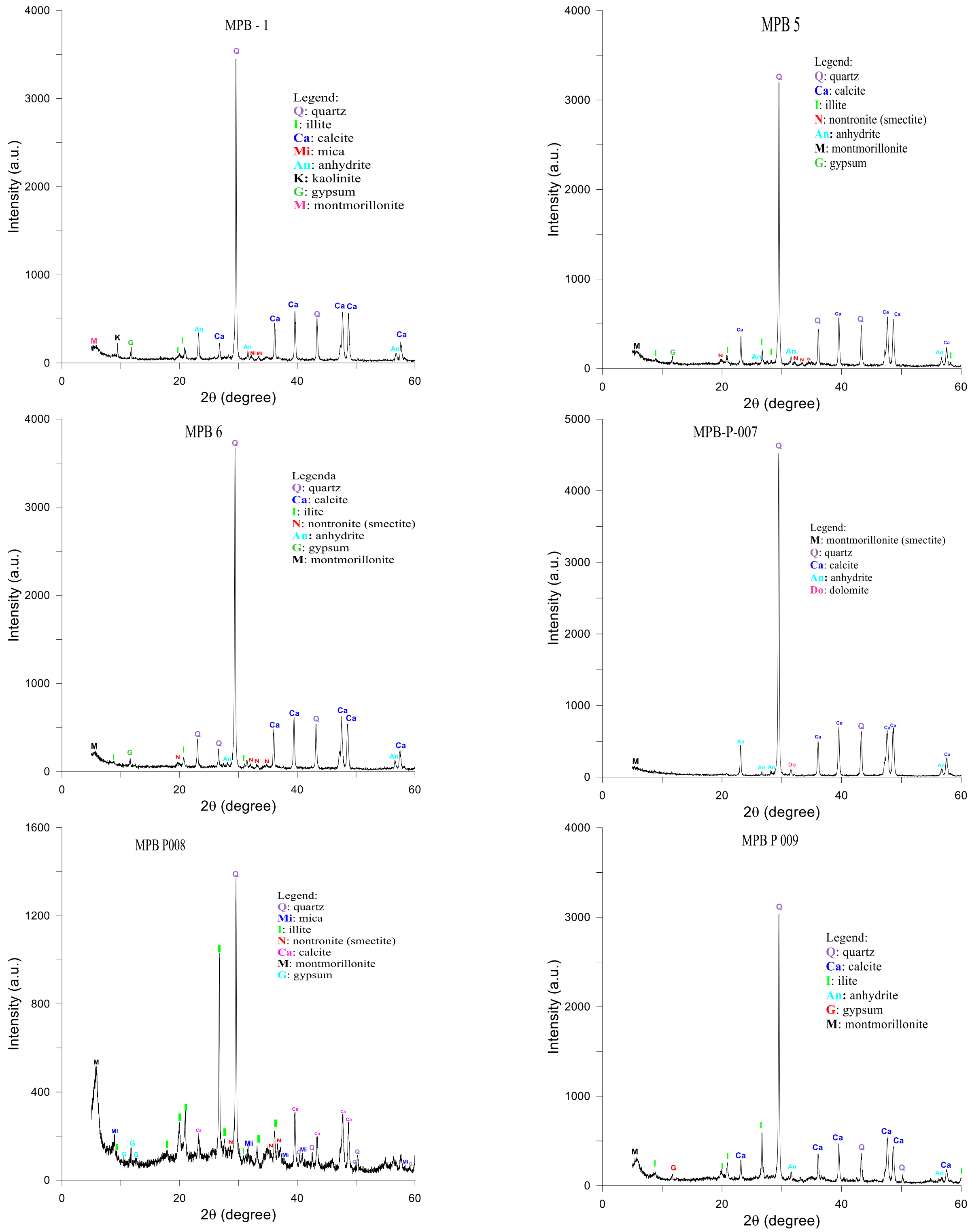

Figure 3 – Diffractograms recorded in samples of bituminous shale from the Pedra Branca Mine. Source: Authors (2025).

Table 2 – Results of the Rietveld refinement performed on samples collected from the MPB Mine.

Phase	**Density (g/cm³)**	**MPB 01-01**	**MPB 02-06H**	**MPB 01-05**	**MPB 01-04**	**MPB 02-03H**	**MPB 03-03**
		% wgt					
Quartz	2.65	7.00	4.26	2.10	5.13	5.7	4.38
Calcite	2.72	23.23	30.62	47.16	32.81	28.78	32.77
Aragonite	2.95	-	-	-	3.62	-	-
Clays	2.55	3.96	2.07	1.33	1.61	2.18	1.71
Orthoclase	2.57	6.10	8.28	7.00	5.37	10.79	18.59
Albite	2.62	7.33	1.01	-	-	-	0.88
Muscovite	2.93	20.24	15.01	13.47	20.07	14.34	16.24
Pyrite	5.02	2.69	4.73	1.18	-	6.18	0.03
Gypsum	2.32	1.81	6.45	-	1.01	7.12	-
Zircon	4.68	1.50	-	-	-	-	-

Source: Authors (2025).

Table 3 – Organic matter content present in the samples.

Sample	**Organic matter (% wgt)**
MPB 01-01	26.1
MPB 01-05	27.8
MPB 02-03H	26.2
MPB 02-06H	27.6
MPB 01-04	30.4
MPB 03-03	25.4

Source: Authors (2025).

Figure 4 shows the relationship between laboratory-measured porosity and the organic matter content in the samples. In general, it can be observed that the higher the organic matter content - typically associated with microporosity - the lower the porosity measured in the laboratory, which predominantly characterizes macropores and mesopores. However, when analyzing samples MPB 02-03 and MPB 03-03, as well as MPB 01-01 and MPB 01-05, it is noted that, despite having similar organic matter contents and, consequently, microporosity, they exhibit different laboratory porosities, reflecting varying proportions of macropores. Therefore, although total porosity results from the sum of micro-, meso-, and macropores, the same percentage of organic matter does not necessarily imply equivalent laboratory porosity responses. This behavior may be associated with differences in the distribution and type of organic matter, as well as its interaction with the mineral matrix, factors that control the proportion of macropores and mesopores detectable in the tests.

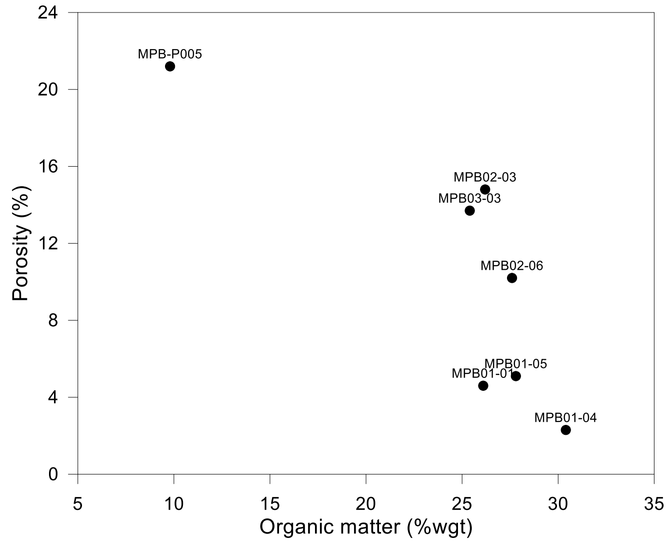

Figure 4 – Relationship between porosity and organic matter.
Source: Authors (2025).

Shales account for almost the entirety of organic matter–enriched rocks. This is due to the fact that, because of their low density, organic compounds have a higher affinity for fine-grained sediments and are commonly deposited in the same environment as these sediments (GAMA & PEREIRA, 2009). This affinity explains why shales generally exhibit the highest concentrations of organic matter, which, in many cases, is matured in the form of hydrocarbons. In such cases, these rocks are referred to as black shales (KLEMME & ULMISHEK, 1991).

When relating grain density to organic matter content in the samples, it is observed that the higher the organic matter content, the lower the grain density. This behavior occurs because grain density is influenced by both the inorganic and organic fractions of the rock (Figure 5).

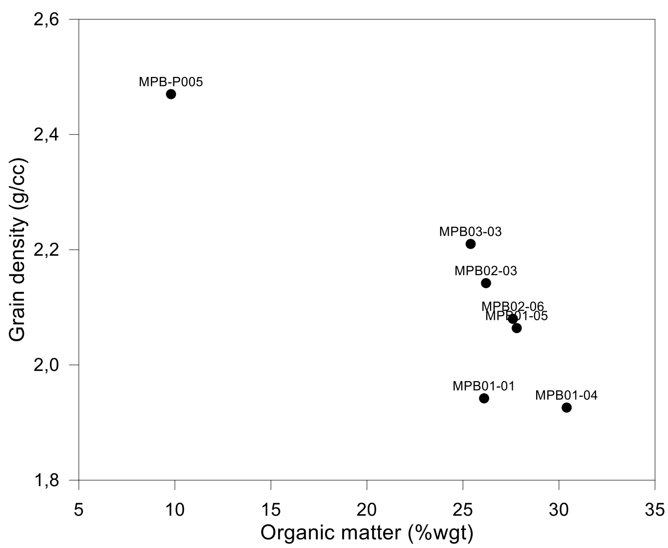

Figure 5 – Relationship between grain density and organic matter.
Source: Authors (2025).

Ultrasonic velocities are widely used in the mechanical characterization of rocks because they constitute an economically viable, rapid, environmentally safe, and non-destructive technique. From these velocities, it is possible to calculate the dynamic elastic moduli of the rocks (ALTAWATI, EMADI & KHALIL, 2021). Table 4 presents the results of the elastodynamic tests performed on black shale samples from the Araripe Basin.

Table 4 – Results of the elastodynamic tests performed on shale samples from the Pedra Branca Mine.

Sample	VP (m/s)	VS_1 (m/s)	VS_2 (m/s)
MBP 01A-02	1927	1123	1131
MPB 01-01	2134	1133	1271
MPB 01-02	2136	1075	1179
MPB 01-03	2079	1432	1431
MPB 01-04	1352	862	886
MPB 01-05	1529	1014	1007
MPB 01A-01	1978	1168	1178
MPB 01A-03	1991	1122	1136
MPB 01A-04	2017	1088	1084
MPB 02-01	2063	1327	1095
MPB 02-03	2817	1489	1681
MPB 02-04	2760	1365	1516
MPB 02-05	1409	750	751
MPB 02-06	2807	1488	1653
MPB 03-01	1925	968	916
MPB 03-03	2021	1108	1131
MPB_02	2974	1783	1735
MPB_1D	2594	1488	1484
MPB_P05	3223	1758	1757
MPB_P07B	3847	2309	2317
MPB_04	2973	1709	1705
MPB_1A	2627	1492	1517
MPB_1F	2805	1607	1459
MPB_1C	2447	1411	1433
MPB_1B	3152	1806	1764
MPB_P03	2972	1736	1744
MPB_1H	3039	1776	1499
MPB_1E	3044	1756	1481
MPB_1G	3024	1531	1518
MPB_1J	2790	1371	1695
MPB_P07A	2627	1631	1644
MPB_1I	3392	1702	1698

Source: Authors (2025).

The propagation velocities of elastic waves and the bulk density of black shales are consistently lower than those of the host rocks (laminated limestones and gypsum) in the Araripe Basin, as discussed by Gurjão *et al.* (2013), thus providing an important elastic impedance contrast, which is a key factor for the success of a subsurface imaging campaign using seismic methods.

Porosity is a fundamental parameter that influences P- and S-wave velocities, being related to the predominant type of pores—macropores or micropores (REIJMER *et al.,* 2021). Figure 6 indicates that the higher the presence of micropores (generally associated with higher organic matter content in this case), the lower the wave propagation velocity. However, samples with approximately the same organic matter content, such as MPB 01-05 and MPB 02-06, may exhibit significant variations in P-wave velocity values. This variation is explained by the influence of other factors, such as rock anisotropy. Samples MPB 02-06 and MPB 02-03 were extracted in the horizontal direction (sample axis parallel to bedding/lamination), while the remaining samples were collected in the vertical direction (sample axis perpendicular to bedding). This behavior is consistent with the expected trend for anisotropic rocks, in which the P-wave propagation velocity parallel to bedding is higher than in the direction perpendicular to bedding (SOARES, 2006).

The predominance of macropores, identified through petrophysical tests, is associated with increased wave velocities. This comparison suggests that macroporosity and microporosity are complementary parameters in these samples, such that higher microporosity is associated with lower macroporosity, and vice versa. Macropores offer less resistance to wave propagation than micropores due to the wavefront healing effect (NOLET & DAHLEN, 2000; LIU & DONG, 2012). For this reason, P-wave velocity in these samples increases with increasing macroporosity, as shown in the graph in Figure 7. Conversely, micropores - which tend to occur in greater proportion in these samples - promote greater wave dispersion and attenuation, thereby reducing the propagation velocity of elastic waves (EBERLI *et al.,* 2003). Similar results were observed by Wang, Sun & Ge (2024), who reported a decrease in velocities with increasing kerogen content.

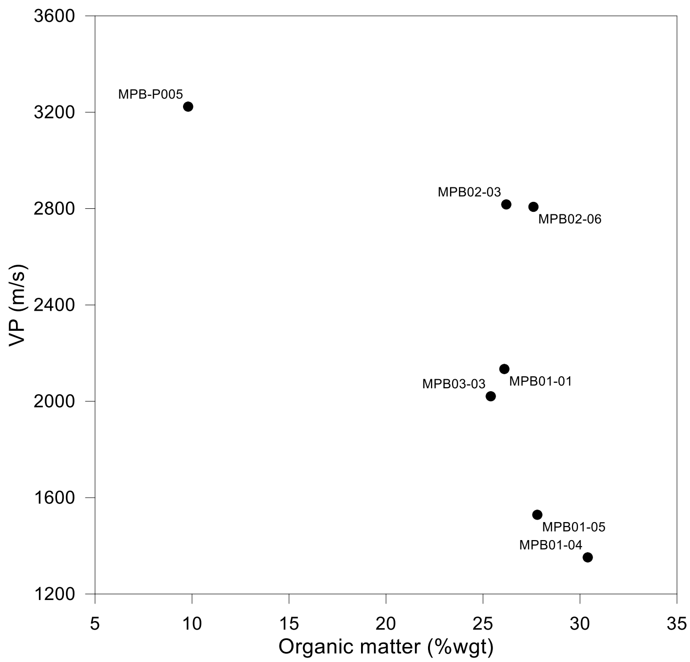

Figure 6 – Graph showing the relationship between VP and organic matter.
Source: Authors (2025).

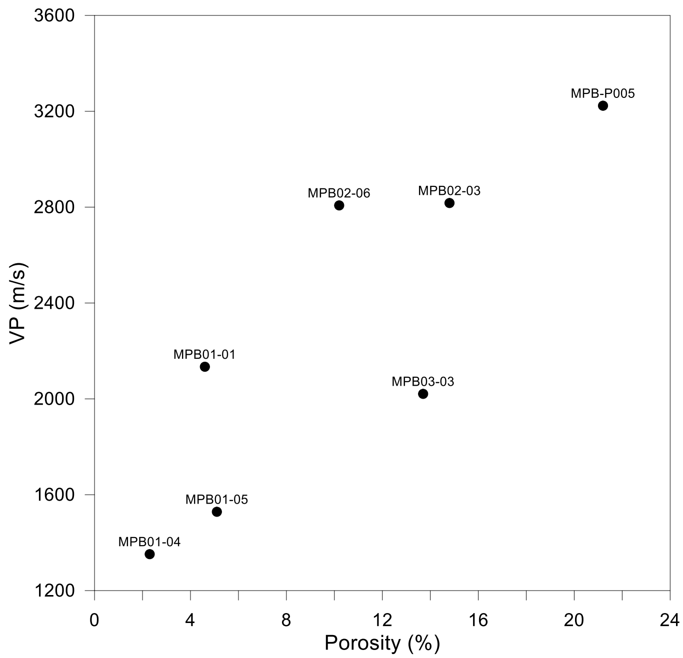

Figure 7 – Relationship between VP and porosity.
Source: Authors (2025).

4. Final considerations

The compositional and petrophysical analyses conducted on black shale samples from the Ipubi Formation of the Araripe Basin indicate that these rocks have calcite, muscovite, and orthoclase as their main inorganic mineral phases, with relatively low clay mineral content. On the other hand, organic matter contents are remarkably high. This significant organic phase has led to hydrocarbon generation and accumulation in predominantly micro- and nanometer-scale pores. The high organic matter content is responsible for the low grain density values, and its associated microporosity results in low elastic wave propagation velocities. Although the black shales of the Araripe Basin exhibit very low permeabilities, their mineral composition - dominated by brittle minerals such as calcite and orthoclase - favors the formation of hydraulic fractures, making these rocks sufficiently permeable for hydrocarbon production. The much lower elastic velocities and densities of the black shales compared to the prevailing host rocks in the basin (laminated limestones and gypsum) result in a strong impedance contrast, which is an essential factor for seismic exploration of any type of oil and gas reservoir.

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