

The use of microwave radiation for obtaining carbonaceous adsorbents from biomass and their use in elimination of inorganic pollutants

Justyna Kazmierczak-Razna¹ · Robert Pietrzak¹

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Abstract A new technology of obtaining activated carbons by physical and direct activation of biomass with the use of microwave radiation is described. The effect of activation temperature (700 and 800 °C) and two periods of time (15 and 30 min) on the textural parameters, acid–base character of the surface and sorption properties of activated carbons was tested. The resulting carbons were characterized by low-temperature nitrogen sorption and determination of pH as well as the number of surface oxygen groups. The sorption properties of the activated carbons obtained were characterized by determination of nitrogen dioxide and hydrogen sulphide adsorption in dry and wet conditions as well as by iodine removal from aqueous solution. The final products were adsorbents of surface area ranging from 291 to 368 m²/g and pore volume from 0.20 to 0.26 cm³/g, showing basic character of the surface. The results obtained in our study have proved that suitable choice of the pyrolysis and activation procedure for hay with the use of microwave radiation permit producing adsorbents with good capacity toward toxic gases of acidic character as well as inorganic pollutants of molecules of size similar to that of iodine molecules.

Keywords Biomass · Microwave radiation · Activated carbons · Iodine number · NO₂/H₂S adsorption

1 Introduction

Climate changes and the need to reduce the use of non-renewable resources and energy have become the fundamental problems of our times. Their solution should improve the standard of life of the present and future generations. The issue of the natural environment protection has reached global character (Nowicki et al. 2012). The measures aimed at reducing the level of emission of pollution include development of new technologies and correct storage of waste products (Yuen and Hameed 2009). On the other hand, rapid development of economy and industry has brought a significant increase in the emission of nitrogen(IV) dioxide and hydrogen sulphide to the environment. These gases are highly toxic and harmful for humans and vegetation. Long-lasting exposure to these gases can cause irritation, nausea, problems with concentration and even death. In view of that, the legal rules on the maximum admissible levels of NO₂ and H₂S concentrations are increasingly restrictive, which forced the need for monitoring of the level of these gases both in the natural environment and in certain workplaces (Kazmierczak-Razna et al. 2015a). The technologies developed to reduce or eliminate the emission of toxic gases have recently been using activated carbons whose very high adsorption capacity makes them attractive for removal of pollutants from the air (Nowicki and Pietrzak 2011; Nowicki et al. 2013, 2014; Guo et al. 2006; Hauchhum and Mahanta 2014; Adib et al. 1999; Badosz 2002) and water (Nethaji et al. 2010; Ferrero 2007; Malik 2004; Pal and Deb 2014; Ahmad and Hameed 2010; Wu and Tseng 2008; Benadjemia et al. 2011).

Literature provides a large number of methods for the production of activated carbons, employing mainly conventional heating (Nowicki et al. 2008, 2009; Sych et al.

✉ Justyna Kazmierczak-Razna
justykaz@amu.edu.pl

✉ Robert Pietrzak
pietrob@amu.edu.pl

¹ Laboratory of Applied Chemistry, Faculty of Chemistry, Adam Mickiewicz University in Poznań, Umultowska 89b, 61-614 Poznań, Poland

2012; Puziy 2011; Malik et al. 2002). The drawbacks of this way of heating include non-uniform heating of the samples, long time of heating and high temperatures of carbonisation and activation, which means high energy consumption and thus high cost of production of carbonaceous materials (Jiang and Ma 2011; Remya and Lin 2011). That is why alternative methods have been proposed, among them the method based on the use of microwave radiation that has attracted much interest recently (Kazmierczak-Razna et al. 2015b; Li et al. 2008; Liu et al. 2010; Hesas et al. 2013).

The main aim of this work was to obtain a series of low cost activated carbons from hay with the use of microwave radiation as well as characterisation of their physicochemical and sorption properties towards gas phase pollutants represented by nitrogen dioxide and hydrogen sulphide and liquid phase pollutants represented by inorganic substances of molecules of size similar to that of iodine molecules. Different preparation parameters were examined in order to evaluate the influence of temperature and time of activation on the adsorption properties of the final product. Finally, we tried to correlate the surface properties with the ability of the activated carbons to remove a nitrogen dioxide and hydrogen sulphide and inorganic pollutant from water.

2 Experimental section

2.1 Samples preparation

The precursor of char was the post-agricultural waste—low quality hay in the form of cylinders of 0.8 cm in diameter and 1 cm in length. The initial material was subjected to pyrolysis (P) in a muffle chamber in a microwave furnace (Phoenix, CEM) under a stream of nitrogen with a flow rate of 0.170 L/min in 700 °C (HP). In the microwave furnace used it is not possible to control the power consumed by the furnace. However, it was possible to control temperature by a thermocouple installed inside the muffle chamber. In the final temperature, sample was kept 60 min and then cooled down in inert atmosphere. The char obtained were next subjected to physical activation (A) in a microwave oven. This process was carried out at two temperatures 700 (HPA7) and 800 °C (HPA8), under a stream of carbon dioxide with a flow rate of 0.250 L/min, for 15 and 30 min (HPA7-15 or HPA7-30) and 800 °C (HPA8-15 or HPA8-30). Some part of the precursor was also subjected to direct activation (DA) with CO₂, which was performed in the same conditions as physical activation (HDA7-15, HDA7-30, HDA8-15 and HDA8-30).

2.2 Analytical procedures

The elemental analysis of the activated carbons were performed on an elemental analyser CHNS Vario EL III (Elementar Analysensysteme GmbH, Germany).

The content of ash was performed according to the Data National Standards ISO 1171:2002. The dried sample was burned in a microwave oven at 850 °C, for 60 min. The ash content was expressed as: %ASH = ((mass of crucible and ash – mass of crucible)/(mass of crucible and sample – mass of crucible)) * 100 %.

Characterization of the pore structure of activated carbons was performed on the grounds of low-temperature nitrogen adsorption–desorption isotherms measured on Autosorb iQ surface area analyser (Quantachrome Instruments USA). On the grounds of results of the measurements BET surface area, total pore volume and pore diameter were determined. The S_{BET} surface area was assessed within the range of relative pressures from 0.05 to 0.3. The total pore volume (V_t) was calculated by measuring the amount of liquid nitrogen adsorbed at a relative pressure $p/p_0 = 0.99$. The average pore diameter (d) was calculated from equation $d = 4V_t/S_{\text{BET}}$. Moreover micropore volume and micropore surface area were calculated using t-plot method.

The content of surface oxygen functional groups were determined by the Boehm method. 0.25 g of carbon sample was placed in 25 mL of 0.1 M solutions of either sodium hydroxide or hydrochloric acid. The vials were sealed and shaken for 24 h and then 10 mL of each filtrate was pipetted and the excess of base or acid was titrated with 0.1 M HCl or NaOH, as required. The numbers of acidic sites of various types were calculated under the assumption that NaOH neutralizes all acidic groups and HCl reacts with all basic group.

The pH surface of samples was determined with the following procedure: approximately 0.20 g of each sample were suspended on 10 mL of distilled water. Suspensions were stirred overnight to reach equilibrium and measuring the pH periodically until readings were constant.

2.3 Adsorption studies

2.3.1 H₂S and NO₂ adsorption

The samples sieved to a particle size between 0.75 and 1.65 mm were packed into a glass column (length 300 mm, internal diameter 9 mm, bed volume 3 cm³). Dry or moist air (70 % humidity) with 0.1 % of H₂S or NO₂ was passed through the dry bed of the adsorbent at 0.450 L/min. The breakthrough of H₂S or NO₂ were monitored using Q-RAE PLUS PGM-2000/2020 with electrochemical sensors. The tests were stopped at the breakthrough concentration of

100 ppm (in case of H₂S) or 20 ppm (for NO₂) because of the electrochemical sensor limits. The interaction capacities of each sorbent in terms of milligram of H₂S or NO₂ per gram of adsorbent were calculated by integration of the area above the breakthrough curves, and from the toxic gas concentration in the inlet gas, flow rate, breakthrough time and mass of sorbent (Pietrzak and Bandosz 2007). Additionally to check the NO₂ reduction, in a result at its reaction with carbonaceous material the concentration of NO in the system was also monitored till 200 ppm.

After the breakthrough tests, for 30 min (for NO₂) and 5 min (for H₂S) all samples were exposed to a flow of carrier air (360 mL/min).

2.3.2 Iodine adsorption

Determination of the iodine adsorption was performed according to the ASTM D4607-94(2006) standard.

3 Results and discussion

3.1 Elemental composition of the activated carbons

The precursor used, hay (H), is characterised with low content of carbon (48.1 wt%), very high content of volatile matter (over 80 wt%) and a low content of mineral substances (ash) of about 5.7 %. Therefore, in order to increase the degree of carbon content and reduce the content of volatile matter, one part of the precursor was subjected to direct activation, while the other part was at first subjected to pyrolysis at 700 °C and then by physical activation by CO₂ in two different temperature and temporal variants.

Analysis of the data collected in Table 1 shows that the processes of activation, both the precursor and the char obtained from this precursor, cause significant changes in

the structure of the precursor. The greatest changes with respect to the initial material, that is the greatest increase in the content of C^{daf} and the greatest decrease in O^{daf}, are observed for the samples subjected to direct activation at 800 °C for 30 min (HDA8-30), at which the processes of pyrolysis and activation take place simultaneously. The activated sample obtained in this way shows much higher content of carbon C^{daf}, increased by 38.6 wt% and much lower content of O^{daf}, reduced by 33.8 wt%, with respect to those of the precursor. Moreover, the higher the activation temperature, the greater the increase in the content of C^{daf}, while the lower the content of O^{daf}. The thermochemical processing gives also a significant decrease in the content of hydrogen with respect to that in the precursor, the most pronounced decrease in the content of hydrogen was observed in the samples subjected to physical activation at 800° for 15 min (HPA8-15). The activation causes also significant changes in the content of nitrogen and sulphur. In all samples after activation the content of these heteroatoms considerably increased with respect to their content in the precursor, which is a consequence of much decrease in the volatile matter and oxygen. On the basis of the data obtained, it can be concluded that the content of nitrogen and sulphur in all products of activation was at a similar level.

3.2 Textural parameters of activated carbons

According to the data presented in Table 2, irrespective of temperature and time of activation, the activated carbons obtained are characterised by not much developed surface area (from 291 to 368 m²/g) and moderate pore volume (0.20–0.26 cm³/g). Comparison of the data for particular samples implies that the textural parameters of the samples activated for 15 min are more beneficial than those of the samples activated for 30 min, irrespectively of the temperature of activation. A possible reason is that a too long time of activation (30 min) could be too drastic for the precursor used and cause burning of considerable amount of the organic substance and therefore, destruction of the porous structure of the materials obtained. The data from Table 2 also indicate that the temperature of activation influences the textural parameters of the samples. The activation at 800 °C has a beneficial effect on the surface area development only in the process of direct activation. All the samples subjected to physical activation have similar textural parameters. According to the data given in Table 2, the activated carbon samples are characterised by microporous structure as the contribution of micropores in the total pore volume varies from 72 to 83 %. The other part are small mesopores of diameters varying from 2.61 to 2.87 nm.

Table 1 Elemental analysis of the activated carbons and the yield of activation process (wt%)

Sample	Ash	C ^{daf1}	H ^{daf}	N ^{daf}	S ^{daf}	O ^{daf2}	Yield
HPA7-15	29.1	83.4	1.3	2.3	0.4	12.6	71.0
HPA7-30	25.6	84.8	0.7	2.3	0.3	11.9	72.3
HPA8-15	27.6	86.1	0.3	2.1	0.3	11.1	70.3
HPA8-30	27.2	86.0	0.5	2.2	0.4	10.9	68.1
HDA7-15	23.6	81.7	1.9	2.5	0.4	13.4	21.6
HDA7-30	21.8	79.1	1.1	2.4	0.3	17.1	23.1
HDA8-15	26.1	82.2	1.5	2.3	0.5	13.5	18.0
HDA8-30	23.7	86.7	0.5	2.1	0.3	10.4	20.2

¹ Dry-ash-free basis; ²determined by difference

Table 2 Textural parameters of the activated carbons obtained

Sample	Total surface area (m ² /g)	Micropore area (m ² /g)	Total pore volume (cm ³ /g)	Micropore volume (cm ³ /g)	V _{mic} /V _t	Average pore diameter (nm)
HPA7-15	368	338	0.25	0.19	0.76	2.76
HPA7-30	354	325	0.25	0.18	0.72	2.80
HPA8-15	363	339	0.24	0.19	0.79	2.65
HPA8-30	360	336	0.23	0.19	0.83	2.61
HDA7-15	319	293	0.22	0.16	0.72	2.78
HDA7-30	291	268	0.20	0.15	0.75	2.76
HDA8-15	365	343	0.26	0.20	0.77	2.87
HDA8-30	356	326	0.25	0.18	0.72	2.75

3.3 Acid–base properties of the activated carbons

The data from Table 3 show that the activated carbon adsorbents have surface of basic character as their pH values vary from 9.2 to 10.5. The content of surface oxygen groups varies from 2.22 to 3.32 mmol/g and dominant among them are those of basic character. Therefore, the type and number of surface oxygen functional groups depend first of all on the temperature and time of activation. The highest total content of functional groups was determined on the surface of sample HPA8-30, while the lowest content of functional groups was found on the samples obtained by direct activation of the precursor at 700 °C, that is samples HDA7-15 and HDA7-30, containing 2.22 and 2.24 mmol/g of functional groups. The data from Table 3 provide evidence that both temperature and time of activation influence the content of surface oxygen functional groups, while the influence of the method of activation is much smaller. For the majority of samples, an increase in the activation temperature from 700 to 800 °C and extension of the activation time from 15 to 30 min are conducive to generation of a greater number of basic groups and disappearance of groups of acidic character. The samples obtained by direct activation show a greater content of surface oxygen groups of acidic character than

the analogous samples obtained in the two-stage process of activation.

3.4 Sorption abilities of the activated carbons toward nitrogen dioxide

Analysis of the data collected in Table 4 shows that the sorption capacity of the activated carbons studied towards nitrogen dioxide differ, depending on the parameters and the method of activation and conditions of the adsorption process, so it is difficult to select one sample of the best properties. The majority of activated carbon samples show greater adsorption capacity in dry conditions. The most effective adsorbent in dry conditions was sample HPA8-30, while the most effective in wet conditions was HDA8-15. The least effective adsorbents of nitrogen dioxide in dry and wet conditions were samples HDA8-15 and HDA8-30, obtained by direct activation at 800 °C for 15 or 30 min. On the basis of the same data from Table 4, it was possible to confirm the earlier assumption that the activation temperature also influences the effectiveness of nitrogen dioxide removal by the samples studied. Irrespective of the variant of adsorption, almost all samples activated at 800 °C show greater sorption capacity than those activated at 700 °C. The time of activation has only slight effect on

Table 3 Acid base properties of the activated carbons obtained

Sample	PH	Acidic groups (mmol/g)	Basic groups (mmol/g)	Total content (mmol/g)
HPA7-15	10.5	0.10	2.64	2.74
HPA7-30	9.7	0.03	3.24	3.27
HPA8-15	10.4	0.00	2.51	2.51
HPA8-30	9.2	0.00	3.32	3.32
HDA7-15	10.3	0.22	2.02	2.24
HDA7-30	10.0	0.15	2.07	2.22
HDA8-15	10.1	0.12	2.41	2.53
HDA8-30	10.5	0.00	2.34	2.34

Table 4 NO₂ breakthrough capacities in dry and wet conditions of the activated carbons obtained (mg/g_{ads})

Sample	NO ₂ breakthrough capacities (mg/g)	
	Dry conditions	Wet conditions
HPA7-15	37.3	34.0
HPA7-30	40.4	34.5
HPA8-15	44.9	37.5
HPA8-30	46.0	44.7
HDA7-15	32.0	52.6
HDA7-30	29.2	41.6
HDA8-15	31.0	55.1
HDA8-30	36.7	28.9

the sorption capacity of the samples towards NO₂. The extension of the time of physical activation from 15 to 30 min only to a small degree (of a few mg of NO₂ per 1 g of the adsorbent) improves the sorption capacity towards NO₂ both in dry and in wet conditions. The extension of the time of the process of direct activation of the precursor deteriorated the sorption capacity of the sample in wet conditions of adsorption. Small deterioration of the sorption capacity with extension of the activation time was also noted for sample HDA7-15 when the adsorption process was performed in dry conditions.

Sorption capacities of most of the samples we studied are similar to those reported by Nowicki et al. (2010) for the carbon samples obtained by chemical activation of plum stones by KOH. However, the procedure of obtaining activated carbon to be used as adsorbent should be further developed to reach the level of 140 mg/g, reported by Bashkova and Bandosz (2009) for carbon samples modified with nitrogen.

Figure 1 presents the adsorption–desorption curves of NO₂ recorded for the process in dry (Fig. 1a) and wet

(Fig. 1b) conditions. The majority of the curves have similar shapes, which suggests a similar mechanisms of adsorption of NO₂ on the samples studied. For many samples, for some time of adsorption the concentration of NO₂ is close to zero and the adsorption curve runs almost in parallel to the x axis. Then a breakthrough is observed and the particular curves differ in the time at which particular ppm values are recorded. For none of the samples a rapid increase in the concentration of nitrogen dioxide to the limiting values of 20 ppm was observed in a short time.

The adsorption–desorption curves also imply that for the majority of samples, both in dry and wet conditions, at the stage of desorption a rapid decrease in the concentration of NO₂ is observed. This fact suggests that most of the adsorbed nitrogen dioxide undergoes chemisorption on the activated carbon samples studied. It suggests that most of the adsorbed NO₂ was strongly bonded through the surface oxygen functional groups of basic character that were able to react with NO₂.

While performing sorption capacity measurements, also the concentration of nitrogen oxide was recorded. NO can be formed as a result of NO₂ reduction in the reactions with the surface functional groups on the carbon samples. The character of curves presented in Fig. 2a and b implies that the carbon samples studied show different abilities to reduce NO₂ to NO. The rate of such reduction depends considerably on the conditions of adsorption and is the highest in wet conditions. This observation means that the maximum concentration of NO (200 ppm) is reached in much shorter time in the presence of moisture in the system.

3.5 Sorption abilities of activated carbons toward H₂S

The samples studied were also tested as adsorbents of hydrogen sulphide and similarly as noted for adsorption of NO₂, the sorption capacity of the samples depended on the

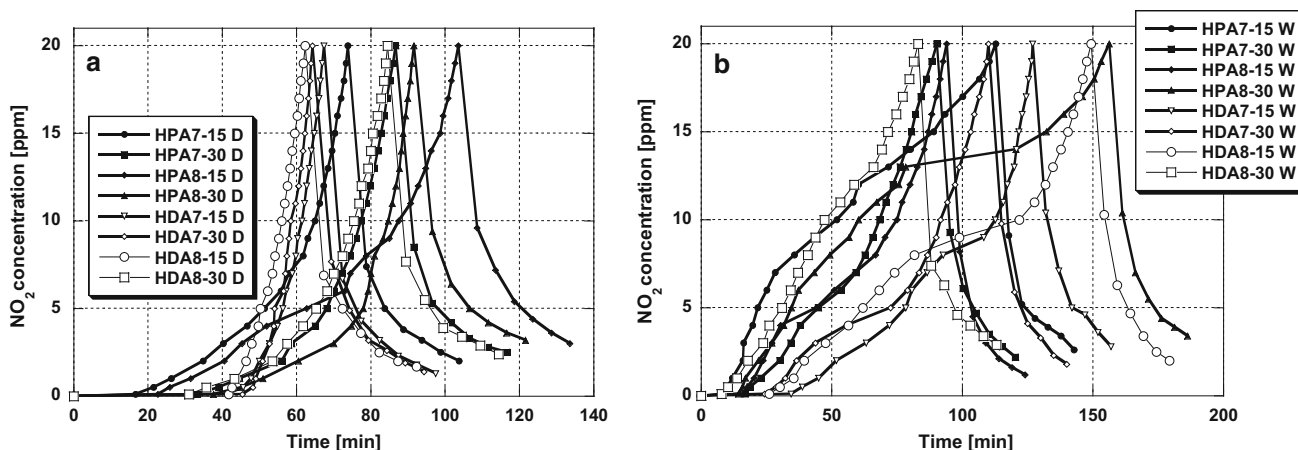


Fig. 1 NO₂ breakthrough curves obtained during adsorption in dry conditions (a) and wet conditions (b)

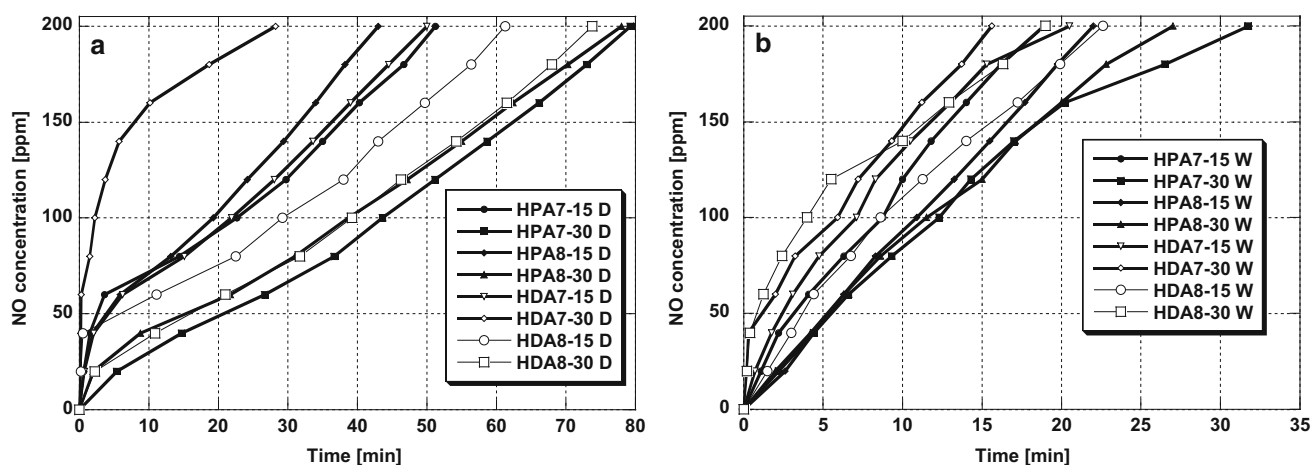


Fig. 2 NO emission curves obtained during adsorption in dry (a) and wet (b) conditions

temperature and time of activation. As follows from analysis of the data collected in Table 5, the most effective adsorbent of H_2S was sample HPA7-15, whose sorption capacity in dry and wet conditions was 11.0 and 39.0 mg/g, respectively. The least satisfying results were obtained for the sample obtained by direct activation of precursor at 700 °C for 30 min, whose sorption capacity towards H_2S was ~ 5 and ~ 12 mg/g in dry and wet conditions, respectively. The activation temperature has a significant effect on the sorption capacity towards H_2S , which was particularly pronounced for the samples obtained by direct activation—with the temperature increase from 700 to 800 °C, and when adsorption took place in wet conditions, the sorption capacity increased twice. The time of activation also influences the ability of H_2S removal, extension of the activation time from 15 to 30 min increased the sorption capacity of samples HPA8-15 D and HDA8-15 D, had a very small influence on the capacity of HDA7-15 W, while for the other samples it was not beneficial and even caused a decrease in the sorption capacity. As mentioned above, also the conditions of adsorption influenced the

sorption capacity towards H_2S , for all samples the sorption capacity in wet conditions was higher.

In our previous work (Kazmierczak-Razna et al. 2015a) we used activated carbons prepared from sawdust pellets from coniferous wood to remove hydrogen sulphide, the highest sorption capacity was 6.2 mg/g. It shows that we were able to optimize the conditions for the activation process and obtain materials characterised by higher sorption capacities reaching 39.0 mg/g. Unfortunately, these results are very modest when compared to those obtained for other carbonaceous sorbents, especially as far as H_2S removal is concerned. The reported sorption capacity achieved towards this gas exceeded 100 mg/g_{ads} (Bandosz 2002), and for some materials—even 200 mg/g (Nowicki et al. 2014).

The adsorption–desorption curves recorded for all samples are presented in Fig. 3a and b and their character is similar. They are composed of a flat section when the adsorption curve coincides with the x axis and then show a characteristic point corresponding to breakthrough of the bed and from this point on the concentration of H_2S increases to the limit value of 100 ppm. For the samples showing low sorption capacities towards H_2S , this fragment of the adsorption curve is close to a line ascending in parallel to the y axis, while for the other samples (e.g. HDA7-15 D, HDA7-15 W, HPA8-15 W) this section of the curve has a more gentle character.

Analysis of the adsorption–desorption curves indicates that for all samples, in both dry and wet conditions, at the stage of desorption a rapid decrease in H_2S concentration is observed. This fact suggests that the majority of H_2S that was adsorbed was in fact strongly chemisorbed by the surface functional groups on the samples studied.

3.6 Iodine number

As follows from the data presented in Fig. 4, the activated carbons are characterised by the iodine number varied from

Table 5 H_2S breakthrough capacities in dry and wet conditions of the activated carbons obtained (mg/g_{ads})

Sample	H_2S breakthrough capacities (mg/g)	
	Dry conditions	Wet conditions
HPA7-15	11.0	39.0
HPA7-30	5.9	24.0
HPA8-15	6.0	32.3
HPA8-30	8.4	25.0
HDA7-15	10.9	12.5
HDA7-30	5.2	12.3
HDA8-15	8.3	26.1
HDA8-30	9.7	25.7

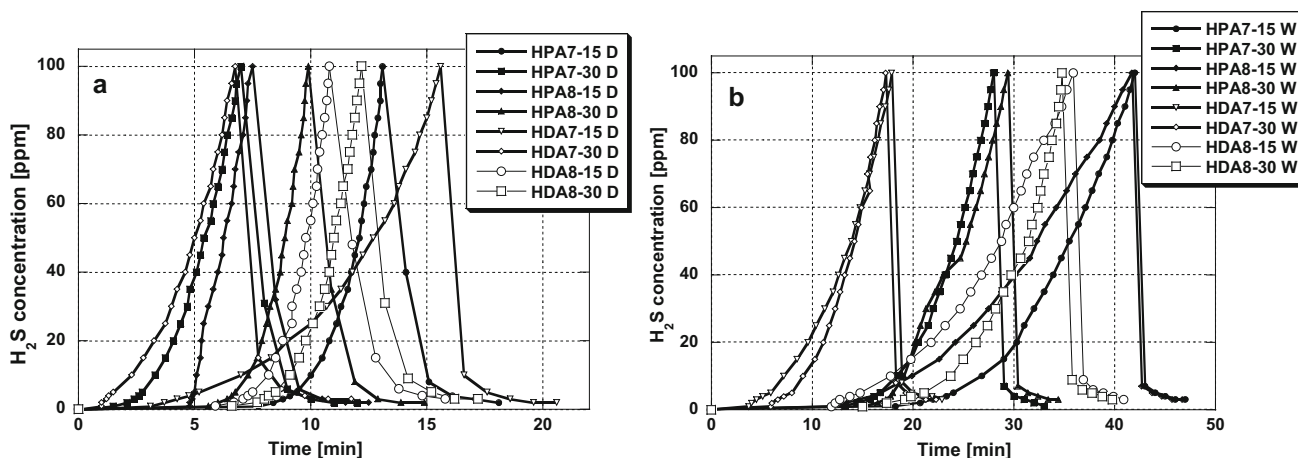
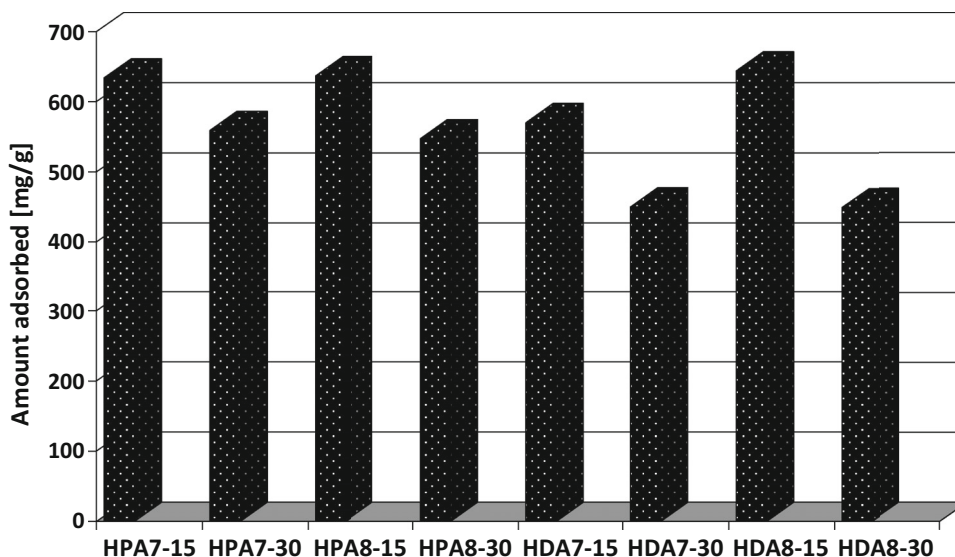


Fig. 3 H₂S breakthrough curves obtained during adsorption in dry conditions (a) and wet conditions (b)

Fig. 4 Adsorption of iodine onto activated carbons



449 to 644 mg/g, so lower than the commercial adsorbents available on the market. The sorption capacity of the activated carbon samples studied towards iodine was found to change in response to the activation temperature and time. The most effective adsorbent (nearly 644 mg/g) was the sample obtained by direct activation of the precursor at 800 °C for 15 min (HDA8-15), whose sorption capacity towards iodine was by almost 200 mg/g greater than that of the sample obtained by direct activation at the same temperature but for 30 min (HDA8-30). Irrespectively of the temperature of activation, the samples subjected to activation for 15 min had higher iodine numbers. Most probably it is a consequence of a better developed surface area in the samples activated for 15 rather than 30 min (Table 2). The increase in the activation temperature, irrespectively of the activation method, had no significant influence on the iodine adsorption. For samples HPA7-15

and HPA8-15 the higher temperature increased the adsorption capacity by only 3 mg/g, while the sorption capacity of samples HPA7-30 and HPA8-30 towards iodine decreased for activation at the higher temperature. Similar relations were noted for the adsorbents obtained by direct activation of the precursor.

4 Conclusions

It has been shown that the materials obtained by direct activation of low quality hay or by pyrolysis of this precursor followed by physical activation by microwave radiation are adsorbents show interesting physicochemical properties. Although the materials obtained are characterised by not much developed surface area and porous structure, thanks to the basic nature of their surface they

can be attractive adsorbents for removal of acidic pollutants such as nitrogen dioxide and hydrogen sulphide. The sorption capacity of the adsorbents obtained depend on the temperature, time and method of activation as well as on the conditions of adsorption, therefore it is difficult to select one sample of the optimum sorption properties.

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