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AN IMPACT OF MECHANICAL STRESS IN COAL BRIQUETTES ON SORPTION OF CARBON DIOXIDE

ZALEŻNOŚĆ NAPRĘŻENIE – SORPCJA DITLENKU WĘGLA NA PODSTAWIE BADAŃ WYKONANYCH NA BRYKIETACH WĘGLOWYCH

The presence of gases (methane or carbon dioxide) in hard coal is connected with numerous threats for miners employed in underground mining facilities. When analyzing the coal-methane system, it is necessary to determine the relationship between pressure and gas sorption. Such a relationship should be determined under conditions similar to the natural ones – when it comes to both temperature and pressure. The present paper discusses the results of research conducted with the use of coal briquettes under the state of mechanical stress. Carbon dioxide sorption isotherms were determined for different values of stress affecting the coal material. For five coal samples collected in different mines of the Upper Silesian Coal Basin, Langmuir's sorption isotherms were determined. The results point to significant impact that mechanical stress has upon the sorption process. It is about 1 percent of the value obtained for coal not subjected to stress per 1 MPa. The research results can also prove useful when analyzing hard coal seams from the perspective of their carbon dioxide sequestration abilities.

Keywords: CO_2 storage, gas danger in coal mines CO_2 sorption on coal

Obecność gazów (metanu lub dwutlenku węgla) w węglu kamiennym połączona jest z licznymi zagrożeniami dla pracowników zatrudnionych w podziemnych zakładach górniczych. Analizując układ węgiel-metan, konieczne jest określenie zależności między ciśnieniem a wielkością sorpcji gazu. Taki związek powinien być określony w warunkach podobnych do tych naturalnych – jeśli chodzi zarówno o temperaturę układu węgiel-metan i ciśnienie. Niniejszy artykuł omawia wyniki badań prowadzonych z użyciem brykietów węglowych poddanych naprężeniom mechanicznym. Próbki węgla pobrano w pięciu kopalniach Górnośląskiego Zagłębia Węglowego, pokazanych na mapie z Rys. 1. Pobrane próbki poddane zostały analizie technicznej, analizie składu mecerałowego oraz badaniom powierzchni właściwej metodą sorpcji niskociśnieniowej Właściwości pobranych węgli zestawiono w Tab. 1. W brykieciarce, pokazanej schematycznie na Rys. 2 wykonano brykiety węglowe o znanych właściwościach oraz znanych wartościach rezydualnych naprężeń radialnych. Wartości tych naprężeń zestawiono w Tab. 3. Wyznaczono

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izotermy sorpcji (z modelu Langmuira) dla dwutlenku węgla dla różnych wartości naprężeń mechanicznych oddziałujących na materiał węglowy. Wyniki wskazują na znaczący wpływ naprężeń w brykiecie na proces sorpcji. Wywarcie naprężenia mechanicznego wartości około 10 bar skutkuje ograniczeniem maksymalnej sorpcji Langmuira o około 1%, co pokazano na Rys. 9. przedstawiającym zależność maksymalnej sorpcji Langmuira od wartości naprężeń radialnych dla wszystkich przebadanych próbek węgla. Wyniki badań mogą okazać się przydatne przy analizie pokładów węgla z punktu widzenia ich zdolności do sekwestracji dwutlenku węgla.

Slowa kluczowe: sekwestracja dwutlenku węgla, zagrożenie gazowe w kopalniach, sorpcja na węglu kamiennym

1. Introduction

There are three areas of hard coal exploitation in Poland: the Lower Silesian Coal Basin (LSCB), the Upper Silesian Coal Basin (USCB), and the Lublin Coal Basin (LCB) – cf. Fig. 1. In the LSCB, the exploitation was terminated in the early 1990s. One of the reasons why this happened was the presence of numerous natural threats – among them, the gas (especially carbon dioxide) and coal outburst hazard. Currently the exploitation is conducted in the "Bogdanka" coal mine in the Lublin Coal Basin, and in 28 coal mines in the Upper Silesian Coal Basin. The USCB is located in the southern part of Poland and in the northern part of the Czech Republic. Its total area is ca. 7400 km^2, out of which 5800 km^2 lies within the territory of Poland (Jureczka & Kotas, 1995). The exploitation of coal seams in the USCB started as early as in the 16th century. The USBC is at present the largest area of hard coal exploitation in Europe. In 2014, over 63 mln tons were extracted from its mines.

In the USCB, the process of coal exploitation is accompanied by numerous natural threats, the most important of which are: the rockburst hazard (e.g. Konicek et al., 2013) and the methane hazard (Kędzior, 2009, Krause, Skiba, 2014). Methane content in a coal seam increases with the depth at which the seam is located – at the depth of 1400 m, it may exceed 20 m^3/Mg$_{daf}$ (Kędzior, Jelonek, 2013). Information regarding the geological structure, the maceral composition and the methane-bearing capacity of the USCB coal seams can be found in (Kędzior, 2015). The map of the Upper Silesian Coal Basin is presented in Fig. 1.

A very important element of the analysis and evaluation of the state of risk concerning the gas hazard in mines is getting to know the sorption properties of coal in relation to gas. This knowledge is particularly important in terms of assessing the possibility of storing CO_2 in coal seams and during CO_2/CH_4 exchange sorption studies (Dutka et al., 2013). Attempts were made in the Upper Silesian Coal Basin (van Bergen et al., 2004).

The sorption capacity of coal depends, among other things, on the temperature of the coal-gas system, the coal rank, the maceral composition of coal, and coal humidity. Relevant information on these parameters can be found in numerous publications (Wierzbicki, 2013; 2013a; Baran et al., 2016; Skoczylas, 2016; Wang et al., 2015; Weishauptová et al., 2015;, Czerw et al., 2017; Kudasik et al., 2017). The sorption capacity of coal is also influenced by the applied stress. Although this issue seems to be very important – both from the perspective of safety and the possibilities of underground carbon dioxide sequestration – publications that describe the results of research in this area are scarce. The reason may be that it is quite difficult to measure minor sorption changes in the course of the long measurement process.

One of the first papers discussing the impact of mechanical stress on sorption was paper of Denis et al. (2009). The authors conducted their research using CO_2 and CH_4. They found

Fig. 1. The map of the Polish part of the USCB

that the application of 13.8 MPa of confining stress contributed to 64% and 91% CO_2 and CH_4 sorption capacity reduction, respectively. In the paper (Hol et al., 2011), the title itself says that "Applied stress reduces the CO_2 sorption capacity of coal". Using pore pressure values between 10 MPa and 20 MPa, and applying the temperature of 400 °C, they reduced sorption capacity by an amount in the range between 0.014 mol g_{coal}^{-1} $MPa^{-1}$and 0.014 mmol g_{coal}^{-1} MPa^{-1}. An instrument for investigating sorption under the state of mechanical stress and the initial research results are discussed in (Dutka, Topolnicki, 2011).

The hard coal from the USCB is characterized by low values of the effective diffusion coefficient *De* (Crank, 1975). The values of this coefficient fall within the range of 5×10^{-10} cm^2/s do $2\times10^{-9}cm^2/s$. Therefore, the processes of methane accumulation and release from coal occur very slowly. For example, for De = 5×10^{-9} cm^2/s, the release of 98 percent of methane from a granular coal sample whose diameter is 0.2 mm lasts ca. 20 hours. The model research based on the solution of the unipore diffusion equation shows that it would take about 100 days to sorptively fill a sphere of the diameter of 5 cm in about 98 percent. This is the time necessary to determine one sorption point. The results demonstrate how time-consuming it would be to conduct relevant research on solid coal samples. This, together with the difficulties with ensuring the tightness of the equipment for such a long time, would result in abandoning the research described in (Dutka, 2011) until later. This is why the research discussed here was carried out with the use of coal briquettes pressed without binder.

As a research material, coal briquettes have numerous pluses: they can be made in a known, controlled and repeatable way. Because of this, they are widely used in investigations carried out under laboratory conditions (Skoczylas, 2012; Dutka et al., 2013; Sobczyk, 2013).

2. The coal samples and measurements tools

The purpose of the research described in the present paper was to determine the isotherms of the sorption of CO_2 on coal by means of the volumetric method, under the conditions of the mechanical stress exerted on a coal sample. The research was conducted with the use of coal samples collected from five hard coal mines: „Brzeszcze", „Budryk", „Pniówek", „Sobieski", and „Zofiówka", marked on the map in Fig. 1. Out of these mines, only the "Sobieski" mine is a non-methane one. The rest of the mines are characterized by a high or very high level of risk as regards the methane hazard. In 2014, the total methane content in the "Budryk" mine was 69.93 mln m^3, in the „Brzeszcze" mine – 94.88 mln m^3, in the „Pniówek" mine – 122.93 mln m^3, and in the "Zofiówka" mine – 80.64 mln m^3. As can be seen, the "Pniówek" mine was characterized by the highest methane content in Poland.

Table 1 contains information on the coals that were investigated. The maceral composition of coal was established by means of an optical microscope, at 400× magnification and with immersion. The dominant maceral group in the investigated coals is the vitrinite group. The reflectance of vitrinite varies from 0.65 % to 1.14 %. Ash content falls within the range of 2.60%-11.54 %. The skeletal density was determined by means of the helium method, with the AccuPyc pycnometer provided by Micromeritics. The specific surface area was measured using the method of the low-pressure carbon dioxide sorption, by means of the ASAP 2020 analyzer. The surface in question is between 110.2 m^2/g and 168.9 m^2/g.

TABLE 1

The location of the samples and the results of the technical and maceral analysis of coal

No. of sample		**PR_1/137**	**PR_2/208**	**PR_3/213**	**PR_4/219**	**PR_5/231**
Coal Mine		**„Sobieski"**	**„Budryk"**	**"Pniówek"**	**„Brzeszcze"**	**"Zofiówka"**
Seam No.		**207**	**358/1**	**404/1**	**401**	**411/2**
Maceral groups, %	Witrinite	72.8	69.4	79.2	63.2	68.4
	Inertynite	16.8	26.7	20.1	26.1	22.7
	Liptinite	9,4	3.8	10.7	0.7	8.9
Ash cont. A^a, %		11.5	2.6	3.9	6.0	7.1
Volatile matter V^{daf}, %		32.3	29.1	26.3	33,1	27.1
Vitrynite reflectance *Rr*, %		0.71	0.99	1.05	0.65	1.14
Helium density ρ, g/cm^3		1.44	1.47	1.38	1.38	1.34
Dubinin-Radushkevich surface area, m^2/g		168.9	121.9	110.2	149.5	124.8

The samples collected in mines were ground, and then sieved with a laboratory sifter. The grain fraction of <0.2 mm was separated. The material was homogenized (averaged), and then divided – by means of quartering – into portions of ca. 10 g each. The analysis of sorption in coal was carried out using a material in a loose state, and on coal briquettes of different porosities. Measurements of ash content, volatile matter and vitrinite reflectance were made in accordance with Polish standards.

Coal briquettes were made from the described samples The process of briquetting was carried out using the double-sided pressing technology. The briquetting machine had a form of a steel tube, whose external diameter was 20 mm, and the wall was 2.5 mm thick (Fig. 2).

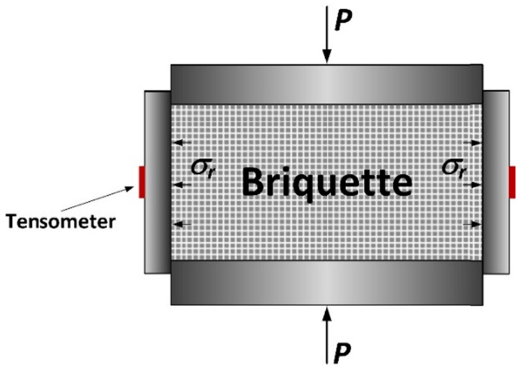

Fig. 2. The process of pressing the coal briquette

3. Theoretical foundations of research methods

While pressing the briquette, some endometric research was carried out in order to determine the lateral thrust coefficient η and the Poisson ratio υ. To this end, extensometers were fixed on the side surface of the form, the purpose of which was to measure the strain caused by the application of the axial pressing stress. The relationship between the stresses is as follows:

$$\sigma_r = \sigma_\phi = \eta\sigma_z$$

where:

$$\eta = \frac{\sigma_r}{\sigma_z} = \frac{\upsilon}{1-\upsilon}$$

υ – Poisson ratio.

Knowing the dimensions of the briquetting form and Young's modulus of steel, it is able to calculate the value of the radial stress σ_r of the coefficient η, as well as the value of the Poisson ratio. Detailed information in this regard is at work (Wierzbicki, 2003). A sample relationship between the axial and radial stress was shown in Fig. 3. The results were presented in Table 2. Also, the friction coefficient between coal and steel was measured. The method of measurement is shown in the paper (Wierzbicki, 2003a). Its value was similar for all the investigated briquettes. The adopted mean value was $f = 0.51$.

TABLE 2

The results of the quasiedometric measurements carried out on the coal briquettes, and of the measurements of the friction coefficient between coal and steel

	PR_1/137	PR_2/208	PR_3/213	PR_4/219	PR_5/231
η	*0.22*	*0.22*	*0.28*	*0.32*	*0.27*
υ	0.18	0.18	0.22	0.24	0,21
f	0.51				

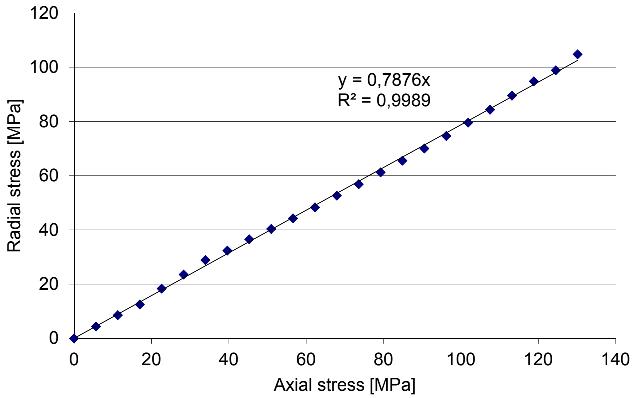

Fig. 3. The relationship between the radial and axial stress during the process of pressing the briquette from the PR_3/213 sample

From the perspective of the objectives of the present analysis, what is of interest to us is knowing the values of the residual radial stress. In order to calculate the mean values of residual stresses, the values obtained in the course of the tensometric measurements were used – cf. Fig. 2. Sample changes of the tube strain during the process of pressing the briquette were shown in Fig. 4. The red curve shows the strains occurring during pressing with the force of 4 kN. The maximum strain of the tube was 0.121 percent. Subsequently, the briquette was relieved. As a result, some residual stresses remained in the briquette, which was demonstrated by the strain of more or less 0.0028 percent. The next step was to increase the pressing force to the value of 10 kN (the blue curve), and then relieving the briquette again. The strain of the tube after applying the pressing force of 10 kN was 0.296%, and the residual strain was 0.006%. Knowing the value of the strain, Young's modulus for steel (E = 210 GPa) and the geometric dimensions of the cylinder, we can calculate the value of the radial stress in the coal briquette from the following formula:

$$\sigma_r = \frac{E\varepsilon\left(D_2 - D_1\right)}{2D_1}$$

where:

- ε — the strain;
- E — the steel elasticity modulus, MPa;
- D_1 and D_2 — the internal and external diameter of the cyllinder (respectively), m;
- h — the cylinder height, m.

The graphs presenting changes in the radial stress during the process of pressing were shown in Fig. 5. From the graphs, one can also read the residual stress values after consecutive steps of pressing the briquette with the force of 4 kN and 10 kN. For sample PR_4/219, these values are 5.95MPa and 10.43MPa respectively.

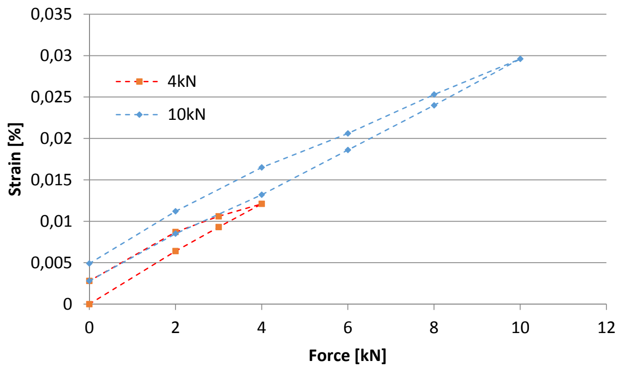

Fig. 4. The relationship between the pressing force for the pressing force values of 4 kN and 10 kN (PR_4/219)

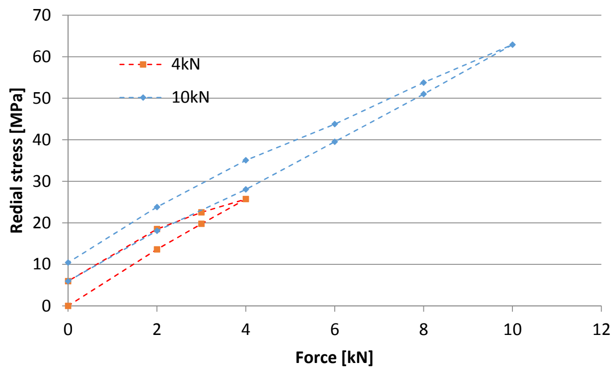

Fig. 5. The relationship between the pressing force and the radial stress, for the pressing force values of 4 kN and 10 kN (PR_4/219)

The helium porosity of coal briquettes were calculated on the basis of the known apparent briquette density ρ_{br} and the skeletal coal density ρ_{sk}, from the following formula:

$$\phi = 1 - \frac{\rho_{br}}{\rho_{sk}}$$

The apparent porosity was determined by means of the quasi-liquid method, using the instrument GeoPyc provided by Micrometrics. The helium porosity ϕ shall be from now on referred to as simply porosity of coal briquettes. The porosity of briquettes depends on the applied pressing stress σ_{z0}, which is demonstrated by the graphs from Fig. 6.

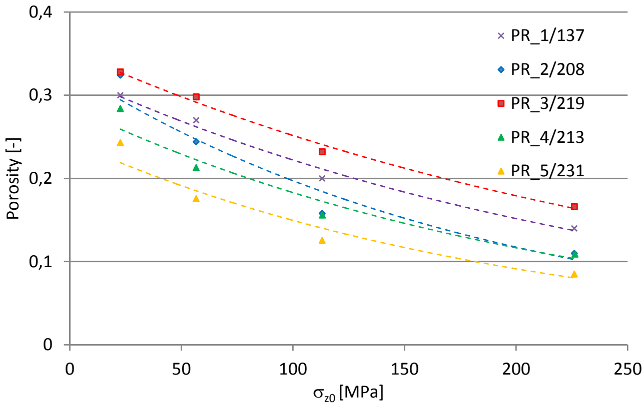

Fig. 6. The relationship between the porosity of briquettes and the value of the axial stress during pressing

4. Conducting the research and the research results

The cycle of research, carried out for each of the collected coal samples, encompassed the following activities:

a) determining the skeletal helium density of the coal matrix,
b) preparing a loose sample for sorption measurements,
c) determining the helium capacity of coal and the form,
d) determining the isotherm of sorption of carbon dioxide in relieved (heap) coal, on the basis of at least 5 sorption points, in the temperature of 298 K, within the range of pressure values of up to 1.6 MPa,
e) preparing the coal briquette by using the pressing force of 4 kN, and performing the tensometric measurement of the form strain,
f) calculating porosity and the mean residual stress σ_r, for a given pressing load σ_{z0},
g) performing sorption measurements in order to determine the sorption isotherm for a known mean value of residual stress σ_r,
h) performing the activities described in points e)-g) for another coal briquette, prepared using a larger stress σ_{z0}. The applied pressing forces were: 4 kN, 10 kN, 20 kN, and 40 kN.

The values of residual radial stresses, determined from the tensometric measurements and porosities ϕ of the coal briquettes, were presented in Table 3. The maximum residual stresses in briquettes made with the use of force of 40 kN, were 21 MPa, which constitutes ca. 10 % of the value of the pressing load.

The isotherms of sorption of carbon dioxide in coal were determined by means of the volumetric method. Before the sorption measurements briquettes (together with the tube) were outgassed under the pressure of 5×10^{-2} Pa. The changes of pressure during sorption were measured by means of pressure transducers, with the frequency of 0.5 Hz. The measuring system was immersed in water of the constant temperature of 298 +/-0.05 K. For each of the briquettes (whose residual stress values differed), a sorption isotherm was determined within the range of up to 10 bar, based on at least 5 measurement points. The results obtained in such a way were then matched with Langmuir's sorption equations:

$$a = \frac{a_m bp}{(1+bp)}$$

where:

a — the sorbed amount of carbon dioxide at the equilibrium pressure p, cm^3/g,
a_m — the Langmuir sorption capacity at $p \to \infty$ (saturation state), cm^3/g,
b — a constant characteristic for the coal-gas system, bar^{-1},
p — the gas pressure, bar.

TABLE 3

The briquette porosities and their corresponding residual radial stress values σ_r

σ_{z0}, MPa	PR_1/137		PR_2/208		PR_3/213		PR_4/219		PR_5/231	
	σ_r, MPa	ϕ, -	σ_r, MPa	ϕ, -	σ_r, MPa	ϕ, -	σ_r, MPa	ϕ, -	σ_r, MPa	ϕ, -
22.6	1.16	0.30	1.18	0.32	2.66	0.28	5.95	0.33	2.66	0.24
56.6	4.97	0.27	5.13	0.24	6.30	0.21	10.43	0.30	6.30	0.17
113.1	10.02	0.20	10.44	0.16	11.34	0.16	12.95	0.23	11.34	0.12
226.1	13.3	0.14	13.13	0.11	17.40	0.11	21.00	0.17	18.34	0.09

The results were re-calculated to *dry ash free* basis (daf) and shown on graphes from Figure 7. The solid lines were used to mark Langmuir's isotherms. The positioning of isotherms in the figures suggests that an increase in the radial stress value results in reducing the carbon dioxide sorption in the investigated coals.

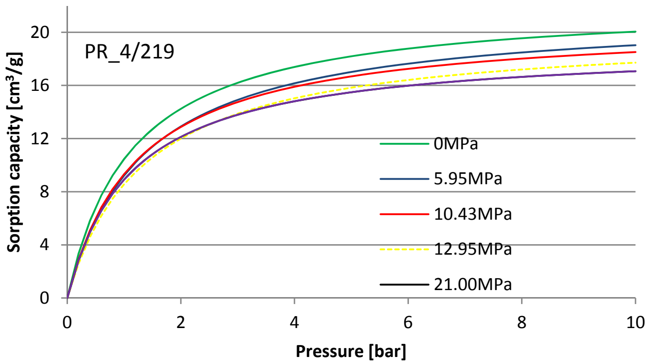

Fig. 7. Sorption isotherms in the sample PR_4/219 under the temperature of 298 K

The values of the Langmuir's sorption isotherms' coefficients were collected in Table 4, which also provides the previously determined values of residual radial stresses.

TABLE 4

The values of the constants of Langmuir's equation for particular coal samples

Force, kN		0	4	10	20	40
PR_1/137	σ_r, MPa	0	1.16	4.97	10.02	13.03
	a, cm^3/g	24.41	24.31	24.02	22.48	21.61
	b, bar^{-1}	0.78	0.66	0.57	0.61	0.50
PR_2/208	σ_r, MPa	0	1.18	5.13	10.44	13.30
	a, cm^3/g	16.07	16.23	15.51	14.67	13.85
	b, bar^{-1}	0.55	0.70	0.57	0.56	0.54
PR_3/213	σ_r, MPa	0	2.66	6.30	11.34	17.40
	a, cm^3/g	20.79	20.70	20.50	18.41	17.31
	b, bar^{-1}	0.66	0.63	0.69	0.71	0.71
PR_4/219	σ_r, MPa	0	5.95	10.43	12.95	21.00
	a, cm^3/g	22.32	21.58	20.78	19.72	17.48
	b, bar^{-1}	0.88	0.75	0.83	0.78	0.88
PR_5/231	σ_r, MPa	0	2.66	6.30	11.34	18.34
	a, cm^3/g	20.64	19.33	18.04	17.78	15.75
	b, bar^{-1}	0.56	0.62	0.74	0.61	0.81

The PR_1/137 sample from the "Sobieski" coal mine proved to be the best sorbent to carbondioxide. Its maximum sorption capacity under zero load was 24.41 cm^3/g$_{daf.}$ The smallest sorption capacity is displayed by the PR_2/208 coal from the "Budryk" coal mine.

The changeability of Langmuir's maximum sorption capacity in the function of the radial stress was shown in Fig. 8. The changes of the maximum sorption capacities can be described with linear equations $a = a_m{}^{-}k\sigma_r$, where a_m is the Langmuir sorption value for $\sigma_r = 0$. The values of the coefficients k matched with the results of particular tests, together with the values of R^2, were presented in Table 5. The same table provides the percentage changes of Langmuir's maximum sorption capacity caused by the presence of mechanical stress of 1MPa – $100\,k/a_m$

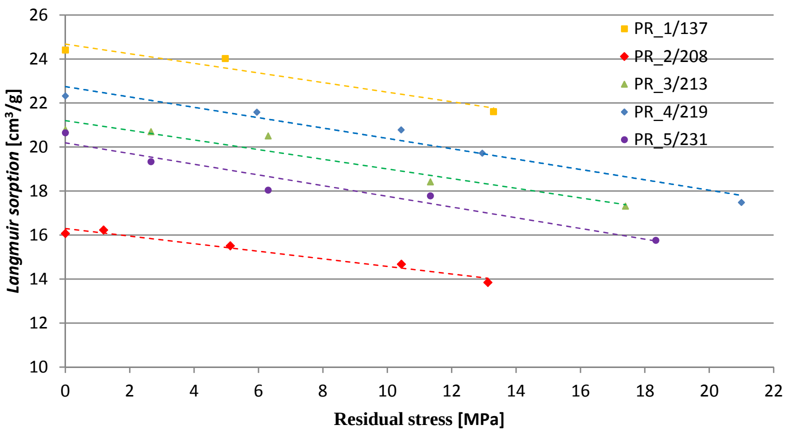

Fig. 8. The relationship between Langmuir's maximum sorption and the value of the radial stress in a briquette

(cf. column 5). These changes vary from $0.0088a_m/\mathrm{MPa}$ to $0.0120a_m/\mathrm{MPa}$. Average changes were about $0.01\ a_{\max}/\mathrm{MPa}$.

TABLE 5

Changeability of Langmuir's coefficients for particular samples

1	2	3	4	5
Sample no.	$a_{\max}$, cm^3/g_{daf}	k, $cm^3 g_{daf}^{-1}\ MPa^{-1}$	R^2	$100k/a_m$, MPa^{-1}
PR_1/137	24.68	0.218	0.938	0.88
PR_2/208	22.74	0.235	0.962	1.03
PR_3/213	21.20	0.219	0.926	1.03
PR_4/219	20.19	0.243	0.943	1.20
PR_5/231	16.30	0.172	0.964	1.06

5. Conclusions

Conducted a series of laboratory studies on the sorption of coal briquettes allow the following conclusions:

- The coal briquettes constitute a material which is fit for conducting repeatable research under controlled conditions.
- The stresses of 1 MPa in a coal briquette reduce Langmuir's maximum sorption by ca. 1 percent (on average) of the value obtained for coal under zero stress.
- If we assume that the mean density of overlay rocks is $\rho = 2.5\ \mathrm{Mg/m^3}$, then the hydrostatic stress at the depth of 1 000 m can be estimated as 25 MPa. For such a stress value, the reduction of the sorption capacity shall be ca. 25 percent (from 22 percent in the case of coal from the "Sobieski" coal mine to 30 percent in the case of the coal from the "Zofiówka" coal mine).
- As the depth increases, the sorption capacities of coal may be diminished, which – given the constant methane-bearing capacity – shall have a negative impact on the value of the seam pressure in hard coal beds.
- The influence of mechanical stress on the sorption capacity is so large that it should be taken into account in the aspect of the underground storage of CO_2.

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