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**THE EFFECT OF TEMPERATURE ON THE SORPTION PROPERTIES OF COAL
FROM UPPER SILESIA COAL BASIN, POLAND****WPLYW ZMIAN TEMPERATURY NA WŁASNOŚCI SORPCYJNE NA PODSTAWIE BADAŃ
WYBRANYCH WĘGLI Z GÓRNOŚLĄSKIEGO ZAGŁĘBIA WĘGLOWEGO**

This paper presents the results of studies on gas sorption performed by means of the gravimetric method. The tests were performed on two coal samples of different metamorphism degrees, came from two regions of Upper Silesian Coal Basin, Poland. The changes in sorption capacity of coals were measured in the pressure range from 0.1 MPa to 17 MPa and in temperatures ranging from 291K to 333K. Coal of a lower coalification degree was a better methane sorbent. Changes in sorption capacity of tested coals were linearly dependent on the temperature. The increase in temperature of 10K reduces the Langmuir sorption of about 0.7-0.8 [cm³/g]. Such increase of temperature causes a nonlinear increase of the Langmuir pressure (b^{-1}). These results showed that the rise of rock temperature, caused by geothermal gradient, can induce a significant increase of equilibrium pressure of methane in coal seam. An increase of coal seam temperature may cause an increase of gas and coal outburst risk in a coal mine.

Keywords: methane sorption, temperature, coalbed methane

W pracy przedstawiono wyniki badań sorpcyjnych wykonanych na dwóch rodzajach węgla o różnym stopniu uwęglenia. Próbki do badań pochodziły z kopalń „Sobieski” w Jaworznie oraz „Pniówek” w Pawłowicach. Badania polegały na przeprowadzeniu serii pomiarów izoterm sorpcji w różnych temperaturach układu węgiel-metan. Badania przeprowadzono metodą grawimetryczną, polegającą na pomiarze zmian masy węgla wywołanych sorpcją metanu. Maksymalne ciśnienie sorpcji wynosiło 1.7 MPa. Zakres zmian temperatury wynosił 40K. Dla węgla z kopalni „Pniówek” wykonano dodatkowo pomiary desorpcji metanu we wszystkich badanych temperaturach. Wyniki pozwoliły na wyznaczenie izoterm sorpcji przedstawionych w pracy oraz dopasowanie współczynników izoterm Langmuir’a.

Węgiel o niższym stopniu uwęglenia okazał się lepszym sorbentem dla metanu. Zmiany wartości maksymalnych sorpcji Langmuira w funkcji temperatury mają charakter liniowy a współczynniki kierunkowe prostych dopasowanych do wyników pomiaru mają bardzo zbliżone wartości. Analizując względne zmiany pojemności sorpcyjnej w procesach sorpcji i desorpcji można, że największe różnice pomiędzy wartościami sorpcji i desorpcji występują przy niskich wartościach ciśnień. Przy ciśnieniu 0.1 MPa

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wynoszą one od ok. 16% do ok. 20%. Przy ciśnieniu powyżej 0.5 MPa wielkość histerezy nie przekracza 5%. W pracy przedstawiono również rozważania dotyczące wpływu zmian temperatury złożowej, wywołanego gradientem geotermicznym na zmiany ciśnienia równowagowego metanu w pokładzie. Do obliczenia wykonano dla przykładowych głębokości od 600 m do 1400 m. Wyniki pokazują, że wraz ze wzrostem metanonośności i głębokości gwałtownie może wzrastać ciśnienie równowagowe metanu w pokładach węgla. Ciśnienie metanu jest jednym z najważniejszych parametrów wpływających na możliwość występowania wyrzutów metanu i skał w kopalniach. Należy przypuszczać, że zagrożenie wyrzutami metanu i skał będzie wzrastało wraz z głębokością prowadzenia robót górniczych.

Słowa kluczowe: metan, sorpcja, temperatura, zagrożenie metanowe

1. Introduction

Yearly coal production in Poland approached 79 mln ton in 2012, about 75% of coal came from methane seams. 26 accidents associated with the methane hazard were registered during the period 2002-2012, with 62 fatalities. Methane emission per one ton of mined coal is now 50% higher than 10 years before. The maximal methane contents in coal beds exceeds now $15\text{m}^3/\text{Mg}_{\text{daf}}$. Methane explosion hazard and gas and coal outbursts hazard considered to be a major natural hazard experienced in the Polish coal mines (Szlązak & Kubaczka, 2012; Młynarczuk & Wierzbicki, 2009) and in the world (He & Song, 2011). The gas and rock outburst hazard is also present in copper mines (Młynarczuk & Wierzbicki, 2013).

Methane is present in coal basically in two different forms: as a gas sorbed on the surface of pores and microcracks and methane filling pores and cracks in coal, not bound to the surface and hence referred to as free gas. The state of equilibrium is established between the amount of free and adsorbed methane, associated with the methane pressure, pore volume and the coal's internal surface. Gas in the sorbed state accounts for 85-90% of total gas contained in coal (Ceglarska-Stefańska & Brzóska, 1998). The sorption capacity of coal is associated with the properties of the coal-gas system, such as pressure, temperature, moisture content, metamorphism of coal and maceral composition. The depth of mining operations in Poland progressively increases- by 8m-10m yearly, on the average. In Chinese mines, the mine depth can increase at a rate of 50m per year (Wang et al., 2013). The geothermal gradient gives rise to a temperature increase, which, in turn, leads to deterioration of the methane sorption capacity on coal (Levy et al., 1997; Nodzeński & Hołda, 2001; Sakurovs et al., 2008). Deterioration of the coal's sorption capacity in relation to the constant or growing methane content, brings about an increase in the equilibrium pressure of methane in underlying seams. The methane pressure is a major determinant of the gas and rock outburst occurrence (Lama & Bodziony, 1996). The influence of pressure as a factor triggering the outburst is well evidenced in research conducted by Skoczylas (2012). who ran a series of experiments to test the probability of outburst occurrence depending on the equilibrium pressure of gas. The role of methane pressure as an indicator of the potential outburst occurrence is corroborated by numerical modelling data (Xu et al., 2006). Three methane and rock outbursts were registered in recent years in the Upper Silesia Coal Basin (Wierzbicki, Młynarczuk, 2006, Młynarczuk, Wierzbicki, 2009). They might have been associated with the growing methane pressure due to the geothermal gradient. Further research aimed at determining the influence of temperature on the process of methane sorption on coal is fully justified.

2. Coal samples

Testing was done on two coal samples from the Upper Silesia Coal Basin (USCB). The USCB is located in southern Poland and in the northern part of the Czech Republic and it covers an area of about 7,400 km². More detailed information about geology of this region can be found in other studies (Nowak, 2004; Kędzior, 2011). Coal samples were chosen in a way ensuring similar maceral compositions and different metamorphism. The first sample was collected from methane-free coal seam 209 in the “Sobieski” colliery in Jaworzno. This colliery is located in the northern part of the Upper Silesia Coal Basin. The other coal sample was obtained from coal seam 404/1 in the “Pniówek” colliery in Pawłowice (coal sample “P”), in the southern part of the Polish part USCB. Coal samples in the form of polished sections were subjected to a maceral composition analysis. A technical analysis of selected coal samples was performed to determine their vitrinite reflectance. The maceral composition and other parameters are summarised in Table 1.

TABLE 1

Coal mine	Maceral group [%]			Reflectance of vitrinite [%]			Technical analysis [%]	
	Vitrinite	Inertinite	Liptinite	R _r	R _r _{max}	R _r _{min}	V _{daf}	A ^a
Pniówek	76,30	21,00	2,20	1,08	1,00	1,13	27,30	8,10
Sobieski	73,85	15,16	2,38	0,47	0,50	0,42	21,43	30,69

The vitrinite content in the tested coals is above 70%, while the liptinite contents are rather low – about 2% for the two samples. Vitrinite reflectance R_r tests reveal that the test samples differ in their degree of metamorphism. The plot given in (Sablík & Róg, 1995) gives the fixed carbon content C. For the “Sobieski” coal the fixed carbon content is below 80%, in the case of coal from “Zofiówka” it is approx. 87%. The coal sample “S” has a high ash content, over 30%. The results of sorption tests are expressed as dry ash-free (daf).

Collected coal samples were hand-crushed and sieved on a vibratory sieve shaker. Particle sizes from 0.25 mm to 0.20 mm were separated. Coal samples for testing weighing about 100 mg were dried at a temperature of 363K until a constant mass was obtained.

3. Sorption measurements

Tests were performed by the gravimetric method described in the studies by (Benham & Ross, 1989), with the use of a device which determines the sorption capacity in the pressure range up to 20 MPa. The advantages of the gravimetric method applied to gas sorption measurements on coal are discussed in works by (Beamish et al., 1991; Levine, 1992; Saghafi et al., 2007).

Five series of measurements were run accordingly to test the methane sorption on both kinds of coal. In each series, the temperature of the coal-methane system would be different.

Prior to each series of measurements, samples were dried and outgassed at 353K in a vacuum for 24 h. When the sample was outgassed and the temperature established, the sorption would be triggered by admission of methane under the pressure of 0.1MPa to the sorption chamber.

The measurement procedure was as follows:

- Outgassing of the coal sample using a turbo-molecular pump ($T_{\text{out}} = 353\text{K}$, $p_{\text{out}} = 1 \times 10^{-6}$ mbar, $t = 24$ h),
- Establishing the temperature of measurement (T_1),
- Sorption measurements at $p = 0.1$ MPa, T_1 ,
- Sorption measurements at $p = 0.5$ MPa, T_1 ,
- Sorption measurements at $p = 0.9$ MPa, T_1 ,
- Sorption measurements at $p = 1.3$ MPa, T_1 ,
- Sorption measurements at $p = 1.7$ MPa, T_1 .

After making a series of measurements at a given temperature followed outgassing of the sample (point a) and the next series of measurements in temperature, increased by 10K.

Measurements were carried out at the following temperatures:

- from 291K to 331K for coal from “Pniówek” colliery,
- from 293K to 333K for coal “Sobieski” colliery.

The gradient of pressure changes at each control point was 200 mbar/min. Additionally, methane desorption measurements were taken on coal from the “Pniówek” colliery at all temperatures.

4. Results

4.1. Sorption measurements

The plots of methane sorption on coal (expressed in terms of 1 g_{daf}) are shown in Fig. 1. Further branches of the plot represent different temperatures. On account of low diffusivity of coal from the “Pniówek” colliery (diffusion coefficient of the order of $10^{-9} \text{ cm}^2/\text{s}$), the measurements proved to be time-consuming. All of the sorption and desorption measurements on coal “Pniówek” required 4 months’ continuous operation of the gravimetric device. Diffusivity of

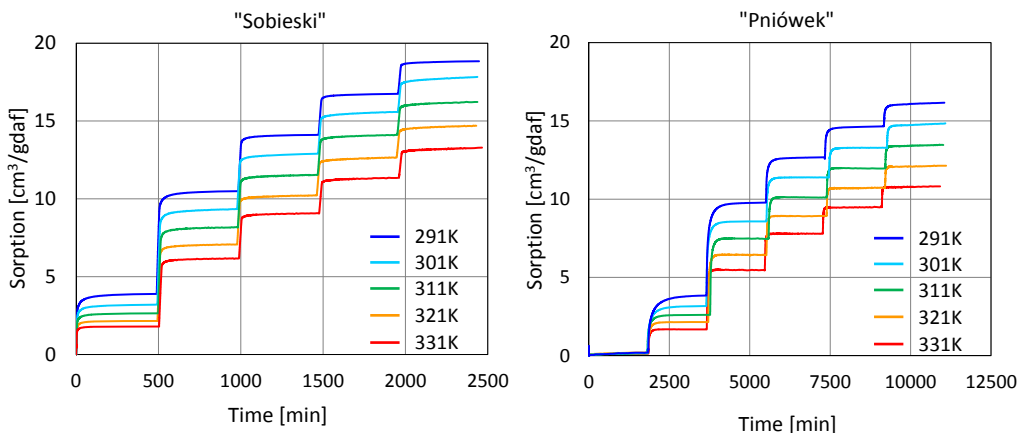


Fig. 1. Methane sorption as a function of time at various temperatures of the coal-methane system

coal from the colliery “Sobieski” was decidedly higher and the measurement procedure was shorter (about 30 days).

Measurement data allow for the determination of sorption isotherms, shown in Fig. 2. The least difference method was employed to fit the measurement points (Fig. 2) with the Langmuir isotherm in the following form:

$$a = a_m bp / (1 + bp) \quad (1)$$

where

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

Individual branches of diagrams (dotted line) are Langmuir isotherm equations. The coefficients from Eq (1) are summarised table 1. The last column gives the values of the determination coefficient R-squared. These values of R-squared are no less than 0.997.

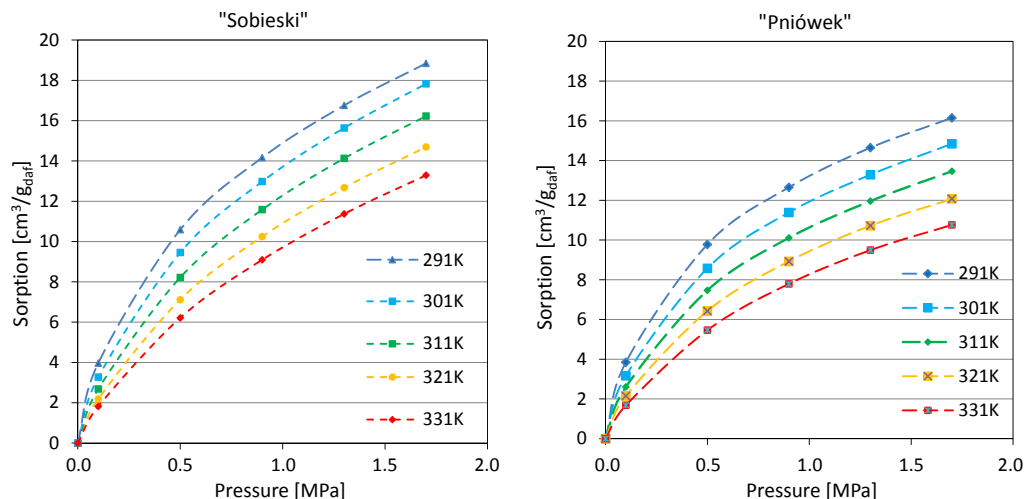


Fig. 2 Methane sorption isotherms for different temperatures

The coefficient b in the Langmuir equation (1) is the inverse of the equilibrium pressure, under which the sorption saturation level becomes $0.5a_m$. The relationship between the pressure corresponding to the half surface coverage in the function of temperature is shown in Fig. 4. Higher pressure levels are registered for the coal from “Sobieski” colliery. The relationship $b^{-1}(T)$ is rather strong and appears to be exponential. At a temperature of 300K the 50% coverage is achieved at about 0.63 MPa for the coal “P” and 0.83 MPa for the coal ”Sobieski”. At 330K, this level of coverage is achieved at 1.09 MPa and 1.48 MPa, respectively.

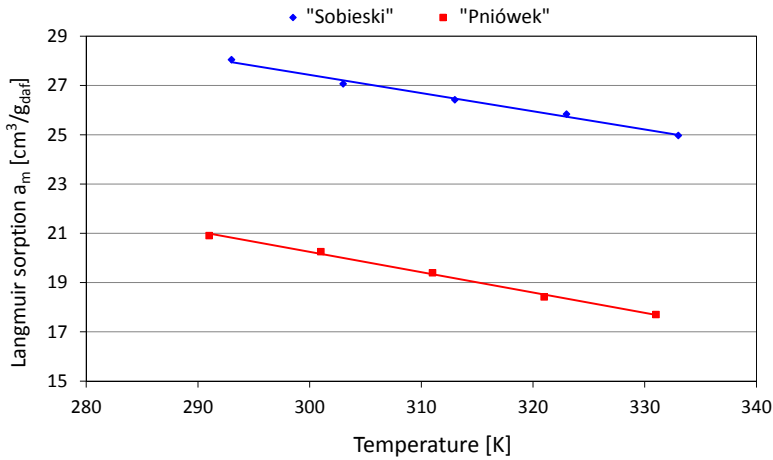


Fig. 3. Langmuir sorption capacity of investigated coals in the function of temperature

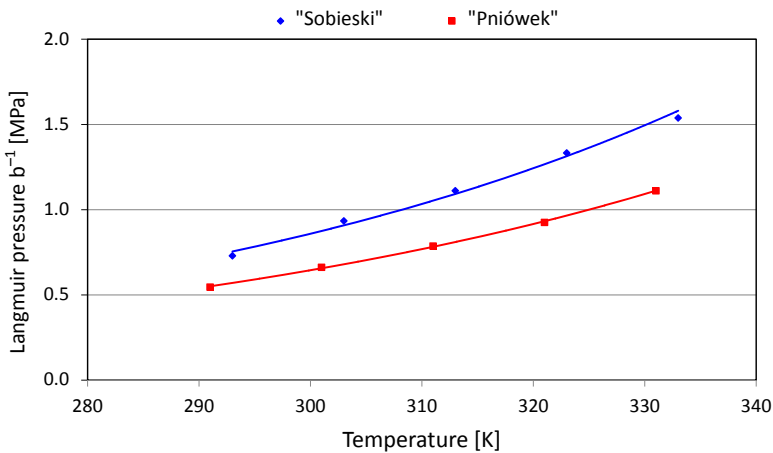


Fig. 4. Variability of Langmuir half-time sorption pressure as a function of temperature

Sorption isobars obtained for the investigated coals are shown in Fig. 5. In the considered temperature range, the methane sorption tends to vary linearly as a function of temperature:

$$a(T) = -mT + n \quad (2)$$

The values of the slope coefficients m and n of the straight lines fitted to relevant isobars are compiled in Table 2. The quality of the fit is evidenced by high values of the determination coefficient R -squared, summarised in Table 2.

The values of the slope coefficients m and n from Eq. (2) as a function of equilibrium pressure in the range from 0.1 MPa to 1.7 MPa are shown (in the semi-log scale) in Figs. 6 and 7.

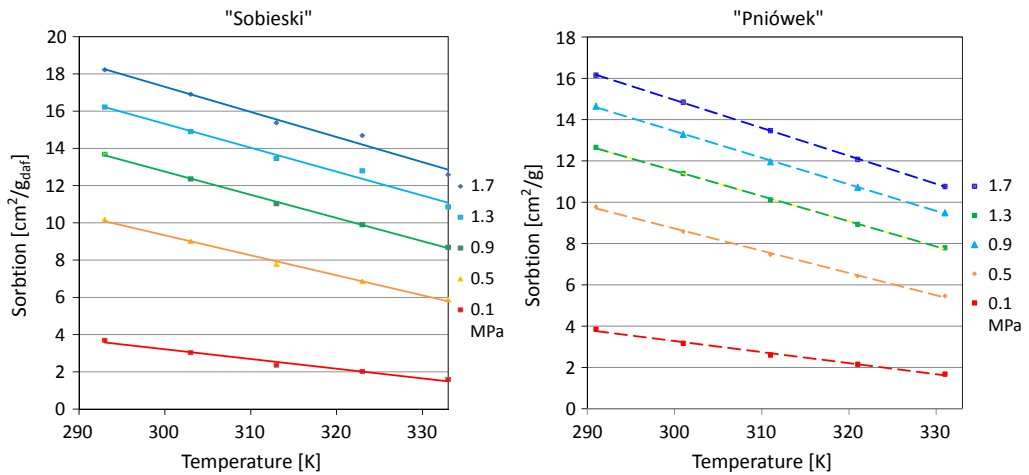


Fig. 5. Methane sorption isobars as a function of temperature

TABLE 2

Slopes of straight lines fitted to sorption isobars

Pressure	0.1 MPa	0.5 MPa	0.9 MPa	1.3 MPa	1.7 MPa
Coal from "Pniówek" colierey					
m [cm ³ /(g _{CSW} K)]	0,054	0,108	0,122	0,129	0,136
n [cm ³ /g _{CSW}]	19,331	41,082	48,066	52,077	55,59
R^2	0,993	0,998	0,999	0,999	0,999
Coal from "Sobieski" colierey					
m [cm ³ /(g _{CSW} K)]	0,049	0,108	0,130	0,143	0,152
n [cm ³ /g _{CSW}]	17,916	42,173	52,274	58,76	63,69
R^2	0,990	0,995	0,998	0,998	0,999

Variability of the coefficients " m " and " n " is governed by logarithmic equations written as:

$$m_s = 0.0365 \ln(p) + 0.133 \quad (R^2 = 0.999),$$

$$n_s = 16.071 \ln(p) + 54.381 \quad (R^2 = 0.998),$$

$$m_p = 0.0291 \ln(p) + 0.123 \quad (R^2 = 0.999),$$

$$n_p = 12.777 \ln(p) + 49.127 \quad (R^2 = 0.999).$$

These variations of the methane sorption capacity on coal are absolute changes. Relative changes of sorption capacity " s " in relation to the reference capacity level – i.e. the sorption measured at 291K, $a_p(p, 291K)$ for the coal sample from "Pniówek" and at 293K $a_s(p, 293K)$ for the coal sample "Sobieski" are shown in Fig. 10 and 11. The branches of the plots in Fig. 10 represent various temperatures of the coal-methane system, those in Fig. 11 represent various sorption equilibrium temperatures.

$$s_p(p, T) = \frac{a_p(p, 291K) - a_p(p, T)}{a_p(p, 291K)}$$

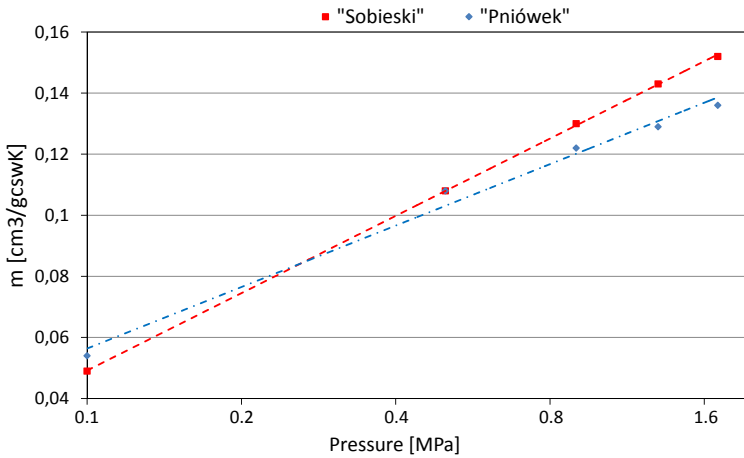


Fig. 6. Coefficient "m" vs sorption pressure

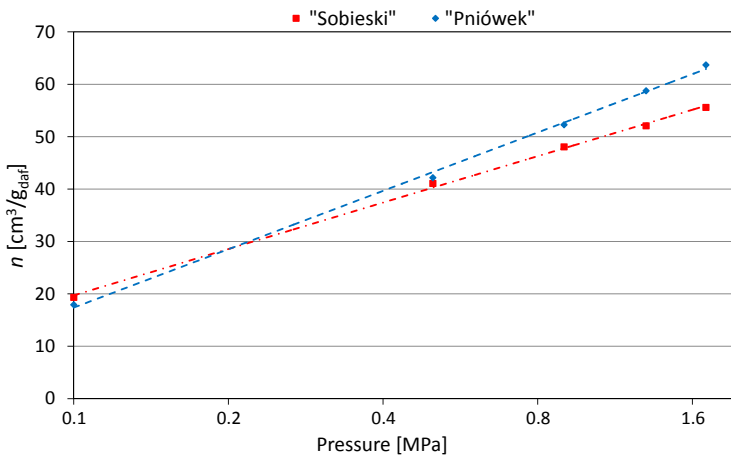


Fig. 7. Coefficient "n" vs pressure

$$s_s(p, T) = \frac{a_s(p, 293K) - a_s(p, T)}{a_s(p, 293K)}$$

Relative changes of sorption assumed to be a linear *function of temperature*, with coefficient w , depend on pressure. ($s = wT$). The plot in Fig. 12 shows the variability of slopes "w" of the straight lines shown in Fig. 8 as a function of the sorption temperature.

Experimental data show that relative changes of sorption capacity of the two coal samples are very similar and the largest changes are registered for low sorption pressures. A change in the sorption capacity due to the temperature increase by 1K is about 1.3% at a pressure of 0.1 MPa. At 1.7 MPa, it changes by about 0.85% (coal sample "Pniówek") and 0.75% (coal sample "Sobieski").

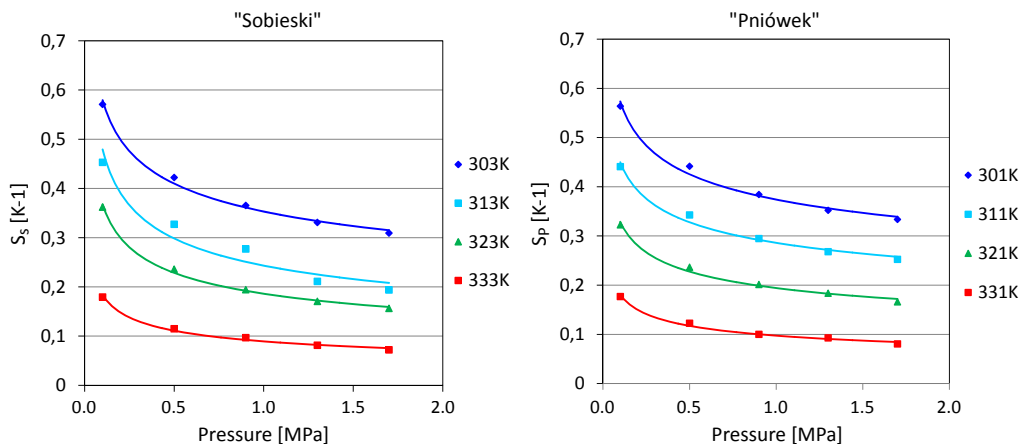


Fig. 10. Relative changes in sorption as a function of pressure

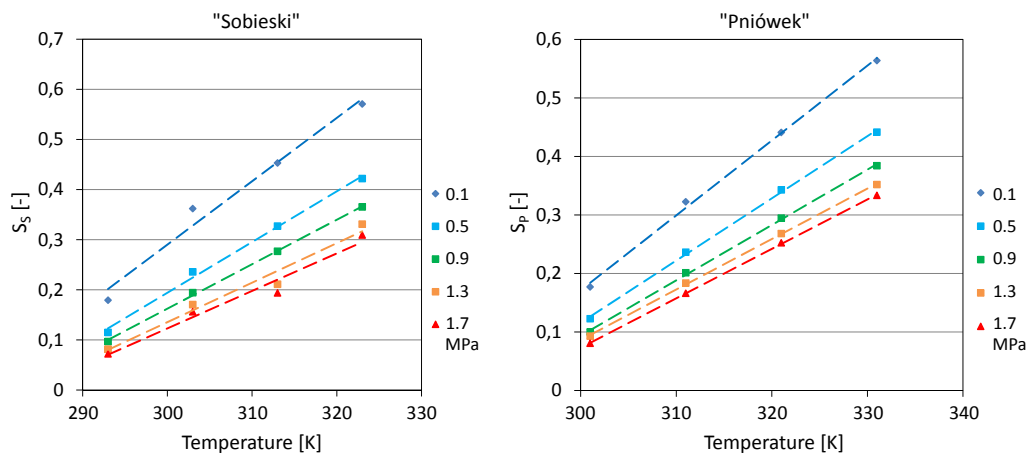


Fig. 11. Relative changes in sorption as a function of temperature

4.2. Desorption measurements

Experiments were run on the coal sample "Pniówek" to test methane desorption from the coal. Measurements were taken for all sorption points, at all temperatures in the range from 291 to 331K. Isotherms of sorption (continuous lines) and desorption (broken lines) of methane on coal from the colliery "Pniówek" are shown in Fig. 13. All desorption isotherms are above the sorption isotherms, which indicates that the investigated coal exhibits sorption hysteresis.

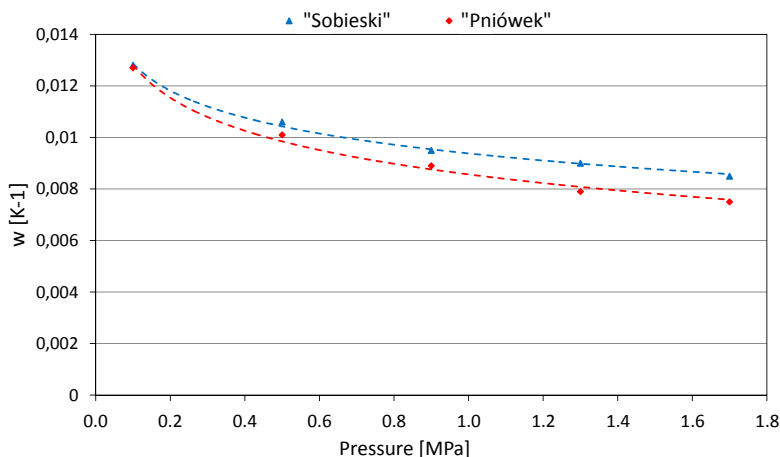


Fig. 12. Slopes of the straight lines “w” vs. equilibrium pressure

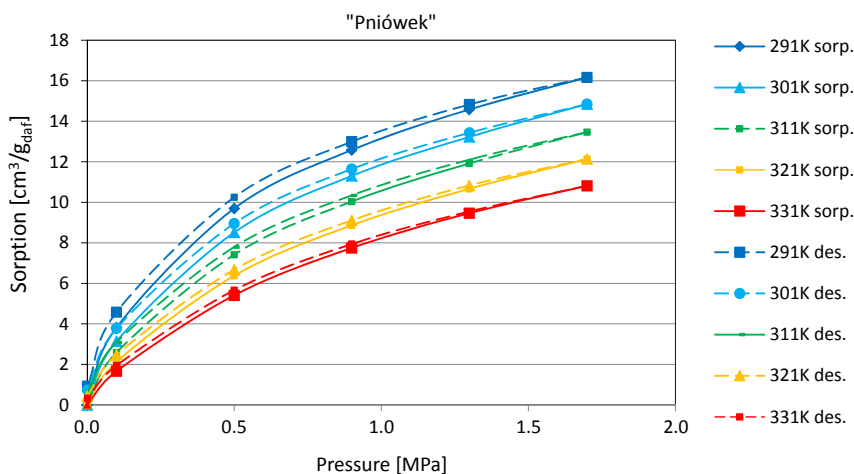


Fig. 13. Isotherms of sorption and desorption of methane on coal “Pniówek”

Fig. 14 presents relative hysteresis of sorption derived for particular sorption/desorption pressures from the equation:

$$h(p, T) = \frac{a_{des}(p, T) - a_{sorp}(p, T)}{a_{sorp}(p, T)}$$

where:

$a_{sorp}(p, T)$ — the amount of sorbed methane at the pressure p and temperature T , measured in the process of sorption,

$a_{des}(p, T)$ — the amount of sorbed methane at the pressure p and temperature T , measured in the process of desorption.

Individual branches of the plot indicate various sorption and desorption temperatures. It appears (see Fig. 14) that the most considerable relative differences between the sorption and desorption values are registered at low pressures: from 16% to 20% at 0.1 MPa. At pressures above 0.5 MPa the magnitude of the hysteresis should not exceed 5%. The band of curves representing different temperatures is relatively narrow, indicating a weak relationship between the magnitude of the hysteresis and the temperature of the coal-gas system. An analysis of particular branches of this plot reveals that an increase in the temperature leads to reduction of magnitude of the sorption/desorption hysteresis.

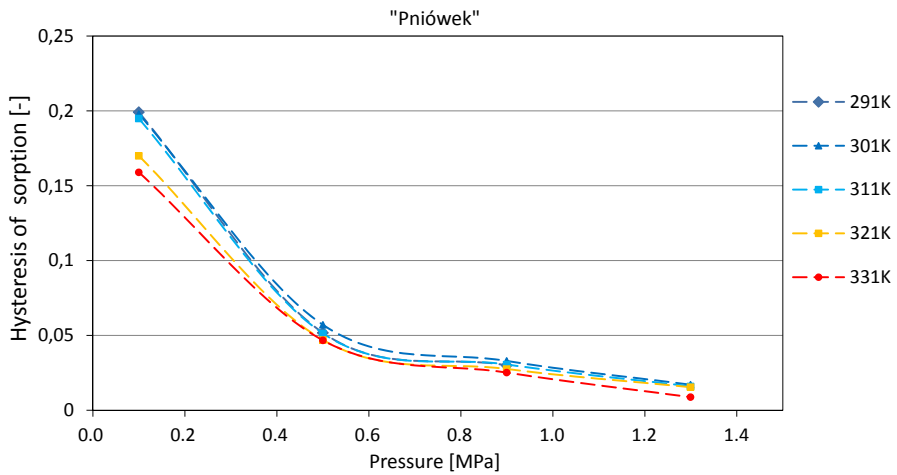


Fig. 14. Magnitude of sorption hysteresis for the coal „Pniówek” vs equilibrium pressure

5. Change of equilibrium pressure of methane due to the increase of mining depth

Let us consider how the equilibrium pressure of coalbed methane would change with an increase of the mining depth? Let us assume that the coal with the properties of the coal sample “Pniówek”, with the known methane content M_c (in $\text{m}^3/\text{Mg}_{\text{daf}}$) is situated at various depths. With an increase in the mining depth, the temperature of the coal-gas system under natural conditions will change due to the geothermal gradient. It is assumed that the average geothermal gradient in the Upper Silesia region is $3\text{K}/100\text{ m}$ (Kędzior, 2009). If we know the porosity of coal and the relationship between the pressure, temperature and the amount of sorbed methane, the equilibrium pressure of coalbed methane can be determined. The coalbed pressure p_c depends on the depth (because of the occurrence of geothermal gradient) and the methane content M_c .

Let us assume that the methane content per unit mass of coal (M_c) is a sum of absorbed methane (a_a) and free gas (a_f). These amounts depend, *inter alia*, on the pressure and temperature of the coal-gas system. Accordingly, we obtain:

$$a_s(p, T) = a_m(T) \frac{b(T)p}{1 + b(T)p}, \quad a_f = \frac{\varepsilon}{\rho_{He}} \frac{P}{P_0} \cdot \frac{298}{T}, \quad M_c(p, T) = a_m + a_f$$

The plot of equilibrium pressure of methane in the function of methane capacity for the coal sample “Pniówek” from the colliery “Pniówek” is shown in Fig. 15. Further branches of the plot correspond to depth isolines in the range from 600 m to 1400 m. For a methane capacity of $16 \text{ m}^3/\text{Mg}_{\text{daf}}$, the equilibrium pressure at a depth of 1400 m can be twice as high as at 600 m. Such a large change in pressure can be caused by the geothermal gradient only.

Experimental data reveal that the equilibrium pressure of coalbed methane can rapidly increase with increased methane bearing capacity and depth. Gas pressure is one of the most important parameters affecting the possibility of a gas and rock (coal and gas) outbursts in mines (Skoczylas, 2002; Młynarczyk & Wierzbicki, 2013). It can be assumed that the threat of methane outbursts and rock will increase with the mining depth.

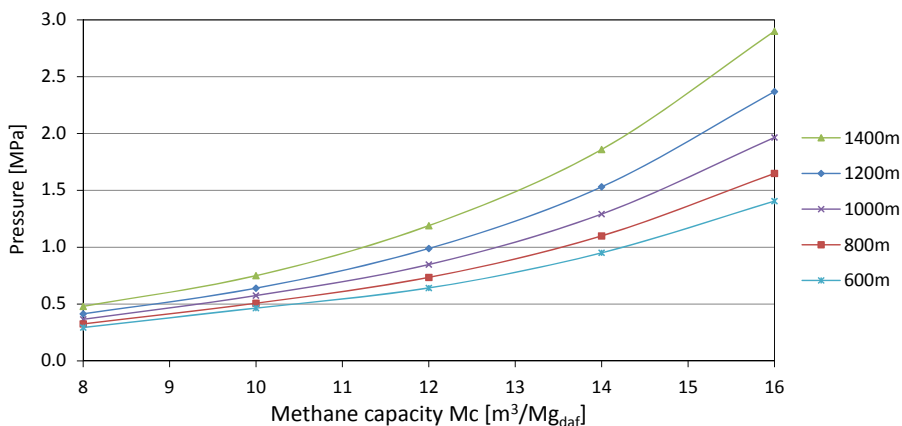


Fig. 15. Variability of methane equilibrium pressure as a function of methane bearing capacity for variable coalbed depth

6. Conclusions

It appears that there is no straightforward relationship between the coal rank and its sorption properties. Higher-rank coal from the colliery “Pniówek” ($R_r = 1.08\%$) will sorb less methane than coal from a non-gassy mine “Sobieski” ($R_r = 0.47$).

An increase in the temperature of the coal-gas system causes a decrease in the sorption capacity. The maximum Langmuir sorption capacity changes linearly with temperature. The reduction of the sorption capacity is $0.083 \text{ cm}^3/(\text{g}_{\text{daf}}\text{K})$ for the coal sample “Pniówek” and $0.074 \text{ cm}^3/(\text{g}_{\text{daf}}\text{K})$ for the sample “Sobieski”. The isobars of sorption at different temperatures are linear.

The most significant changes of sorption capacity due to temperature variations are registered at low sorption equilibrium pressures.

An increase in the temperature of the coal-gas system gives rise to an increase in the partial pressure of Langmuir sorption and this change follows a roughly exponential pattern.

Relative values of sorption/desorption hysteresis tend to decrease with an increase in the coal temperature. At pressures above 0.5 MPa, they are rather small (below 5%).

The occurrence of a geothermal gradient can give rise to a major increase in the equilibrium pressure of methane in coalbeds.

Calculation data suggest that for a methane bearing capacity of $16 \text{ m}^3/\text{Mg}_{\text{daf}}$, the equilibrium pressure of methane at a depth of 1400 m can be twice as high as at 600 m, which is closely associated with the level of potential hazard of coal and gas outburst occurrence in collieries.

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