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Abstract: The work presents the laboratory studies on permeability of two bituminous coal briquettes under confining pressure conditions. The research was carried out in order to assess the possibility of using bituminous coal as a sorbent for CO_2 storage in underground seams. Coal permeability tests were carried out on an original apparatus for testing seepage processes under isobaric conditions on samples subjected to confining pressure. In order to determine the impact of the load on the coal briquettes' permeability, the tests were carried out at four confining pressures: 1.5, 10, 20 and 30 MPa. The obtained results showed that the coal permeability decreases with an increase in confining pressure. At depths below 250 m, the coal can be a rock poorly permeable to CO_2 , and under such conditions, the applicability of technologies related to the underground storage of CO_2 to coal seams is limited or even impossible.

Keywords: bituminous coal; permeability; CO2 sequestration; coal briquette



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1. Introduction

There are many materials of natural origin which have applications as sorbents [1]. Bituminous coal is a sedimentary rock of plant origin, but it has very good reservoir rock properties and is often used as a sorbent [2,3]. In particular, it finds its application for the capture and storage of greenhouse gases, due to its good sorption properties [4,5].

The possible impact of CO_2 emissions on climate change was discussed as early as the late 19th century [6]. However, in the following years, despite the deteriorating forecasts of the effects of greenhouse gas emissions into the atmosphere, no specific solutions were proposed and implemented by global authorities. It was not until the end of the 20th century that the problems related to Earth's climate change began to be discussed more and more often in the media, conferences and international congresses. As a result, the search for new solutions to prevent climate change due to greenhouse gas emissions into the atmosphere has begun. Hence, the idea of CCS (Carbon Capture and Storage) technology was developed [7].

The technology of CO₂ sequestration in deep coal seams has been studied for many years [8,9] and continued to be developed. This technology involves the injection of carbon dioxide into underground coal seams, resulting in the release of methane and is based on the principle of CO₂/CH₄ exchange sorption [10] and coal preference for sorption of CO₂ over CH₄ [11–13]. Due to its sorption and structural properties, coal is a rock which is probably the most promising reservoir rock for CO₂ storage [14]. Laboratory studies of CO₂/CH₄ exchange sorption in the context of assessing the possibility of underground storage of CO₂ in coal seams are carried out on a large scale [15–25]. There have also been many ECBM (Enhanced Coalbed Methane recovery) pilot/demonstration test projects in the world (e.g., in USA, Canada, China, Japan and Poland), by injecting CO₂ into coal seams [26].

The first European field study of CO₂-ECBM technology, started in 2001 and managed by the Central Mining Institute in Katowice (GIG), was carried out as part of the European

RECOPOL project (Reduction of CO_2 Emissions by Means of CO_2 Storage in the Silesian coal basin of Poland). The main goal of the project was to verify the applicability of the ECBM technology in the natural conditions of Europe, by injecting CO_2 into coal and storing it in a long-term and safe manner. According to the project plan, 760 tons of CO_2 were injected into the coal seams and the CH_4 production increased from around 100 m³/day to over 700 m³/day. Field tests carried out within the RECOPOL project showed, among other things, that a major limitation in the implementation of CO_2 -ECBM technology is the low permeability of coal at a depth of 1000 m. This was caused, among other things, by the confining pressure and sorption swelling of coal. To prevent these problems, coal fracturing was performed, followed by improved permeability and gas flow [27]. Despite these technical problems, the results of the RECOPOL project were very promising.

Poland is estimated to have 20–415 billion m³ of CBM resources with the potential to store 470 tons of CO₂ in the Upper Silesian basin according to Sloss [28], and according to Kędzior [29], the CBM estimate for the Upper Silesian Coal Basin is 350 billion m³ and proven reserves of over 98.6 billion m³.

More recent studies, including in situ and laboratory studies, prove that the release of methane from Polish coal seams is a slow process and it is necessary to accelerate it in order to achieve greater efficiency [18]. The analysis of several pilot, field projects showed low coal permeability and its sorptive swelling, which limited the possibility of CO₂ injection into the coal seam [30].

Previous studies performed at the Strata Mechanics Research Institute of the Polish Academy of Sciences on the effect of stress on the coal permeability to N_2 and CH_4 have shown that confining pressure reduces the porosity of coal, which in turn reduces its permeability [31,32]. Coal permeability decreases exponentially with increasing effective stress, due to reducing and closing of flow channels in pores and fractures [33–43]. Increase of the depth of the coal seams and resulting increase of confining pressure induces pronounced decrease of its permeability in comparison with unburdened coal, even to several orders of magnitude, at depths below 1000 m [44–46]. Furthermore, rock permeabilities to sorbed gases are lower than rock permeabilities to non-sorbed or poorly sorbed gases [47].

Problems with the applicability of the CO_2 -ECBM technology in underground coal seams are mainly due to the limited coal permeability under in situ conditions. The purpose of this study was to investigate in detail the impact of the confining pressure, corresponding to in situ conditions, on the permeability of coal to gases (He and CO_2). This research was aimed at identifying possible causes of failures of field studies of CO_2 -ECBM processes in underground coal seams.

2. Measuring Apparatus

Coal permeability tests were carried out on an original, innovative apparatus for testing seepage processes under isobaric conditions on samples loaded by the confining pressure [48]. This apparatus (Figure 1) provides measurements under isobaric gas and load conditions. The test sample is placed in a high-pressure chamber filled with water. The water applies a confining pressure to the sample corresponding to the in situ conditions. Constant confining pressure is provided by a precise mechanical actuator. Gas is injected into the sample inlet at a constant pressure, which is ensured by a pressure regulator. At the outlet of the sample, the pressure is also kept constant and the gas flow rate is measured. All necessary measurement parameters are registered by the control system.

The measuring apparatus used in the permeability tests ensures, among others [48]:

- The confining pressure regulation in the range of 0.1–40 MPa, and the stabilization accuracy equal to ± 0.02 MPa;
- The gas pressure regulation at the sample inlet and outlet in the range of 0.1–1.6 MPa and 0.1–1.0 MPa, respectively, and the stabilization accuracy of 0.12% of the full scale;
- The flow rate measurement at the outlet of the sample in the range of 0–5 cm³/min, and the accuracy of 1.0% of the full scale.



Figure 1. Scheme of the measuring apparatus for testing seepage under confining pressure conditions.

3. Research Material

Two bituminous coal samples with different degrees of coalification were used in the study. The coals originated from the Upper Silesian Coal Basin, Poland. Table 1 presents select technical and petrographic parameters of the samples.

			- daf	, d			
Origin	Coal Rank	R _o [%]	[%]	A ^u [%]	W _t [%]	ρ_{sk} [g/cm ³]	φ [%]
Sobieski mine	medium-rank D Para-bituminous coal	0.565	39.63	8.41	5.35	1.410	32.3
Silesia mine	medium-rank C Ortho-bituminous coal	0.678	39.32	12.00	2.65	1.411	24.3

Table 1. Technical and petrographic parameters of the studied coal samples.

 R_o is the vitrinite reflectance; V^{daf} is the volatile matter; A^d is the ash content; W_t is the moisture content; ρ_{sk} is the skeletal density; ϕ is the porosity.

Based on the classification of coal according to the average vitrinite reflectance, in line with the UN-ECE International Classification of In-Seam Coals, the Sobieski sample, with the reflectance of 0.565%, represents a medium-rank D Para-bituminous coal, while the sample Silesia, with the reflectance of 0.678%, represents a medium-rank C Orthobituminous coal [49].

The samples used for the permeability tests were briquettes prepared from bituminous coal grains. Briquettes were made by pressing grains on a hydraulic press with a pressure of about 40 MPa. The value of the sample forming pressure was set to obtain a briquette porosity similar to the original coal. Such sample preparation made it possible to obtain test material that can be considered representative of the original coal material [50]. In order to protect the sample from water applying a confining pressure on the sample, it was prepared in a thin Teflon sealing coat. In addition, during the pressing of the sample, two needles were embedded in it, which made it possible to measure the pressure inside the sample. A picture of the coal briquette sample prepared for testing is shown in Figure 2.

Before the permeability measurement, the bituminous coal material samples were subjected to scanning electron microscopy (SEM) and low-pressure nitrogen adsorption (LPNA) analyses at the temperature of 77 K. Example surface images of both coal briquette samples, at $500 \times$ magnification, obtained by SEM are shown in Figure 3.



Figure 2. Coal briquette sample prepared for measurements.



Figure 3. SEM images (magn. 500×) of the microstructure of the surface of coal briquette.

The results of low-pressure nitrogen adsorption analyses (LPNA method) at temperature of 77 K, as well as pore size distribution of both coal samples, are presented in Figure 4. These tests were performed on the ASAP2020 analyser (Micromeritics). The obtained sorption capacity of "Sobieski" coal was about twice as high as that of the "Silesia" sample. The maximum quantity of adsorbed of the "Sobieski" coal was 12.37 cm³/g, and of the "Silesia" coal 7.08 cm³/g, at the relative pressure $p/p_0 = 1$.



Figure 4. Results of low-pressure nitrogen adsorption (LPNA method) analysis of coals: (**a**) adsorption and desorption isotherms; (**b**) pore size distribution as a function of average pore diameter.

The results of adsorption analyses performed by the LPNA method are presented in Table 2. The specific surface areas determined by BET and Langmuir models were about three times larger for the "Sobieski" sample in comparison with the "Silesia" sample.

Table 2. Adsorption properties of samples.

Coal Sample Maximum Quantity [cm ³ /g] BET Surface Area [cm ³ /g]		[cm ³ /g]
12.37 10.9 7 08 3 7	92 16.49 2 5.71	0.019
	12.37 10.9 7.08 3.7	12.37 10.92 16.49 7.08 3.72 5.71

4. Methodology

Two gases, helium (He) and carbon dioxide (CO₂), were used to study the permeability of coal samples under confining pressure conditions. In order to determine the influence of the load on the coal permeability, the tests were carried out at 4 confining pressures: 1.5, 10, 20 and 30 MPa. The gas inlet and outlet pressures were regulated in the range of 0.1–0.8 MPa.

The permeability of coal was determined from Darcy's law [51]:

$$k_g = \frac{2 \cdot Q \cdot p_{atm} \cdot \mu \cdot l}{A \cdot (p_{in}^2 - p_{out}^2)} , \qquad (1)$$

where: $k_g [m^2]$ —is the Darcy permeability coefficient $Q [\frac{m^3}{s}]$ —is the gas flow rate at the outlet of the sample; p_{atm} [Pa]—is the atmospheric pressure; μ [Pa·s]—is the dynamic viscosity coefficient of gas; $A [m^2]$, l [m]—are the surface and length of the sample; p_{in} , p_{out} [Pa]—are the inlet and outlet pressure of the gas.

The Darcy permeability coefficient depends on the gas pressure. In order to describe a coal sample with a single permeability coefficient value at a given confining pressure, the Klinkenberg correction was applied, which determines the permeability for a gas pressure close to infinity [52]:

$$k_g = k_\infty \left(1 + \frac{b}{p_{avg}} \right),\tag{2}$$

where: k_{∞} [m²]—is the Klinkenberg absolute permeability at a gas pressure tending to infinity; *b* [Pa]—is the Klinkenberg slippage factor; p_{avg} [Pa] = $\frac{p_{in}+p_{out}}{2}$ —is the average gas pressure in the sample.

The procedure for determining the permeability coefficients followed that presented in Kudasik [31] and Braga and Kudasik [32] and consisted of measuring the gas flow rate Q at the outlet of the sample at different gas pressures at its inlet p_{in} and outlet p_{out} . By substituting these parameters into Equation (1), it was possible to determine the Darcy permeability coefficients. Figure 5 shows schematic diagrams of the changes in p_{in} , p_{out} , and Q parameters (Figure 5a) and the method of determining the Klinkenberg permeability coefficients k_{∞} (Figure 5b), based on the values obtained for these parameters at the seepage tests of a specific gas under stationary conditions (points P1, P2, P3, P4, and P5). These measurements were repeated at 4 different confining pressures (p_h) for both helium (He) and carbon dioxide (CO₂). Based on the determined Klinkenberg absolute permeability coefficients k_{∞} , at different confining pressure p_h conditions, it is possible to determine the relationship $k_{\infty}(p_h)$ (Figure 5c). In the work, this relationship was the main subject of detailed analysis.



Figure 5. Schematic diagrams of the changes in p_{in} , p_{out} , and Q parameters (**a**), the method of determining the Klinkenberg permeability coefficients k_{∞} (**b**), and the relationship $k_{\infty}(p_h)$ (**c**).

The tests were carried out in isothermal conditions at a temperature of 30 °C, which was ensured by placing the measuring equipment in a Q-Cell 1400 (Pol-Lab) thermostatic cabinet.

5. Results

Based on the registered measurements, it was possible to calculate the Klinkenberg absolute permeability coefficients k_{∞} of both coals, at 4 different confining pressures and in relation to two gases (He and CO₂). The values of the determined coefficients are shown in Table 3.

	Confining Pressure <i>p_h</i> [MPa]	Klinkenberg Permeability Coefficients in Relation to:					
Sample		He	2	CO ₂			
		$k_{\infty}[mD]$	b	$k_{\infty}[mD]$	b[Mpa]		
	1.5	81.032	0.004	71.696	0.003		
<i>#C</i> 1 · 1 · <i>#</i>	10	33.798	0.027	23.435	0.049		
"Sobieski"	20	26.029	0.029	18.563	0.040		
	30	21.902	0.023	16.218	0.033		
	1.5	6.615	0.005	2.038	0.020		
//C:1//	10	2.248	0.039	0.657	0.011		
Silesia	20	1.413	0.030	0.427	0.011		
	30	0.758	0.020	0.258	0.016		

Table 3. Results of coal permeability to He and CO₂ at different confining pressures.

Changes in the permeability of coal to He and CO_2 due to increasing confining pressure are shown in Figure 6. For each value of the Klinkenberg permeability coefficient determined from the fit in accordance with the procedure presented in Figure 5, error bars were also determined, which are the uncertainty of determining the y-intercept constant of Equation (2). The maximum error bar value was about 20% of the k_{∞} value for the highest confining pressure. Based on the obtained results, it can be seen that coal permeability decreases with an increase in confining pressure, and this decrease can be described by an exponential function:

$$k_{\infty} = \kappa_1 + \kappa_0 \cdot \exp\left(-\frac{p_h}{\vartheta}\right),\tag{3}$$

where: κ_1 , κ_0 —are the permeability constants; ϑ —is the confining pressure constant.



Figure 6. Dependence of coal permeability on confining pressure.

Using Equation (3), it is possible to determine the Klinkenberg absolute permeability coefficient for a stress-free sample ($p_h = 0$), and this value will be $\kappa_1 + \kappa_0$. The permeability of "Sobieski" coal to He under stress-free conditions was calculated at 100.4 mD. Under confining pressure condition ($p_h = 30$ MPa), this value decreased almost 5 times. A similar decrease in k_{∞} value was for CO₂. The calculated permeability of "Silesia" coal to He at the stress-free sample was 8.16 mD, and an increase in the confining pressure to 30 MPa caused a decrease in permeability by more than an order of magnitude. A similar order of magnitude decrease was observed for CO₂.

The permeability of coal to He was higher than to CO_2 , which results, among others, from the particle size of both gases, where the kinetic diameter of He is 0.26 nm, and the kinetic diameter of CO_2 is 0.33 nm.

The "Sobieski" coal sample, which was characterized by almost two times higher sorption capacity and almost three times higher specific surface area, had about one order of magnitude higher permeability to He and CO₂ than the "Silesia" coal.

By substituting the parameter of the confining pressure on the x axis (Figure 6) to the depth of the coal seam, using the formula for hydrostatic pressure, the following relationship can be obtained:

$$h = \frac{p_h - p_{atm}}{\rho_{avg} \cdot g},\tag{4}$$

where: h [m]—is the depth of the coal seam; $\rho_{avg} \left[\frac{\text{kg}}{\text{m}^3}\right]$ —is the average density of overburden rock ($\rho_{avg} \approx 2.5$), $g \left[\frac{\text{m}}{\text{s}^2}\right]$ —is the acceleration of gravity.

Using Equation (4), it is possible to estimate the dependence of coal permeability in relation to CO_2 , depending on the depth of the coal seam (Figure 7). This procedure was performed for CO_2 in the context of assessing the possibility of using bituminous coal as a sorbent for underground CO_2 storage.

When analysing the relation between the permeability of both coals and their depth of deposition (Figure 7), it may be noticed that according to the classification [53], the sample of "Sobieski" coal belongs to the rocks with good permeability. In case of the "Silesia" coal, below the depth of deposit of 250 m, this coal is classified as poorly permeable rock.



Figure 7. Coal permeability to CO₂ at different depths of the seam.

6. Conclusions

Fluid transport processes in a porous medium can be divided into diffusion—which occurs mainly in micropores, and seepage—which takes place in meso- and macropores [54]. Among the investigated coals, the sample "Sobieski" was characterized by a higher distribution of meso- and macropores (Figure 4b), which also resulted in higher values of permeability coefficients than the sample of "Silesia" coal. The difference in grain and pore size of the two coal samples can also be seen in the SEM images (Figure 3), which also has a direct impact on their permeability differences.

The poorly developed pore network of both samples (Figure 4) resulted directly into their low permeability to He and CO_2 . An additional factor changing the structural properties as well as reducing the seepage properties of the coals was the confining pressure, which simulated in situ conditions.

Bituminous coal is a rock with good sorption properties which has been used for many years for injection and underground storage of CO_2 . However, many of the attempts to inject CO_2 into coal seams have been unsuccessful or the effectiveness of in situ tests has not been satisfactory [29]. In addition to good sorption properties and preferential sorption to CO_2 in relation to CH_4 of coal, permeability is an important parameter for effective application of CCS and CO_2 -ECBM technologies. The obtained results showed that the coal permeability decreases with an increase in confining pressure, and this decrease can be described by an exponential function. The coal briquettes tested had different permeabilities, where the "Sobieski" sample was highly permeable and the "Silesia" sample was poorly permeable. The study did not take into account swelling, which, in addition to confining pressure, could also affect the reduction in permeability. By relating the obtained results to the in situ conditions, it can be concluded that at depths below 250 m, coal can be a rock poorly permeable to CO_2 . However, coal briquette samples with porosities similar to the original raw coal were used in this study, which means that under in situ conditions, raw coal permeability values may differ from those determined in the laboratory. **Author Contributions:** Conceptualization, M.K. and N.S.; designing, M.K., N.S. and L.T.P.B.; formal analysis, M.K. and L.T.P.B.; investigation, N.S. and L.T.P.B.; writing—original draft preparation, M.K.; writing—review and editing, M.K. and N.S.; visualization, M.K.; supervision, N.S. and L.T.P.B.; project administration, N.S.; funding acquisition, N.S. All authors have read and agreed to the published version of the manuscript.

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