Environ Monit Assess (2016) 188: 416
DOI 10.1007/s10661-016-5423-z

CO_2 adsorption properties of char produced from brown coal impregnated with alcohol amine solutions

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Received: 15 January 2016 / Accepted: 12 June 2016 / Published online: 17 June 2016

Abstract Carbon dioxide (CO_2) emission reduction is critical to mitigating climate change. Power plants for heating and industry are significant sources of CO_2 emissions. There is a need for identifying and developing new, efficient methods to reduce CO_2 emissions. One of the methods used is flue gas purification by CO_2 capture through adsorption. This study aimed to develop CO_2 adsorbent out of modified brown coal impregnated with solutions of first-, second-, and third-order amines. Low-temperature nitrogen adsorption isotherms and CO_2 isotherms were measured for the prepared samples. The results of experiments unexpectedly revealed that CO_2 sorption capacity decreased after impregnation. Due to lack of strait trends in CO_2 sorption capacity decrease, the results were closely analyzed to find the reason for the inconsistencies. It was revealed that different amines represent different affinities for CO_2 and that the size and structure of impregnating factor has influence on the CO_2 sorption capacity of impregnated material. The character of a support was also noticeable as well for impregnation results as for the affinity to CO_2. The influence of amine concentration used was investigated along with the comparison on how the theoretical percentage of the impregnation on the support influenced the results. The reaction mechanism of tertiary amine was taken into consideration in connection to no presence of water vapor during the experiments. Key findings were described in the work and provide a strong basis for further studies on CO_2 adsorption on amine-impregnated support.

Keywords CO_2 adsorption · Amines · Brown coal · Impregnation

Introduction

Carbon dioxide is a greenhouse gas produced through anthropogenic emission that affects Earth's climate. Currently, many environmental scientists and politicians are focused on implementing restrictions and regulations on CO_2 emissions. Carbon dioxide emission reductions are a priority of European Union environmental policy.

The Polish energy sector is mainly based on fossil fuels. To align with European Union obligations, there is a need for introducing environmentally-friendly solutions and low emission technologies. Creating higher energy efficiencies and developing renewable energy sources, biological sequestration, and emissions reduction technologies using carbon dioxide capture can reduce CO_2 emissions. Carbon dioxide from fossil fuel combustion can be removed from flue gases through pre- and post-combustion capture and oxy-fuel and chemical-looping combustion (Metz and Davidson 2005). Carbon dioxide capture aims to collect and store concentrated streams of carbon dioxide at high pressure.

There are many post-combustion capture techniques for carbon dioxide separation from industrial sources.

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Table 1 Proximate analysis of brown coal

Sample	C^a_t, %	H^a_t, %	N^a_t, %	W^a, %	A^a, %	V^{daf}, %
Polish standards	PN-G-04571:1998			PN-G-04560:1998	PN-G-04560:1998	PN-G-04516:1998
A	53.01 ± 0.38	3.66 ± 0.20	0.69 ± 0.24	10.91 ± 0.28	8.20 ± 0.30	44.86 ± 0.68

W moisture, *A* ash, *V* volatile matter, *a* analytical state, *daf* dry and ash free state

These can be divided into absorption and adsorption methods (chemical and physical), membrane separation (Global CCS Institute 2012), and cryogenic separation (Consonni et al. 2007). Advantages of adsorption methods include ready availability, flexible operating conditions, automated processing, and the production of pure final products. Furthermore, in comparison to chemical absorption, the investment and operational costs of adsorption methods (for a medium size installation) are much more competitive.

To improve adsorption methods, it is necessary to identify effective CO_2 adsorbents (Webley 2014). Impregnation of selected materials is one possibility for preparing novel carbon dioxide adsorbents. Recent studies have suggested that impregnation of coal-derived materials (anthracite (Maroto-Valer et al. 2005), mesoporous materials (Majchrzak-Kucęba and Nowak 2008; Wang et al. 2013), fly ash (Maroto-Valer et al. 2008), and palm shell-based activated carbon (Lee et al. 2013)) could produce excellent CO_2 adsorbents. Several studies have focused on impregnating agents that can be used for obtaining the best adsorption and desorption properties (Gray et al. 2004; Plaza et al. 2007, Wang et al. 2013).

In chemical absorption processes, amine solutions and ionic liquids are typically used (Yu et al. 2012). Previous work found that 30 % solutions of monoethanolamine (MEA) or methyl-diethanolamine (MDEA) produced good results, including high process effectiveness and high purity CO_2 streams obtained from flue gas. Using MEA is profitable because of its high biodegradability (Shao and Stangeland 2009), high reactivity, stability, regeneration possibilities, and low price. The disadvantages of amine use in absorption processes include corrosive reaction effects, high energy use during regeneration, necessity of large absorbent volumes, and negative effects from trace contaminants in the input flue gas (Yu et al., 2012, Deng et al. 2015). Based on the literature, amines were chosen as impregnating agents in this study. Different concentrations of MEA, diethanolamine (DEA), and triethanolamine (TEA) were selected for impregnation to compare sorption capacities of first-, second-, and third-order amines.

Experimental

Materials

Polish brown coal, denoted as A, was used in the experiments. Results from proximate analysis of the coal are presented in Table 1. The proximate analysis was performed in an accredited laboratory at the Department for Solid Fuels Quality Assessment of the Central Mining Institute (GIG) in Katowice according to Polish standards (Table 1).

For the impregnation studies, analytical grade amines from Avantor Performance Materials were used. Coal sample A was activated in a laboratory reactor designed and built at the Faculty of Energy and Fuels, AGH University of Science and Technology (Buczek 2012).

Methods

Selected coal samples were subjected to an activation process in the laboratory reactor under a CO_2 atmo-

Table 2 Impregnated sample identifiers and amine compounds and concentrations used in this study

Amine treated sample symbol	Amine compound	Amine concentration [mol/dm^3]
A2 MEA 1,5	Monoethanoloamine	1.5
A2 MEA 3	Methanolanoamine	3
A2 DEA 1,5	Diethanoloamine	1.5
A2 DEA 3	Diethanolanoamine	3
A2 TEA 1,5	Triethanolanoamine	1.5
A2 TEA 3	Triethanolanoamine	3

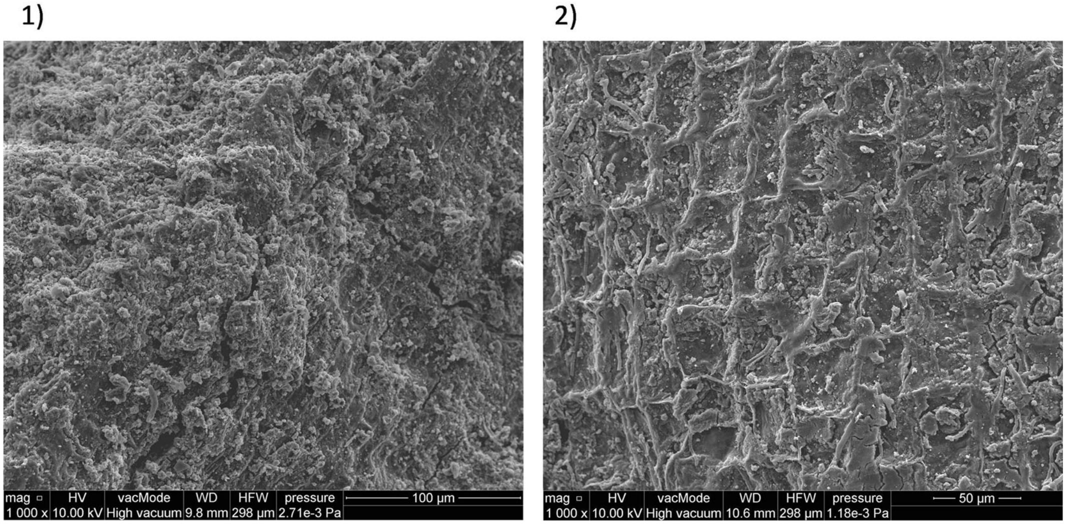


Fig. 1 SEM photographs of brown coal sample A (*1*) and activated brown coal sample A2 (*2*)

sphere. Activation was conducted for 30 min at a temperature of 1123 K. Produced activated samples were marked with symbol A2. Activated coal samples then underwent wet impregnation with amine solutions (based on procedure presented by Maroto-Valer et al. 2005). The amine compounds and concentrations used and the sample identifiers are presented in Table 2. The impregnated samples were dried at 378 K for 1 h.

Brown coal and activated material SEM photographs, made with the use of FEI QUANTA FEG 250 Scanning Electron Microscope, are presented in Fig. 1 for the purpose of comparison of the activation process influence on the structure of received materials.

A Micromeritics Accu Pyc 1330 pycnometer was used for helium density analyses of all the samples at 296 K. To determine the influence of activation and impregnation on the specific surface area, low-temperature nitrogen (N_2) adsorption isotherms for all of the samples were measured. The measurements were made at liquid nitrogen temperature (77 K) using a Micromeritics Gemini V 2380. Specific surface areas were calculated based on the linear Brunauer, Emmett, and Teller (BET) equation in the relative pressure (p/p_0) range of 0.05–0.3, and the molecular cross-section of the adsorbate molecule was 0.162 nm^2.

To determine the impact of impregnation on the adsorption of CO_2, low-pressure sorption measurements were performed using an accelerated surface area and porosimetry system (Micromeritics ASAP 2010 instrument). Measurements were carried out at 298 K at pressures up to 1 atm. Formal descriptions of the experimental sorption results were performed using the Dubinin-Radushkevich (DR) isotherm equation (Nguyen and Do 2001).

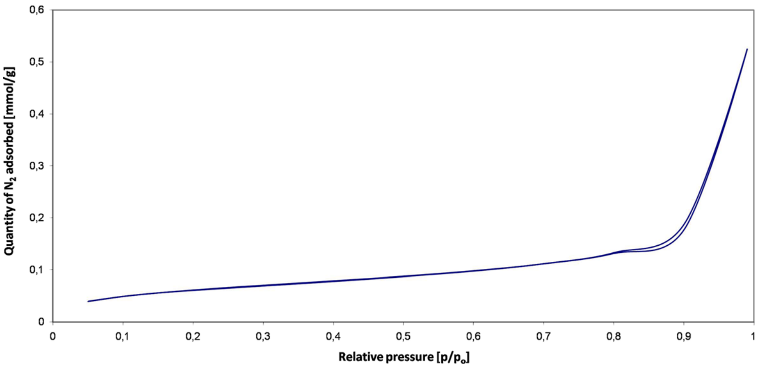
Results

Helium density analysis results are presented in Table 3. The activation process resulted in increased carbon material density. Impregnation with different amines led to density decreases, and these decreases were larger as amine concentrations and orders increased. Density changes may indicate the implantation of introduced substances into the samples.

Table 3 Helium densities of raw brown coal and activated and impregnated char

	Sample symbol	Helium density [g/cm^3]
1	A (brown coal)	1.4469
2	A2 (activated char)	1.9267
3	A2 MEA 1,5	1.8154
4	A2 MEA 3	1.7687
5	A2 DEA 1,5	1.7693
6	A2 DEA 3	1.6731
7	A2 TEA 1,5	1.7215
8	A2 TEA 3	1.4859

Fig. 2 BET analysis of sample A



To determine the influence of impregnation on the sorption capacities, nitrogen and carbon dioxide sorption measurements were conducted (Figs. 2 and 3). Low-temperature nitrogen-sorption-specific surface area measurements revealed relatively low values for raw brown coal. After activation, the value increased to 337.6 m^2/g. Compared with an activation time of 15 min (Baran et al. 2015), doubling the coal activation time led to an increase of 18 % in the specific surface area, from 285.5 to 337.6 m^2/g.

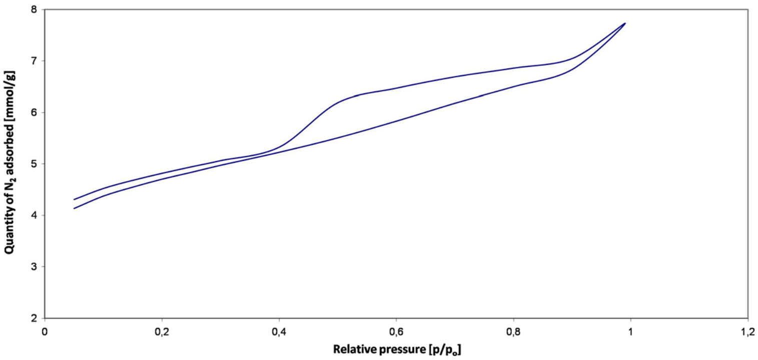
Analogous measurements were performed for selected samples impregnated with MEA and TEA at concentrations of 3 mol/dm^3 (Fig. 4). BET results for both samples indicated that the specific surface areas had decreased. In the case of sample A2 TEA3, the sample became a nonporous material. A summary plot is presented in Fig. 4.

The SEM photograph of sample A2 TEA3 is presented in the Fig. 5. From the picture, it can be seen that TEA has coated on the external surface of the material (in comparison of A2 presented in the Fig. 1).

CO_2 adsorption measurements were performed for each of the samples. It was necessary to investigate correlations between N_2 and CO_2 and to measure CO_2 affinity of the different samples. The CO_2 adsorption isotherms (Table 4) did not correlate with the associated N_2 isotherms. CO_2 adsorption on the activated, but not impregnated, samples resulted in higher specific surface areas than those resulting from N_2 adsorption. For samples impregnated with alcohol amine solutions, CO_2 sorption values were much higher than those for N_2 sorption.

Figure 6 presents CO_2 sorption results for non-impregnated char, and Figs. 7 and 8 present CO_2 sorption results for samples impregnated with different types and concentrations of amines. Figures 7 and 8 show noticeable differences in sorption values for impregnation with the different amines and amine concentrations. Results for samples impregnated with DEA were particularly noticeable. Impregnation with 1.5 mol/dm^3 DEA resulted in a specific surface area of 135.63 m^2/g, while impregnation with 3 mol/dm^3 DEA resulted in a decrease in the specific surface area to 122.29 m^2/g. Simultaneously, a slight increase in the sorption capacity was observed, from 15.19 cm^3/g (A2 DEA 1,5) to 18.77 cm^3/g (A2 DEA 3). This phenomenon was

Fig. 3 BET analysis of sample A2 (activated char)



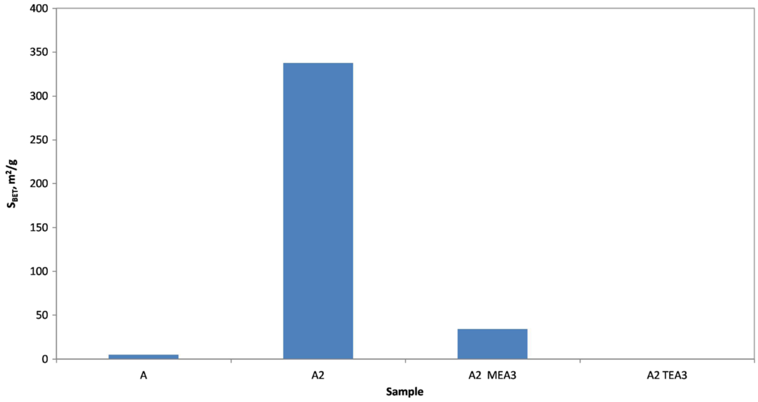


Fig. 4 Summary of BET surface area changes for selected samples

specific to samples impregnated with DEA. Samples impregnated with MEA and TEA had decreased CO_2 sorption capacities with increasing amine concentrations. This suggests that adsorbate pores are being blocked, which is consistent with a previous study by Baran et al. (2015). In the Fig. 9, the A2 MEA3 SEM photography is presented. The char framework is thickened by the MEA 3 mol/dm^3 loading, due to visible cover (in comparison to not impregnated char A2, presented in the Fig. 1); it is expected that the pores became blocked, which is in agreement with the provided analysis.

Alcohol amines were selected for this study because of their wide applications and good results for CO_2 absorption in aqueous solutions (Yu et al., 2012). Aliphatic amine solutions react with CO_2 according to Eqs. 1 and 2, which describe the CO_2 reaction mechanisms of fast-reacting (first- and second-order) amines with carbon dioxide (Gray et al. 2004, Wilk et al. 2013).

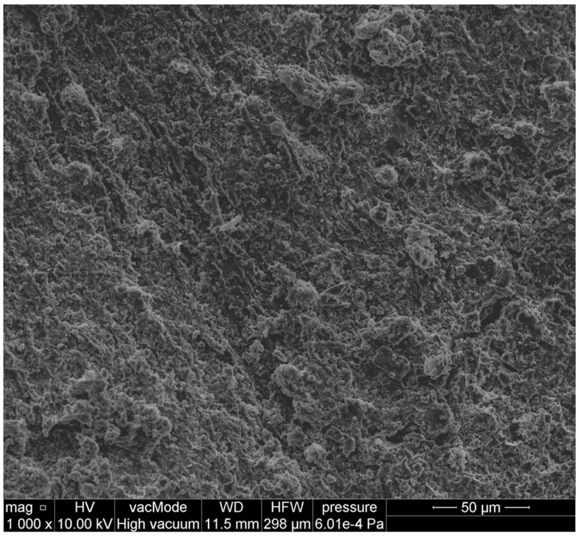


Fig. 5 SEM photographs of sample A2 TEA3

$$R_1R_2NH + CO_2 \leftrightarrow R_1R_2NH^+CO_2^- \quad (1)$$

$$R_1R_2NH + R_1R_2NH + CO_2 \leftrightarrow R_1R_2NH^+ + R_1R_2NCO_2^- \quad (2)$$

Because of higher CO_2 sorption by non-impregnated sample A2, we hypothesized that the adsorption was physical and that CO_2 particles were being deposited in the pores. As a result of the impregnation process, the pores were blocked (decreasing the specific surface area), which led to lower adsorption. This hypothesis can be proven if adsorption decreases as higher concentrations of amines are used, which was the case in this work. It should be noted that samples impregnated with higher concentrations of DEA had higher CO_2 sorption capacities. We initially assumed that CO_2 was adsorbed by amines impregnated on the support.

Table 4 CO_2 adsorption analysis results

Sample symbol	D-R$_{BET}$ [m^2/g]	V$_{mikro}$ [cm^3/g]	a $_{CO2}$ [cm^3/g]
A2	341.08	0.137	38.42
A2 MEA 1,5	141.93	0.057	15.39
A2 MEA 3	119.41	0.048	12.53
A2 DEA 1,5	135.63	0.054	15.19
A2 DEA 3	122.29	0.049	18.77
A2 TEA 1,5	189.55	0.076	20.28
A2 TEA 3	160.79	0.064	15.38

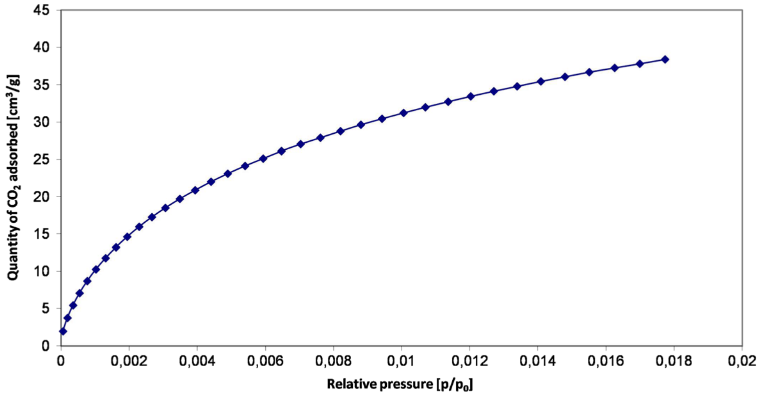


Fig. 6 CO_2 adsorption isotherm of sample A2

Based on previous work (Ko et al., 2011), third-order amines do not directly react with CO_2. Third-order amines can only be protonated. In this case, dissolved CO_2 reacts with water and creates oxonium cations and bicarbonate anions. The oxonium cations can protonate the amines, resulting in bicarbonate ions and protonated amines being present in the solution. Dissociation of dissolved CO_2 is a very slow reaction (Liu et al., 2011). Equation 3 is typical for reactions of amines with slow reaction rates (third-order) (Gray et al. 2004, Wilk et al. 2013) and is a continuation from Eqs. 1 and 2. No water molecules were present during CO_2 adsorption, which could cause low CO_2 adsorption capacities when using TEA as the impregnating agent.

$$R_1R_2R_3N + H_2O + CO_{2(aq)} \leftrightarrow R_1R_2R_3NH + HCO_3^- \qquad (3)$$

Low CO_2 sorption capacities for the char samples impregnated with MEA can be explained by the MEA chemical structure and assuming that the amine group was responsible for the affinity to CO_2. It is also possible that MEA could be positioned within the char such that its reactive amine group was located inside pores, making further reaction with CO_2 impossible. Based on these considerations, it can be assumed that the shape (branches), size, and location of the active amine group may influence its sorption capacities when used for impregnation. Furthermore, the acid-base characteristics of the support used for impregnation should be considered. We assumed that the char used as a support had acidic surface functional groups that would have affinities for the basic amine group present in MEA. Because MEA has only one amine group, it is possible that the amine group was located inside the pores. To prove this hypothesis, additional experiments to determine the surface characteristics of non-impregnated char were carried out. First, infrared spectroscopy (IR spectroscopy) was performed because it allowed for the identification of carboxyl groups that should be present on the surface

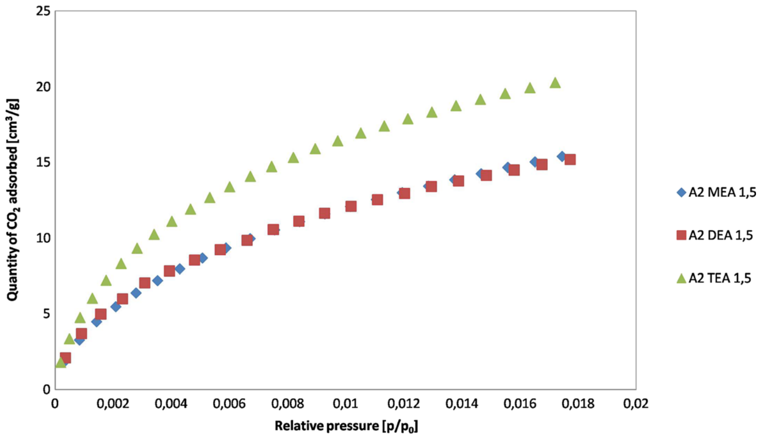


Fig. 7 CO_2 adsorption isotherms for sample A2 impregnated with different amines at 1.5 mol/dm^3 concentration

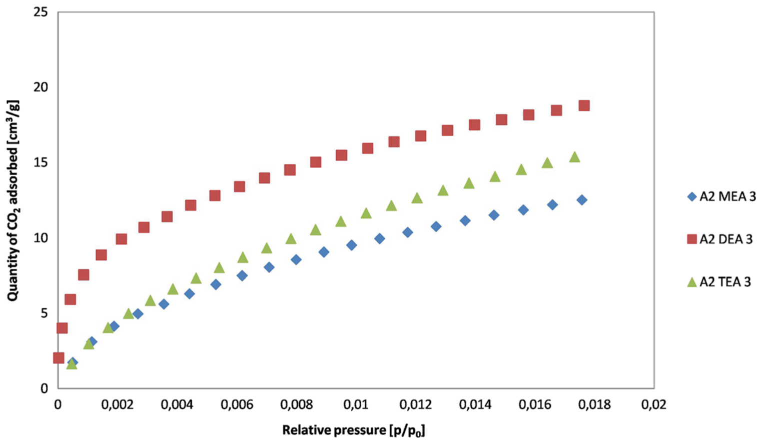


Fig. 8 CO_2 adsorption isotherms for sample A2 impregnated with different amines at 3 mol/dm^3 concentration

of char. IR analyses were performed using a Digilab Excalibur FTS 3000 Mx type spectrometer with an electrically-cooled DTGS detector. The spectrometer was equipped with an ATR attachment featuring a ZnSe crystal for multiple reflections and a transmission attachment. An FT-IR spectrum in the mid-infrared region (4000–400 cm^{-1}) for the examined char is presented in Fig. 10. Considering the char surface chemistry, the most interesting FT-IR peak was located at 1582 cm^{-1}. Despite the fact that it is a characteristic peak for carboxylate groups, it is possible to assign the peak to carboxyl groups.

For verification of the char surface composition and to gain supplementary information, we performed quantitative analysis of the surface groups present on the char using the modified Boehm titration method (Goertzen et al. 2010). This technique was used to determine the acidic oxygen surface functional groups (carboxyl, lactonic, and phenolic) or basic groups on char samples.

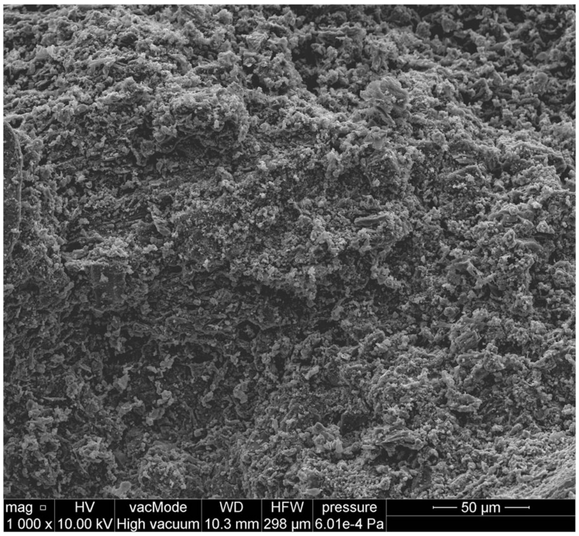


Fig. 9 SEM photography of a sample A2 MEA3

The results presented in Table 5 indicate that the char surface chemistry is basic. This may help to explain the relatively high CO_2 sorption capacity of non-impregnated char sample A2. Because of the high quadrupole moment of CO_2, its adsorption depends on polar groups or ions on the char surface. Furthermore, it is expected that the basic nature of the support will attract amine functional groups to the acidic locations present on the char surface. We believe that this will cause amines to be positioned in a way that restricts activity between CO_2 and the amine functional group. We performed analogical calculations based on those published by Lee et al. (2013) to determine the theoretical quantity of amine impregnated on the surface of the char support. These results are presented in Table 6.

The data presented in Table 6 indicate low theoretical percentages of amines impregnated on the support. The observed increases in surface loading with increasing amine concentrations and orders were preserved in the calculations. Comparing the theoretical results with the CO_2 adsorption values, agreement only occurred for samples impregnated with 1.5 mol/dm^3 amines. We made the additional assumption that because of low amine loading on the support, a significant proportion of physical sorption is possible, despite negligible chemical sorption. To investigate the possibility of physical sorption, analyses of surface areas calculated from carbon dioxide sorption were performed (Table 7).

As noted previously, the specific surface area values decreased because of impregnation blocking the porous

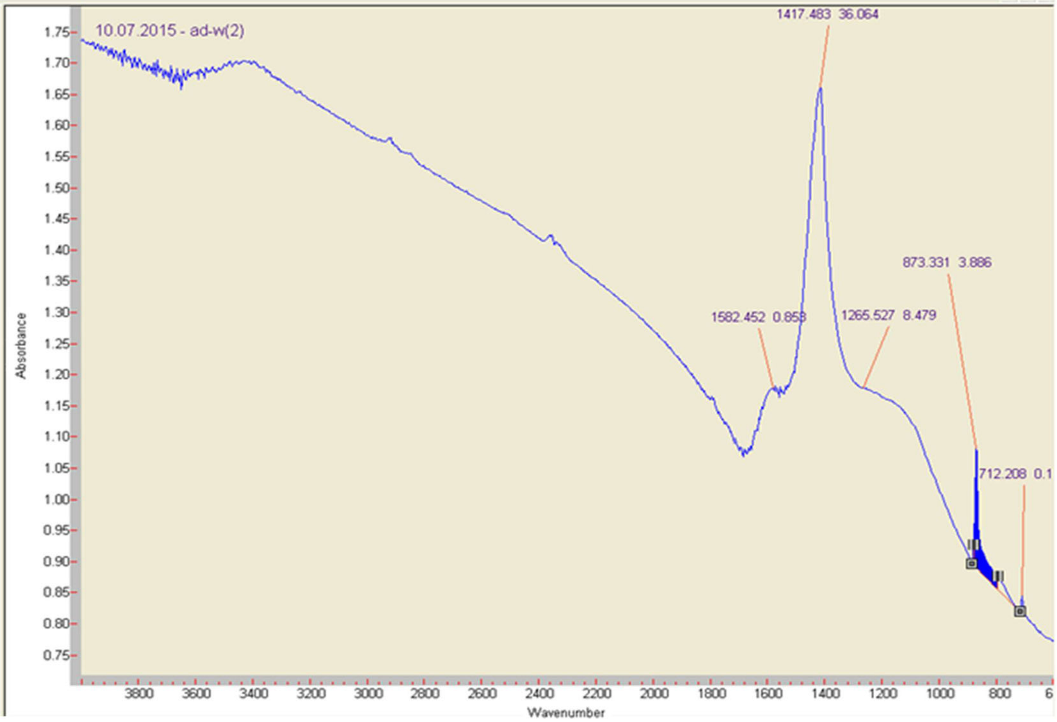


Fig. 10 FT-IR spectrum of char sample A2

char structure. In the case of samples impregnated with 1.5 mol/dm^3 concentration amines, the surface areas calculated from CO_2 adsorption decreased from A2 TEA 1,5 > A2 MEA 1,5 > A2 DEA 1,5. The analogous series for CO_2 adsorption values decreased from A2 TEA 1,5 > A2 MEA 1,5 > A2 DEA 1,5. These results, along with the low theoretical percentages of amines impregnated in the support, suggest that when samples are impregnated with 1.5 mol/dm^3 of different amines, there is no CO_2 chemical adsorption and physical adsorption is dominant. This hypothesis is not accurate for impregnation with amine concentrations of 3 mol/dm^3. In these cases, CO_2 adsorption values decreased in the order: A2 DEA 3 > A2 TEA 3 > A2 MEA 3. These values did not decrease in agreement with the theoretical percentage of amines impregnated in the support. Table 8 shows that increasing surface area was not correlated with increased CO_2 adsorption.

From Table 8, it is evident that physical adsorption for samples impregnated with 3 mol/dm^3 of amines is not dominant. Based on the theoretical percentages of amines impregnated on the support, it should be noted that when higher concentrations of amines were used, there were slight increases in the theoretical percentages of amines impregnated in the support. It should also be noted that samples with the highest theoretical percentages of amines impregnated in the support were not the

Table 5 Boehm method results for the surface compositions of char samples

Basic groups [mmol/g]	Acidic groups [mmol/g]			
	Total	Carboxyl	Lactonic	Phenolic
5.35	0.35	0.3	0.05	0

Table 6 Theoretical quantity of amine impregnated onto the char support

Impregnated char	Theoretical percentage of impregnation
A2 MEA 1,5	13.7
A2 MEA 3	13.8
A2 DEA 1,5	13.9
A2 DEA 3	14.1
A2 TEA 1,5	14.1
A2 TEA 3	14.5

Table 7 Comparison of surface area calculated from CO_2 sorption for sample A2 and samples impregnated with amines of 1.5 mol/dm^3 concentration

Sample symbol	D-R$_{BET}$, [m^2/g]	% of surface are in correlation to A2 surface [%]
A2	341.08	–
A2 MEA 1,5	141.93	41
A2 DEA 1,5	135.63	40
A2 TEA 1,5	189.55	56

samples with the highest CO_2 sorption values. Based on previous work (Maroto-Valer et al. 2008), the amines used have different affinities for CO_2 and the maximum theoretical percentages of amines impregnated in a support do not have to be associated with the highest CO_2 sorption values. Furthermore, in the case of first-, second-, and third-order amines, there are differences in reaction mechanisms. Because no correlations between CO_2 sorption values and surface areas were observed, chemisorption is the dominant process for samples impregnated with 3 mol/dm^3 amines. From the MEA and TEA impregnation results, it was decided to focus more on DEA impregnation. We suggest that the DEA impregnation results provided the most relevant results. The most interesting result from DEA impregnation was the increase in CO_2 sorption values with increasing DEA concentration (Table 9).

The data in Table 9 show that CO_2 adsorption increased with increasing amine concentration and was not correlated with surface area. These results confirm that chemisorption was more important than physisorption. This relationship was not identified for samples impregnated with MEA and TEA. Figure 11 shows that MEA and TEA impregnated samples demonstrate the opposite relationship: when amine concentrations increased, CO_2 adsorption decreased. This supports the reaction mechanism hypothesis and highlights the importance of physisorption for these samples.

Table 8 Comparison of surface area calculated from CO_2 adsorption for sample A2 and samples impregnated with amine concentrations of 3 mol/dm^3

Sample symbol	D-R$_{BET}$, [m^2/g]	% of surface are in correlation to A2 surface
A2	341.08	–
A2 MEA 3	119.41	38
A2 DEA 3	122.29	36
A2 TEA 3	160.79	47

Table 9 Comparison of surface area, calculated from CO_2 sorption, with CO_2 adsorption values for samples impregnated with DEA

Sample symbol	D-R$_{BET}$, [m^2/g]	a CO_2, [cm^3/g]
A2 DEA 1,5	135.63	15.19
A2 DEA 3	122.29	18.77

It should be noted that the same trends were observed for experiments performed on char that had been activated for 15 min (Baran et al. 2015). What is interesting, when comparing the results from earlier experiments with char sample A1 (activated for 15 min) and char sample A2 (activated for 30 min), is that the specific surface areas calculated from low-temperature N_2 sorption showed the opposite pattern compared with those for carbon dioxide sorption. Despite the extended activation time and higher BET surface areas, the CO_2 sorption values for A2 (impregnated and non-impregnated) samples were lower than for those for the A1 samples. We believe that this may be related to higher numbers of mesopores in the A2 samples. Figure 12 presents the proportions of micro-, meso-, and macropores in A1 and A2 char samples. More mesopores were apparent in the A2 samples.

During extended activation times, existing pores broaden or new pores with larger dimensions are created from the disappearance of walls between neighboring micropores. These processes result in the creation of more mesopores. It is questionable whether more mesopores would be advantageous for impregnation. The introduction of more or larger amines may be more effective for mesoporous materials.

Conclusions

Activation of coal samples resulted in obtaining porous materials. Lengthening the activation time from 15 min (Baran et al. 2015) to 30 min increased the specific surface areas of the char samples. Impregnation with different amines decreased the specific surface areas because the pore structure became blocked. Additionally, SEM photographs were provided to present the

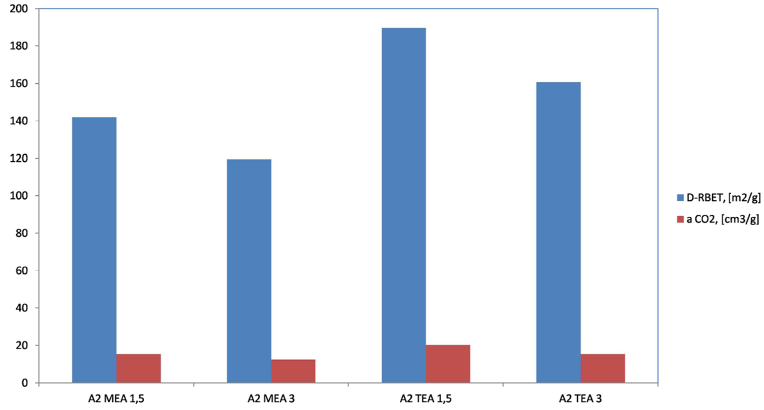


Fig. 11 Comparisons of surface areas, calculated from CO_2 sorption, with the adsorption values for samples impregnated with MEA and TEA

amine cover on the surface. In the impregnated samples, decreased N_2 and CO_2 sorption capacities were observed. No correlations between the N_2 and CO_2 values were apparent. We found that surface areas calculated from CO_2 sorption values, regarded as the surface available for carbon dioxide adsorption, did not show any correlation with N_2 adsorption. The impregnated samples all had different CO_2 sorption values, likely because they would act through different reaction mechanisms. The differences in first-, second-, and third-order amine reaction mechanisms were established. These mechanisms led to an understanding of lower CO_2 sorption values for samples impregnated with a 3 mol/dm^3 TEA solution. While studying the MEA samples impregnated with 3 mol/dm^3 MEA, we investigated the structure of MEA along with the presence of acidic/basic sites on the surface of non-impregnated char. In the presence of one amine group, it is possible that the amine group is located inside the pores, resulting in a lack of activity towards CO_2 adsorption. Char impregnated with 3 mol/dm^3 DEA showed the best CO_2 sorption capacity and the dominant process was identified as chemisorption. Based on comparisons with the surface area, it was found that physisorption would be minimal in this case.

All samples impregnated with 1.5 mol/dm^3 amines featured physisorption as the dominant adsorption process. This is verified by correlations of CO_2 sorption values with surface areas, and low theoretical percentages of amines impregnated in the support. Despite the low theoretical percentage of amine impregnated on the support not giving an accurate quantity of amine impregnated, we assumed that the overall trend was preserved (Lee et al. 2013).

Based on these experiments and previous results (Baran et al. 2015), we suggest that the proportion of mesopores has an influence on CO_2 adsorption. The slight increase in mesopores in sample A2 could cause the lower CO_2 sorption values measured compared with the samples A1 activated for 15 min. This could prove that physisorption is the most important process.

Further work should focus on impregnation using higher amine concentrations and measuring CO_2 sorption at higher temperatures (helping to estimate chemisorption processes). Longer activation times and/or the

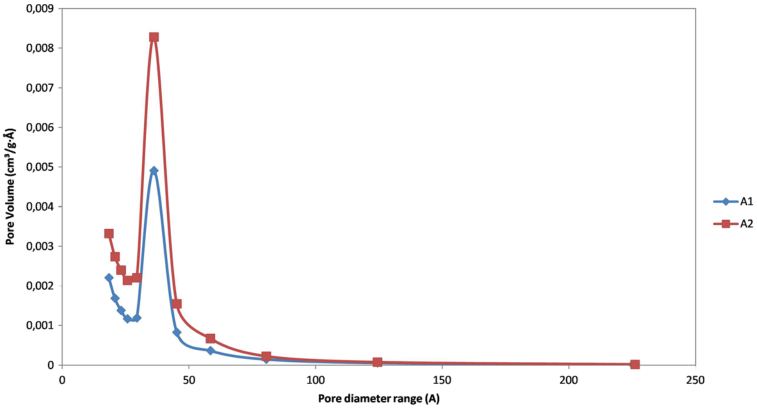


Fig. 12 Proportion of micro-, meso-, and macropores in samples activated for 15 min (*A1*) and 30 min (*A2*), calculated from low-temperature N_2 sorption

introduction of a carbonization procedure should also be studied. Changing the activating agent to water vapor could also be considered. This may prove that third-order amines preferably adsorb CO_2 in the presence of water vapor.

Acknowledgments The authors are grateful to the AGH University of Science and Technology (Project 11.11.210.244) for its financial support of this work.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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