Zeszyt 4

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The relation between ambient temperature and sorption of carbon monoxide on bituminous coals

Introduction

In the Polish hard coal mining both within coal seams and in mine atmosphere several gases are present, among others hydrogen methane, carbon dioxide, unsaturated and saturated hydrocarbons and highly toxic carbon monoxide (Wacławik et al. 2000).

Carbon monoxide is invariantly present at every process linked to spontaneous heating and subsequent endogenous fires (Cygankiewicz 2003). Its volume concentration in mine air can vary from values as low as 0.0001% up to 1% even. High toxicity of carbon monoxide is the reason why a thorough examination is needed of the sorption phenomena occurring on the hard coal outer surface interacting with carbon monoxide and the determination of the accessibility of the inner porous structure of coal for this sorbate at the range of temperatures similar to those encountered in an underground mine. Also, it needs to be determined whether this gas could be adsorbed at the surface structure of coal grains.

A molecule of carbon monoxide has a free pair of electrons and in consequence it is an donor of electrons, forming coordinated bonds in several complex compounds. Also, because of their asymmetric charge distribution, CO molecules interact with certain active sites on the surface of bituminous coal grains. Carbon monoxide is a weak dipole, its dipole moment is 0.3·10⁻³⁰ C·m (Bielański 2002).

Concentration of carbon monoxide in air ventilating a bituminous coal mine can be frequently well above the normal level, in some cases, especially during spontaneous heating

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processes, its concentration is above the level required by mining laws. Constant monitoring of CO level in air is required for he safe run of a coal mine. According to the Polish mining law code (Mine Safety Code, MG 2002) not more than 26 ppm vol. level of CO concentration in ventilation air is permitted.

It is known that CO can be emitted into mine atmosphere from coal undergoing oxidation, but the origin of that emission is not entirely clear (Wacławik et al.1997).

This phenomenon was discussed thoroughly by Wang (Wang 2002). In the tests he had carried out samples of crushed coal were oxidized in the stream of air with 19% of oxygen content during 18 hours period, then the samples were flushed by stream of pure nitrogen. Concentration of gases emitted during this tests (CO and CO_2) was determined chromatographically. As a result of coal oxidation on the surfaces of open pores transient oxygenated complexes were formed – the closed pores did not participate in the oxidation. Thermal decomposition of these complexes is the source of the CO and CO_2 molecules formation. Then the diffusion of oxides in pores takes place and consequently the CO and CO_2 molecules are removed outside coal particles by stream of flowing gas (nitrogen).

According to (Wang 2002) it is likely that only some part of the active sites in complexes is responsible for oxides emission during thermal decomposition.

The difference between the values of activation energies of formation of carbon monoxide and dioxide suggests that separate reactions lead to formation of the two gases. Carbon monoxide is created as a result of thermal decomposition of complexes having carbonyl groups – the weak bonds of carbonyl groups with the structure of coal are broken. On the other hand the complexes having carboxyl groups are responsible for carbon dioxide formation. The formation rate of oxides is determined by concentration of the available complexes in the coal matter. According to the author (Wang 2002) the process of thermal decomposition of oxygenated complexes is somewhat close to the process of chemical desorption.

There is also possibility that at the initial phase of the test emission of oxides into gas stream originates from CO and CO_2 adsorbed physically on the surface of coal grains and on its pores (Wang 2002)

The quantity of carbon monoxide emitted during the initial phase of spontaneous heating (up to 333 K) is not significant, which can be attributed to the thermal effect of the reaction of CO formation, whose activation energy is 72.0 ± 5.8 kJ/mol.

An important role in the oxides emission plays the temperature increase, causing higher rate of CO and CO₂ formation. The temperature increase from 333 K do 383 K brings about 50 times higher rate of CO formation, whereas CO_2 formation increases about 10 times only. It can be stated that at higher temperatures the rate of decomposition is higher (Wang 2002), thus increasing oxides emission.

In the case of some samples of coal a vague correlation was observed between grains size of coal and oxides formation. During the initial stage of the tests at 383 K the rate of formation of carbon monoxide is higher for coarser coal particles sizes. Also in the papers by other authors (Clemens et al. 1991) it was demonstrated that the emission of CO into atmosphere can be attributed to thermal breakdown of carbonyl groups. The volume of CO released was rater low (which can be linked to small thermal effect of the CO formation process) and especially during the first stages of spontaneous heating.

Apart from the oxidation processes, carbon monoxide can be also emitted spontaneously into ambient atmosphere as a result of prior adsorption of carbon monoxide on coal matter. During drilling of boreholes in coal seams emission of some quantities of CO is observed, leading to the increase of concentration of CO in gaseous samples taken from such boreholes up to 1% vol (Wacławik et al. 2000).

Bituminous coal can be treated as a complex organic polymer of well developed system of pores, namely: sub-micro, micro, meso and macropores (Cui et al. 2004). In several studies it was demonstrated that micro- and sub-micropores are located mainly in the aromatic polymer structure and they form the fundamental adsorptive part of bituminous coal (Milewska-Duda 1987; Clarkson and Bustin 1999). The volume of micropores and sub-micropores is the main parameter influencing the sorption of gases having small, polar molecules.

Macropores are located in the vicinity of the perimeter area of the polymer where they are created as a result of combining of acyclic and aliphatic hydrocarbons (Czapliński 1994). Meso and micropores do not play a significant role in gas storage processes, but they are involved in the phenomena of diffusion and transport of gases penetrating into micro- and submicropores.

Beside the structure of the system of pores within coal matter also the chemical composition of coal surface is the factor shaping sorption properties of bituminous coal (Marzec 2002; Żyła 1963). The polarity of coal surface plays important role in sorption processes for such gases as carbon dioxide, water vapor, methanol and CO (Żyła 1963; Kreiner et al. 2007; Cygankiewicz, Dudzińska, Żyła 2007).

In the recent years modern measurement devices and installations have been employed for research on properties of bituminous coal, among others also for the sorption of gases and vapors on coal. The most frequently published are the results of studies on sorption of carbon dioxide, methane an nitrogen (Mastalerz et al. 2004; Ceglarska-Stefańska and Zarębska 2005; Jodłowski et al. 2007; Ceglarska-Stefańska et al. 2007). Generally, bituminous coal is treated as sorbent of medium sorption affinity towards gases present in coal mines atmosphere. The most of sorption though, undergoes with carbon dioxide. Small dimensions of its molecule and specific shape of its double dipole are the reason why CO_2 can easily penetrate into micro- and submicro-pores of bituminous coals (Kreiner and Żyła 2006; Zarębska and Dudzińska 2008).

This paper is devoted to the sorption of carbon monoxide on selected bituminous coals collected in Polish mines, under conditions of ambient temperature similar to that can be encountered underground. As far as it is known to us only in one paper dealing with carbon monoxide sorption on coal (Cygankiewicz, Dudzińska, Żyła 2007) it was demonstrated that the sorption of carbon monoxide is influenced by carbon and oxygen content within a given sample of coal. Also, it was stated there that the process of carbon monoxide sorption on coal

is irreversible at the given temperature level. The volume of gas remaining within coal structure depends on carbon and oxygen content in coal and, probably, on concentration of oxygenated groups on coal surface.

The problem of the correlation between ambient temperature and sorption activity of different sorbates on bituminous coal was also dealt with by other authors, for example a study on relation between temperature and sorption of methane, carbon diozide and nitrogen on bituminous coal was carried out by (Sakurov et al. 2008). It was demonstrated there that sorption volume of these gases decreases with rising temperature. The tests were performed at two values of ambient temperature: 308 K and 328 K. Also (Ruppel et al. 1974) in its work demonstrated that sorption capacity of methane decreases at higher temperatures – tests were performed on several samples of bituminous coal at. 273 K, 303 K, 323 K.

1. The experimental part

Six samples of coals having different metamorphism degrees were selected for the sorption tests. The samples were collected from active Polish mines, namely Jaworzno mine, seam 209, Wesoła mine, seam 501, Pniówek mine, seam 360, Zofiówka mine, seam 404, Chwałowice mine, seam 404/4, Sośnica mine, seam 413. The properties of coal samples are listed in Table 1. Samples were prepared according to the Polish standard PN-90/G-04502.

The samples of coal used for the testing were prepared by crushing and sieving in order to obtain the following grain sizes: 0.063–0.075 mm, 0.125–0.25 mm and 0.5–0.7 mm. The laboratory tests on sorption were carried out under low pressures in the range up to 0.09 MPa with application of the volumetric method in a modern, fully automated apparatus ASAP 2010 made by Micromeritics. This type of apparatus enables to determine sorption and desorption isotherms on granulated and powder-like samples for different gases, among others for carbon monoxide, at wide range of temperatures.

Before measurement the samples were degassed under vacuum of $5.06 \cdot 10^{-7}$ Pa and then by flushing them with helium several times. The measurement was preformed at four temperatures, namely 308 K, 323 K, 343 K and 373 K; that range of temperatures was selected because it corresponds to normal temperatures encountered in real locations in coal mines.

2. Discussion of the results

The isotherms of sorption of carbon monoxide obtained for all coal samples are presented in the Figures 1 to 6. The shape of the isotherms is generally smooth and the points of measurement fit well to the curves, without distinct deviations – it can be seen as an indication that sorption measurements were fairly accurate. In all cases the most distinct

Chemical and petrographic characteristics of coal samples

	Symbol	Unit	Samples from the following mines					
Contituent content			Pniówek s. 360	Wesoła s. 501	Chwałowice s. 404	Zofiówka s. 404/2	Sośnica s. 413	Jaworzno s. 209
Carbon	C _t ^a	%	84.24	79.46	79.29	78.62	70.82	57.83
Sulphur total	Sta	%	0.39	0.32	0.35	0.35	3.50	1.10
Sulphur from pyrite	S ^a p	%	0.01	0.07	0.16	0.01	3.20	0.71
Hydrogen	H_t^a	%	4.58	4.55	4.97	4.37	3.35	3.37
Nitrogen	Na	%	1.52	1.27	1.07	1.15	1.28	0.87
Sulphur from ash	S _A ^a	%	0.07	0.22	0.19	0.18	1.27	0.03
Sulphur combustible	Sca	%	0.32	0.10	0.16	0.17	2.23	1.07
Oxygen (calculated)	Oda	%	4.58	8.07	8.47	6.17	6.29	11.30
Moisture	Wa	%	1.75	3.69	3.39	0.60	1.85	11.11
Ash	Aa	%	3.01	2.86	2.65	8.92	14.18	14.45
Volatile matter	Va	%	27.12	30.40	35.62	27.93	29.88	28.39
Vitynite	Vt	% vol.	73	38	60	91	60	67
Reflexivity of vitynite	R _o	%	0.92	0.72	0.70	1.01	0.78	0.51
Liptinite (exinite)	L	% vol.	7	9	10	1	9	5
Inertynite	Ι	% vol.	20	53	30	8	31	28
Mineral substance	М	% vol.	1	2	1	4	14	11

Charakterystyka chemiczna i petrograficzna

increase of the volume of adsorbed gas was observed at the initial stages of the sorption process, then, with the increasing pressure volume increase was usually lower.

Also, for all measured samples it was observed that the volumes of carbon monoxide adsorbed are diminishing with rising sorption temperature. The highest volume of CO was sorbed on bituminous coal at 308 K. Further, with rising temperatures gradual decrease in volume of CO sorbed was observed. At 323 K coal sorbed about 80% of the volume measured at 308 K. The measurement at next temperatures followed that pattern, namely at 343 K it was 60-70% and at 373 K it was almost half of the volume measured at 308 K.

The above described trends can be clearly seen in the Figures 7–12 containing graphs of volumes of CO sorbed versus sorption temperature for three grain sizes.

The data obtained during the tests confirmed that for all tested coals there was distinct decrease in sorption capacity with increasing temperatures. Also it was noted that CO

TABLE 1

TABELA 1



Fig. 1. Isotherms of CO sorption on coal samples from Sośnica coal mine (grain size class 0.063–0.075 mm)





Fig. 2. Isotherms of CO sorption on coal samples from Wesoła coal mine (grain size class 0.063–0.075 mm)





Fig. 3. Isotherms of CO sorption on coal samples from Pniówek coal mine (grain size class 0.063–0.075 mm)





Fig. 4. Isotherms of CO sorption on coal samples from Jaworzno coal mine (grain size class 0.063–0.075 mm)





Fig. 5. Isotherms of CO sorption on coal samples from Chwałowice coal mine (grain size class 0.063–0.075 mm)





Fig. 6. Isotherms of CO sorption on coal samples from Zofiówka coal mine (grain size class 0.063–0.075 mm)





Fig. 7. Relation between volume of CO adsorbed and temperature of sorption for sample of coal from Zofiówka coal mine





Fig. 8. Relation between volume of CO adsorbed and temperature of sorption for sample of coal from Chwałowice coal mine





Fig. 9. Relation between volume of CO adsorbed and temperature of sorption for sample of coal from Wesoła coal mine





Fig. 10. Relation between volume of CO adsorbed and temperature of sorption for sample of coal from Jaworzno coal mine





Fig. 11. Relation between volume of CO adsorbed and temperature of sorption for sample of coal from Pniówek coal mine

Rys. 11. Zależność objętości zaadsorbowanego tlenku węgla od temperatury sorpcji dla węgla z kopalni Pniówek



Fig. 12. Relation between volume of CO adsorbed and temperature of sorption for sample of coal from Sośnica coal mine



sorption decreased with increasing grain size of a sample. The largest volume of CO was adsorbed by coals of 0.063–0.075 mm class at temperature of 308 K, the least prone to sorption were coal samples having the coarsest grains and tested at 373 K. The influence of coal particles size on sorption ability of coal samples suggests that sorption takes place mostly on outer surfaces of particles and is much less present in the sub- and micro-pores structure. The only exception to this rule were the samples of coal collected in Jaworzno coal mine, where the correlation between particles size of coal samples and their sorption capacity was almost non existent. This deviation suggests that, probably, the internal porous structure of the coal from Jaworzno mine was fully accessible to the adsorbate used during tests.

The results of the investigation presented above suggest that there can be potential hazard arising from carbon monoxide adsorption on coal dust or other fine-grain fractions of coal produced during extraction, drilling and haulage, because the finest coal grades have the greatest sorption ability towards carbon monoxide. Coal dust mixed with air with concentration of $50-1000 \text{ g/m}^3$ is explosive and the rate of advancement of an explosion wave can reach above 7 km/s (Cybulski 2005). Gases produced during dust explosion can contain more then dozen percent of CO vol. Explosion of dust in a mine is possible when coal dust accumulated on the floor or ribs of an underground working is lifted into the air after blast, burst, methane explosion or explosion of fire fumes.

The coal dust that is able to explode has at least 10% volatile matter (ash free). The volatile matter denotes the mass of gases emitted during rapid heating of coal. Typical gas concentration pattern of gases emitted during such heating is as follows: $H_2 = 18\%$, CO = 73%, $CO_2 = 3\%$, $CH_4 = 4.5\%$ (Cybulski 1973).

Carbon monoxide adsorbed in favorable physical and chemical conditions and accumulated within coal structure can be desorbed when temperature gets higher. Due to this phenomenon it can be stated that carbon monoxide can be released into ambient atmosphere from two sources, namely from the foci of spontaneous combustion or fires and also from desorption from within coal matter.

From the relationships presented in Figures 7–12 the volumes of desorbed CO in the temperature ranges 308–323 K, 308–343 K, 308–373 K can be determined, as well as the volumes of CO remaining within coal structure. The highest differences between volumes of CO sorbed at temperatures 308 and 373 K were noted in the case of the samples having grain size of 0.063–0.075 mm; these samples also had the largest volume of CO that remained within coal matter.

The smallest slopes of lines joining the points corresponding the volumes of carbon monoxide sorbed as a function of temperature was for the most coarsely ground coal samples. The slopes rise for finer samples. The most significant differences among the slopes were noted between a coal sample having grain size 0.063-0.075 mm and the sample of 0.125-0.25 mm. There was only slight difference between the slopes of the 0.125-0.25 mm size and the 0.5-0.7 mm size.

The conduct of sorption process and the volume of sorbed gas depend on bituminous coal structure, its chemical properties, carbon, ash and moisture content, composition of macerals

and other factors (Crosdale et al. 1998; Laxminarayana and Crosdale 1999; Karacan and Okadan 2000).

In Figure 13 sorption isotherms are presented of all coals tested at 308 K. It can be seen that the most of CO is sorbed on coal from Jaworzno mine, having the lowest coalification grade – carbon content was – 57.83 % C (see Table 1). High oxygen concentration (11.30%) indicate that there is possibility that high number of oxygen groups is present in the structure, thus causing high polarity of the surface. It is also possible that there is interaction of CO with active centers on the surface of coal particles that favors increased CO sorption. Additionally, the full availability of micro and sub-micro pores of coal also is favorable in terms of higher CO sorption (Cygankiewicz, Dudzińska, Żyła 2007).



Fig. 13. Isotherms of CO sorption at 308 K on coal sample of grain size class 0.063-0.075 mm

Rys. 13. Izotermy sorpcji CO wyznaczone w temp. 308 K na węglach kamiennych dla klasy ziarnowej 0,063–0,075 mm

The coal sample from Wesoła mine adsorbs less CO, while this coal has much higher carbon content (79.46% C) and its oxygen content is lower (8.07%) than the sample from Jaworzno mine. The least active, with regard to CO sorption, are samples of coal from Sośnica and Zofiówka that have low oxygen content (6.29%; 6.17% respectively). Carbon and oxygen contents of all samples are listed in Table 1.

Judging from the shape of sorption isotherms it can be stated that volume of CO sorbed depends on carbon and oxygen content in the coals tested. This relation can be seen in Figures 14 and 15. The graphs from Figure 14 represent relationship between volume of carbon monoxide adsorbed and carbon content of a sample. It can be clearly seen that the volume of CO sorbed is diminishing for increasing carbon content. This relationship is generally



Fig. 14. Relation between volume of CO adsorbed at 308 K and carbon content in coal samples

Rys. 14. Objętość zaadsorbowanego CO na węglach kamiennych wyznaczona w temp. 308 K w zależności od procentowej zawartości pierwiastka C



Fig. 15. Relation between volume of CO adsorbed at 308 K and oxygen content in coal samples Rys. 15. Objętość zaadsorbowanego CO na węglach kamiennych wyznaczona w temp. 308 K w zależności od zawartości tlenu w węglu

linear, especially in the case of samples from coal mines Jaworzno, Pniówek, Wesoła and Chwałowice.

The samples of two coals, namely from Zofiówka and Sośnica mines do not conform to this linear pattern. This discrepancy can b attributed to the fact that mineral substances making up the ash part of coal matter can influence process of sorption on this coals.

Analogically, in Figure 15 the relationship is presented between adsorption intensity and oxygen content in coal samples. Sorption of carbon monoxide increases with increasing oxygen content, thus probably increasing concentration of active sites on samples. It is possible that CO molecules that are slightly polarized interact with active sites on surface of coal particles.

The linear correlation between oxygen content in coal matter and polar active sites centers in coal was demonstrated in the papers (Żyła 1963; Stachurski and Żyła 1995). The authors tested sorption of water vapor and methanol and found that the quantity of polar centers of sorption determined from water vapor sorption isotherms is proportional to oxygen content in a coal sample, and inversely proportional to its carbon content. Similar relationship takes place in the case of carbon monoxide sorption on bituminous coal. These observation suggest that also in the CO sorption process a major role is played by polar exogenous sorption centers (active sites).

Conclusions

The results tests on CO sorption on six samples of coal at four temperatures: 308 K, 323 K, 343 K and 373 K indicate that the higher is temperature of sorption the lower is sorption volume. The volume of CO sorbed at 373 K is almost a half of the volume sorbed at 308 K.

The results of sorption tests that were carried out on samples of three grain sizes: 0.063–0.075 mm, 0.125–0.25 mm, 0.5–0.7 mm indicate that volume of CO sorption is greater for finer samples (with the exception of coal from Jaworzno mine). Probably majority of CO is adsorbed on outer surfaces of coal grains and only small portion of CO is sorbed in submicro- and micropores of coal.

The volume of CO sorbed depends on carbon and oxygen content within coal matter.

Increase of temperature of coal brings about desorption of CO, causing increase in concentration of CO in the ambient atmosphere.

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THE RELATION BETWEEN AMBIENT TEMPERATURE AND SORPTION OF CARBON MONOXIDE ON BITUMINOUS COALS

Key words

Hard coal, sorption, carbon monoxide

Abstract

In this paper the results have been discussed of a study on CO sorption on six samples of bituminous coal collected in Polish mines, having different metamorphism degree. Tests of sorption of CO were carried out at different temperatures, including ones typical for real mine environment, 308K, 323 K, 343 K and 373 K. The study demonstrated that sorption of CO decreases with increasing ambient temperature – the intensity of sorption of CO at 373 K is nearly half of the sorption at 308 K. It is likely that increase of ambient temperature in mines causes desorption of CO from coal seams, thus increasing its concentration in the ventilating air. Desorption of CO is another of such phenomena as spontaneous coal heating or fires, resulting in emission of CO into ventilation air thus posing a threat for miners. The sorption tests were carried out on samples of coal crushed into the following grain size classes: 0.063–0.075 mm, 0.125–25 mm, 0.5–0.7 mm. It emerged that sorption of CO on coal samples increases with coal grains disintegration, suggesting that the sorption of CO occurs mainly on the outer surfaces of coal grains. The exception of that pattern was noted in the case of coal from Jaworzno mine, having high carbon and low oxygen content. It was also demonstrated that the volume of CO adsorbed on a coal sample is related to its carbon and oxygen contents.

WPŁYW TEMPERATURY NA SORPCJĘ TLENKU WĘGLA NA POLSKICH WĘGLACH KAMIENNYCH

Słowa kluczowe

Węgiel kamienny, sorpcja, tlenek węgla

Streszczenie

W pracy przedstawiono wyniki badań sorpcji tlenku węgla na sześciu próbach polskich węgli kamiennych o zróżnicowanym stopniu metamorfizmu. Sorpcję tlenku węgla przeprowadzono w temperaturach zbliżonych do warunków temperaturowych panujących w kopalni: 308K, 323 K, 343 K i 373 K. W wyniku wykonanych badań stwierdzono spadek sorpcji tlenku węgla zachodzący wraz ze wzrostem temperatury. Sorpcja CO w temperaturze 373 K jest niemal o połowę mniejsza w porównaniu z sorpcją w temperaturze 308 K. Prawdopodobnie w kopalniach każdorazowy wzrost temperatury w pokładach węgla powoduje desorpcję tlenku węgla, a tym samym zwiększenie jego stężenia w powietrzu kopalnianym. Oprócz samozagrzewania i pożarów termiczna desorpcja CO jest kolejnym procesem przyczyniającym się do wzrostu toksycznego tlenku węgla w atmosferze kopalni, podnosząc poziom niebezpieczeństwa dla pracujących załóg górniczych. Badania sorpcyjne przeprowadzone na trzech klasach ziarnowych: 0,063–0,075 mm, 0,125–25 mm, 0,5–0,7 mm wykazały wyraźny wpływ rozdrobnienia węgla na sorpcję CO. Sorpcja tlenku węgla wzrasta wraz z rozdrobnieniem węgla, co przemawia za sorpcją tego gazu zachodzącą w dużej części na zewnętrznych powierzchniach ziarna węglowego. Wyjątek stanowi węgiel z kopalni Jaworzno o niskiej zawartości pierwiastka węgla i dużej zawartości tlenu, gdzie sorpcja tlenku węgla zależą od zawartości pierwiastka węgla i tlenu w strukturze węgli kamiennych.