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A thermogravimetric study of the adsorption of CO₂ on zeolites synthesized from fly ash

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Abstract

In the present work, the thermogravimetric characterization of the sorption of carbon dioxide on zeolites from fly ash was presented. Samples of fly ash were obtained from the Turów Power Station in Poland. Fly ash was chemically analyzed and used to prepare the zeolitic material. The synthesis processes were run using the classic alkaline conversion and the method of fusion with sodium hydroxide prior to the hydrothermal reaction. During the modification, the zeolites Na-P1 and Na-A were synthesized. The zeolites samples for dehydration were heated up to 500 °C and after stabilization of measurement conditions, sorption of CO₂ was carried out isothermally in a flow of a mixture of gasses (O₂ (10 vol.%), CO₂ (10 vol.%), N₂ (80 vol.%)). Conducted in the work research of CO₂ adsorption on zeolites synthesized from fly ash showed that these materials are characterized by high CO₂ adsorption capacity. Sample containing Na-A zeolite can take CO₂ in an amount equivalent to 5.2 wt.% sample weight within 120 min. The significant CO₂ adsorption on Na-A zeolite, results from the fact that this zeolite has large surface area (about 100 m²/g) and large pore volume (about 0.27 cm³/g). © 2005 Elsevier B.V. All rights reserved.

Keywords: Fly ash; Zeolites; Carbon dioxide; TGA

1. Introduction

In recent years, much attention has been paid to the synthesis of zeolites from fly ashes. Depending on the type of fly ash used in the synthesis, kind of the method applied, NaOH (KOH) concentration, temperature and solution/fly ash ratio different types of zeolites having different structure and efficiency can be received. Obtained on the basis of fly ash zeolitic materials are characterized by good sorption and ion exchange capacities. These characteristics are closely related to zeolites structure and they also condition existence of many potential applications obtained on the basis of fly ashes products [1–8].

Zeolites from fly ashes have a great potential for immobilization of environmental pollutants like radioactive waste, removal of heavy metals and ammonium ions from industrial sludge and removal of gaseous pollutants as SO₂ from flue gases [7,8]. Numerous potential applications of zeolites synthesized from fly ashes have not been analyzed yet. An example here may be removing CO₂ from flue gas from coalburning power plants, which is the major source of this gas emission into the atmosphere. Among currently proposed methods of CO₂ removal from flue gases there are adsorption processes, such as pressure swing adsorption (PSA) and temperature swing adsorption (TSA) [9,10]. The unique significance of adsorption methods results from the fact that adsorbents provide for the removal of practically all contaminants, at the same time purifying mixtures from several components. The sorbent can be regenerated by desorbing the CO₂ and this concentrated CO₂ can be collected for various industrial uses. The development of adsorption processes is, however, dependent on the availability of inexpensive adsorbents featuring high dynamic adsorption capacity and considerable selectivity. Data in the literature confirm that CO₂ is effectively adsorbed on zeolites [9–17]. Suggested,

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and currently tested methods of CO_2 removal with the application of these compounds make use chiefly of synthetic zeolites Type A (5A, 4A), and Type X (13X), as well as, though to a smaller degree, of natural zeolites. Research that offers more effective and less expensive adsorbents an being actively pursued. Zeolites synthesized on the basis of fly ashes seem to be promising in this aspect.

In order to be able to make use of fly ash-based zeolites for CO_2 removal from flue gases in the amount of 5–15 vol.%, CO_2 adsorption characteristics in the conditions typical for flue gases are necessary. Therefore, it is interesting to evaluate adsorption capacities of zeolitic materials, when exposed to CO_2 -containing flue gas.

The aim of the present study is to investigate the process of the adsorption of CO_2 on zeolites synthesized from fly ash with the application of thermogravimetry (TG). The influence of different operation conditions on CO_2 sorption by zeolitescontaining material was also researched in the present work.

2. Experimental

Starting material (coal fly ash) was obtained from an utility power station in Poland (CFB) circulating fluidized bed boiler of 235 MW in capacity. The fly ash was chemically analyzed and used to prepare the zeolitic materials. Classic alkaline conversion and method of fusion with sodium hydroxide prior to hydrothermal reaction were used to prepare zeolitic products. In the classic synthesis method, fly ash (20 g) was mixed with 3.0 mol/dm^3 sodium hydroxide solution (160 cm³) and heated at 100 °C for 24 h. In the method of fusion with NaOH, fly ash (10 g) and NaOH (12 g) were milled, and then heated up in a platinum crucible at 550 °C for 1 h. The mixture obtained in this manner was cooled to room temperature, milled again and replenished with distilled H₂O (100 ml). Afterwards, it was shaken for 12 h at room temperature. After such preparation, the sample was placed at 100 °C for 12 h.

After this time, the samples were filtered, washed with distilled water in order to remove the remaining NaOH, and dried. The phase structure of the prepared from fly ash products were examined by powder X-ray diffraction (XRD) analysis using a Feifert 3003 diffractometer (Cu K α radiation). The morphology of the samples was observed by using a JEOL 5400 scanning microscope. The surface area and pore structure of the samples were determined by physical adsorption of nitrogen at -196 °C in Sorptometer ASAP 2000 manufactured by Micromeritics. The samples were outgassed at 350 °C prior to the nitrogen adsorption–desorption experiments. Specific surface area was calculated from BET-isotherm [18]. For pore size distribution Barrer–Joyner–Halenda (BJH) model equation was used [19].

Carbon dioxide sorption on the zeolites prepared from fly ash and commercial Na-A zeolite was studied using a Mettler TGA/SDTA 851e thermobalance. The zeolites samples for dehydration were heated up to 500 °C at a rate of 10 °C/min and after about 40 min (stabilization of measurement conditions—cooling) sorption of CO₂ was carried out isothermally at atmospheric pressure in a flow (50 cm³/min) of a mixture of gasses (O₂ (10 vol.%), CO₂ (10 vol.%) and N₂ (80 vol.%) during 2 h.

 CO_2 sorption at three different temperatures (25, 75, 150 °C) was conducted in order to study the temperature effect on the sorption rate.

The influence of the activation temperature of zeolites on CO_2 sorption was researched for sample containing Na-A zeolite. The dehydration of zeolite sample was carried out under dynamic heating conditions from 25 to 500 °C (heating rate, 10 °C/min; nitrogen atmosphere).

After adsorbing carbon dioxide at 25 °C, desorption process was carried out at 150 °C by passing nitrogen flow (50 cm³/min) till sample weight became stable. The process of desorption was researched in order to determine the possibility of zeolites' regeneration.

In all TG analyses, the sample mass was 10 mg. For the acquisition, storage and numerical treatment of the experimental data, Mettler-Toledo Star Software was used.

3. Results and discussion

3.1. Characteristics of zeolitic products

On the basis of X-ray diffraction analysis in the modified samples of fly ash presence of well crystallized Na-A zeolite (method of fusion with NaOH) and Na-P1 zeolite (classic alkaline conversion) was observed. The contents of zeolites in the conversion products amounted to about 40% (Na-P1 zeolite)–60% (Na-A zeolite), the remaining part was residue fly ash [20].

Morphology and texture of fly ash and zeolite samples was observed by scaning electron microscope. The SEM images of original fly ash and its modifications are presented in Fig. 1. In micrograph (a) (untreated fly ash), one can see agglomerates of fine grains, some of which occur in the form of small, scattered plates, either individually shaped or arranged in layers. Also visible is a large morphological differentiation of crystallization products; grains have different shapes (from spherical to plate-like), as well as different sizes $(1-10 \ \mu m)$. Fig. 1b presents microstructures where large and small crystals of Na-P1 zeolite are visible. The emerging forms produce agglomerates, and "grow" on surface of fly ash. Furthermore, in micrograph (c) one can observe small, cubic crystals, typical of the Na-A (4A) zeolite.

In order to evaluate usefulness of the zeolites from fly ash as adsorbents, knowledge of their surface area and pore structure is very important, as it considerably influences adsorption capacity of adsorbed by zeolites compound. Surface area tests of original fly ash and the conversion products showed significant differences in both surface area and pore volume of the tested samples. The specific surface area of fly ash



Fig. 1. SEM photographs of zeolite samples: (a) untreated fly ash; (b) sample containing Na-P1 zeolite; (c) sample containing Na-A zeolite.

was determined to be $4.2 \text{ m}^2/\text{g}$. On the other hand treated fly ash containing, respectively, Na-P1 and Na-A zeolites, has considerably larger surface area: 41.9 and 96.4 m²/g, which results from the fact that these samples are a mixture of zeolites and fly ash. Surface area test results, as presented above, are quite similar to test results quoted in the literature ($42 \text{ m}^2/\text{g}$ [1], 54.4 m²/g [2], 94 m²/g [3]). The differences between the surface areas of zeolitic materials (Na-A and Na-P1) synthesized from fly ash, beside their different content in the sample, may result from differences in the structure of the formed zeolites. This is caused by the fact that surface area of zeolites depends on inner space of zeolite pores.

The conversion products, Na-A zeolite and Na-P1, zeolite beside larger surface area showed also significant increase in the pore volume and decrease in the mean pore diameter in relation to original fly ash. The greatest increase in the pore volume was observed for sample containing Na-A zeolite. Pore volume of this sample $0.27 \text{ cm}^3/\text{g}$ was about 10 times larger in relation to pore volume of original fly ash $(0.023 \text{ cm}^3/\text{g})$. In the case of the sample containing Na-A zeolite the greatest decrease in the mean pore diameter was observed. While it amounted to 21.7 nm for fly ash it reached 11.2 nm for the sample containing Na-A zeolite. In the case of the sample containing Na-P1 zeolite increase in the pore volume $(0.17 \text{ cm}^3/\text{g})$ was observed as well as decrease in the mean pore diameter (16.7 nm) in reference to original fly ash. Obtained on the basis of the research, low value of surface area and pore volume of original fly ash at its relatively large mean pore diameter indicates that there are mesopores and macropores present in its structure. On the basis of effective radius and mechanism of vapour and gases adsorption, IUPAC classification divides pores into three categories: macropores, mesopores and micropores. According to this classification mesopores are pores of 2-50 nm diameter. Mesopores beside their significant participation in adsorption, play also role of transportation pores. Pores of radius larger than 50 nm, and in consequence of small surface area constitute according to IUPAC classification macropores [21].

Fig. 2 presents curves of pore volume distribution as a function of the pore diameter for original fly ash and its two modifications. The figure shows that the largest contribution

in the total pore volume (mesopores) have pores of the following diameters: 21 nm—for fly ash, 3.5 and 8 nm—for sample containing Na-A zeolite, 8–10 and 18–25 nm—for sample containing Na-P1 zeolite. Curves (b and c) for samples containing zeolites are different from curve (a) characteristic of original fly ash. Certain number of micropores is particularly visible in the sample containing Na-A zeolite (curve c), which confirms presence of zeolite in the conversion products. Thus, in the case of zeolitic material in relation to original fly ash the contribution of smaller diameter pores in total pore volume increase.

Many conclusions concerning structure of zeolitic materials can be drawn on the basis of adsorption-desorption isotherms analysis presented in Fig. 3. The first step in physisorption isotherm interpretation is to identify isotherm type and so the nature of the adsorption process: monolayer-multilayer adsorption, capillary condensation or micropore filling [18]. The nitrogen adsorption isotherm received for the original fly ash may be classified according to IUPAC classification as Type II isotherm [18,21]. In the case of fly ash isotherm it can be observed that hysteresis loop, whose shape is connected with structure of pores and which can be classified here as H3 type, begins at relatively low pressure which indicates small contribution of micropores in this structure. Fig. 3b and c present isotherms of samples containing zeolites (Type II isotherms) where it can be observed that adsorption increases at relatively low pressure more than in original fly ash sample. This proves that samples contain certain number of micropores in relation to original fly ash. Hysteresis loops for these two samples of zeolites from fly ash can be classified similarly to fly ash as H3, while they are wider than hysteresis loop obtained for fly ash (particularly in the case of Na-A zeolite), which proves that these samples have far larger pore volume than initial fly ash.

3.2. Carbon dioxide sorption on zeolites

The use of thermogravimetric analysis in the characterization of sorption CO_2 on zeolites enabled analysis of different factors influence on carbon dioxide sorption rate.

Fig. 4 presents TG and DTG curves of CO₂ sorption on zeolitic materials at 25 °C illustrating increase of CO₂ sorption in time.



Fig. 2. Pore volume distribution as a function of the pore diameter for (a) fly ash; (b) sample containing Na-P1 zeolite; (c) sample containing Na-A zeolite.

Sample containing Na-A zeolite (curve b) can take CO_2 in an amount equivalent to 5.2 wt.% sample weight within 120 min (2 h). This is less than the sorption rate on the commercial Na-A zeolite, at the same conditions—8.7 wt.% (curve a). Sample containing Na-P1 zeolite (curve c) can take CO_2 in an amount equivalent to 3.4 wt.% sample weight, that is the least of all researched samples. High carbon dioxide sorption rate obtained for Na-A zeolite from fly ash in comparison with commercial Na-A zeolite may indicate that



Fig. 3. Nitrogen adsorption-desorption isotherms for (a) fly ash; (b) sample containing Na-P1 zeolite; (c) sample containing Na-A zeolite.

this zeolite contains well developed channels and cavities system (primary zeolite porosity) and considerable surface area $(100 \text{ m}^2/\text{g})$ and pore volume, which was proved by the research with the use of the BET method. Pore volume, playing an important role in the adsorption process, obtained for Na-A zeolite from fly ash amounted to 0.27 cm³/g, which was almost the same as pore volume of commercial Na-A zeolite $(0.3 \text{ cm}^3/\text{g})$.



Fig. 4. TG and DTG curves of sorption of CO₂ (isothermally at 25 °C) for: (a) commercial Na-A zeolites; (b) sample containing Na-A zeolites; (c) sample containing NaP1 zeolite.

It is known that, in the case of specific gas molecules adsorption on zeolites, proper choice of zeolite type is crucial. It is caused by the fact that the unique adsorptive properties of zeolites are strictly connected with their structure. First of all, it is geometrical properties of the "windows" through which a molecule must pass that decide about adsorbate molecules adsorption in the channels or cavities. Only those molecules may pass through, whose critical diameters are lower than diameters of the windows. Wider entrances to the zeolite channel network mean better conditions of CO₂ penetration to adsorptive space of zeolites. Molecules of CO2 are small, their critical diameter being about 0.31 nm, which allows them to penetrate into the primary zeolite porosity of the prevailing majority of synthetic zeolites. Cations are specific active places for CO₂ molecules. Carbon dioxide has a high linear quadrupole moment, which interacts with intrazeolitic cations [16]. The specific interactions of CO_2 quadrupole with cations of alkaline earth metals which are contained in crystalline networks of zeolites manifests themselves in this contaminant's high adsorption selectivity from worse-sorbing gaseous environment, which may be made use of for CO_2 removal from flue gases.

Obtaining Na-A zeolite as a result of fly ash modification is good from the point of view of CO_2 adsorption, as this type of zeolite is considered to be effective adsorbent for CO_2 removal (e.g. from natural gas in industrial purification processes [17,25]. The fact of a significant CO_2 adsorption on the Na-A zeolite is undoubtedly an effect of its structure. It is particularly important that this zeolite has wider channels (0.4 nm) [22,18,25]. Higher CO_2 adsorption capacity on Na-A zeolite (obtained from fly ash in the method of fusion) results from the fact that its content in the sample was much larger than Na-P1 zeolite (obtained in the method of direct conversion). Na-A zeolite synthesized from fly ash has been recognized as a very interesting zeolite (beside X zeolite) from the point of view of flue gas cleaning by Querol et al. [8].

On the basis of this data it may be concluded that it is vital to conduct fly ash modification process aiming to obtain specific type of zeolite having proper pore diameter and considerable surface area and thus high CO₂ adsorption capacity.

Very important factor determining CO_2 sorption rate on zeolites is temperature. Carbon dioxide sorption curves (TG and DTG) on fly ash-based zeolites (Na-A, Na-P1) and on commercial Na-A zeolite at three temperatures (25, 75 and 150 °C) are presented in Figs. 5–7.

As shown, for all zeolites types, sorption rate decreases as temperature increases from 25 to 150 °C. Commercial Na-A zeolite sorbed, respectively, 8.7 mg of CO₂ per 100 mg of zeolite (at 25 °C), 4.3 mg of CO₂ per 100 mg of zeolite (at 75 °C) and 1.3 mg of CO₂ per 100 mg of zeolite (at 150 °C). Sample containing Na-A zeolite can take CO₂ in an amount equiv-



Fig. 5. TG and DTG curves of sorption of CO2 on commercial Na-A zeolite, isothermally at 25, 75, 150 °C.



Fig. 6. TG and DTG curves of sorption of CO₂ on Na-A zeolite from fly ash, isothermally at 25, 75, 150 °C.

alent to 5.2 wt.% sample weight at 25 °C, 3.1 wt.% at 75 °C and 0.9 wt.% at 150 °C. For Na-P1 zeolite it was, respectively, 3.4 wt.% (25 °C), 1.5 wt.% (75 °C) and 0.2 wt.% (150 °C). On the basis of the tests results, it can be noted that together with an increase in temperature, the CO₂ adsorption capacity for all zeolite types was decreasing [22]. Therefore, temperature increase is a non-favorable phenomenon in the case of CO₂ adsorption on zeolites. Hence, in the case of CO₂ removal from flue gases on the compounds of this kind as low temperature as possible will be most desirable.

Due to the fact that zeolites reveal their sorption properties only in the dehydrated state the process of their dehydration is very important [22–24]. Sorption of other gas components including CO_2 is not possible without removal of water molecules from the zeolite structure.

During dehydration process entrances to the zeolite channel network may also undergo changes, which is vital from the point of view of gas molecules adsorption including CO₂ [22].

Dehydration process of zeolites was conducted under dynamic heating conditions for sample containing Na-A zeolite. Activation temperature of zeolitic material significantly influenced CO₂ sorption rate, which was shown in Fig. 8.

As it can be observed in the Fig. 8 Na-A zeolite from fly ash after activation at $100 \,^{\circ}\text{C}$ sorbed 2.5 mg of CO₂ per 100 mg of zeolite, after activation at $150 \,^{\circ}\text{C}$ (4.4 mg of CO₂ per 100 mg of zeolite), at 200 °C (4.9 mg of CO₂ per 100 mg of zeolite) and at 500 °C (5.2 mg of CO₂ per 100 mg of zeolite). The results show that activation temperature of zeolite is very important for CO₂ sorption process. Lower dehydration temperature (incomplete dehydration) causes lower CO₂ sorption rate in the same conditions [22]. Therefore, activation temperature is crucial in the case of using zeolitic material as carbon dioxide adsorbents. The fact that adsorption capacity of zeolites from fly ashes increases when temperature of their thermal activation increases from 200 to 400 °C was confirmed in his research by Querol et al. [8].

Conducted in the work research are also included desorption of CO₂ adsorbed on zeolites. After experiments of CO₂ sorption at 25 °C samples were heated up to 150 °C in order to determine possibility of zeolites regeneration. Fig. 9 illustrates CO₂ sorption process (dashed line) and desorption (solid line) on fly ash-based zeolites (Na-P1, Na-A) and commercial Na-A zeolite.

 CO_2 sorption on Na-A zeolites (commercial and from fly ash) was the faster in the first hour of process (TG curves illustrate this fact) and most CO_2 was sorbed at that time (in the case of Na-A commercial zeolite 5.2 mg of CO_2 per 100 mg of zeolite, Na-A zeolite from fly ash 3.9 mg of CO_2 per 100 mg of zeolite). In the next hour of the process sorption increased, respectively, by 3.5 mg of CO_2 per 100 mg of



Fig. 7. TG and DTG curves of sorption of CO₂ on Na-P1 zeolite from fly ash, isothermally at 25, 75, 150 °C.



Fig. 8. TG and DTG curves of dehydration of sample containing Na-A zeolite (dynamically) and sorption of CO_2 (isothermally at 25 °C) at different activation temperature of zeolite.



Fig. 9. TG sorption (dashed line) and desorption (solid line) of CO₂ on (a) Na-A zeolite from fly ash; (b) commercial Na-A zeolites; (c) Na-P1 zeolite from fly ash.

zeolite for Na-A commercial and 1.3 mg of CO₂ per 100 mg of zeolite for Na-A from fly ash.

CO2 desorption process had different course. Almost entire CO₂ desorption from zeolites took place in the first hour of the process and it amounted to 7.6 mg of CO₂ per 100 mg of zeolite for commercial Na-A zeolite, and for sample containing Na-A zeolite 4.9 mg of CO₂ per 100 mg of zeolite. In the next hour of the desorption process, zeolites desorbed as follows: 0.2 and 0.1 mg. Finally, Na-A commercial zeolite desorbed 7.8 mg of CO_2 per 100 mg of zeolite, and Na-A zeolite from ash 5.0 mg of CO2 per 100 mg of zeolite. The fast initial adsorption rate and the low desorption temperature support the assumption that physical adsorption predominantly occurs with little or no chemisorption [18,25]. Carried out tests show that physical sorption constitutes more than 90% of CO₂ sorption on zeolites (for commercial Na-A zeolites—90% and for Na-A zeolite from fly ash—97%) (determined on the basis of the ratio of adsorbed CO2 amount

to desorbed CO_2 amount). Desorption data suggest that zeolites may be to a large extent regenerated.

4. Conclusions

Conducted in the work research of CO₂ adsorption on zeolites synthesized from fly ash showed that these materials are characterized by high CO₂ adsorption capacity. Particularly high potential here was shown by sample containing Na-A zeolite whose CO₂ adsorption capacity turned out to be clone to Na-A (4A) commercial zeolite, which may indicate that zeolitic material is a new way of obtaining high quality adsorbents. The significant CO₂ adsorption on Na-A zeolite, obtained from fly ash by the fusion with NaOH, results from the fact that this zeolite has large surface area (about $100 \text{ m}^2/\text{g}$, while Na-P1 zeolite form fly ash—about $40 \text{ m}^2/\text{g}$) and large pore volume (about 0.27 cm³/g) which is almost

the same as the one of commercial zeolite Na-A $(0.3 \text{ cm}^3/\text{g})$. Thus, Na-A zeolite from fly ash shows high CO₂ adsorption capacity as compared to commercial Na-A zeolite synthesized in the classical ways from chemically pure components, which confirmed that zeolitic material has a well-developed system of channels and cavities. This zeolite may prove to be a very good adsorbent for purifying flue gases, especially because physic sorption constitutes more than 90% of CO₂ sorption process, which indicates possibility of regeneration of zeolitic material. Thermogravimetric studies show that CO₂ sorption process is considerably influenced by temperature. Equally important for CO₂ sorption on zeolites is their activation temperature. Higher activation temperature and its proper duration enable to obtain higher level of zeolite dehydration, and consequently higher CO₂ adsorption capacity. Conducted research of CO₂ desorption process shows that the CO₂ was largely physically adsorbed on the surface of the fly ash-based zeolites. Flue gases, apart from their main components, contain also water vapor, which would influence the adsorption behavior of CO₂. The concentration of water vapor in the flue gas must be considerably lowered when the adsorption process of carbon dioxide is operated H2O. One of the possible solutions may also be to run the adsorption process in two stages: water steam adsorption at stage one, and CO₂ adsorption at stage two.

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