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THE UTILISATION OF FLY ASH IN CO_2 MINERAL CARBONATION

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Dedicated to Prof. Ryszard Pohorecki on the occasion of his 80th birthday

The fixation of CO_2 in the form of inorganic carbonates, also known as mineral carbonation, is an interesting option for the removal of carbon dioxide from various gas streams. The captured CO_2 is reacted with metal-oxide bearing materials, usually naturally occurring minerals. The alkaline industrial waste, such as fly ash can also be considered as a source of calcium or magnesium. In the present study the solubility of fly ash from conventional pulverised hard coal fired boilers, with and without desulphurisation products, and fly ash from lignite fluidised bed combustion, generated by Polish power stations was analysed. The principal objective was to assess the potential of fly ash used as a reactant in the process of mineral carbonation. Experiments were done in a 1 dm^3 reactor equipped with a heating jacket and a stirrer. The rate of dissolution in water and in acid solutions was measured at various temperatures (20 – 80ºC), waste-to-solvent ratios (1:100 – 1:4) and stirrer speeds (300 – 1100 min^{-1}). Results clearly show that fluidised lignite fly ash has the highest potential for carbonation due to its high content of free CaO and fast kinetics of dissolution, and can be employed in mineral carbonation of CO_2.

Keywords: mineral carbonation, sequestration of CO_2, industrial waste, fly ash, dissolution

1. INTRODUCTION

It is believed that climate change observed in recent years has been caused primarily by CO_2 generated from human activity, mainly by burning of fossil fuels (IPCC, 2014). In Poland the production of energy is largely based on coal, thus contributing considerably to a rising CO_2 atmospheric concentration. All the forecasts show that the global energy consumption will be increasing, while in Poland coal will remain the primary source of energy. Therefore, various techniques for CO_2 sequestration have to be further developed.

The process of mineral carbonation is regarded as one of a number of options for the capture and storage of CO_2 (Huijgen et al., 2003; Huijgen et al., 2005; Metz et al., 2005). Carbonation employs a reaction of carbon dioxide with metal oxides (usually those of Ca and Mg), and mimics the naturally occurring process of rock weathering. Following the reaction, geologically stable and environmentally neutral carbonates are formed which may then be stored or used. The idea of using mineral carbonation to bind CO_2 was first proposed by Seifritz (1990). The first detailed study was made by Lackner et al. (1995). A major advantage of this method is that the resulting product is thermodynamically stable. It is therefore possible to store it permanently with minimal environmental impact and without the possibility of CO_2 release from the disposal site. The product can also be utilised (e.g. as a road base or construction material). The drawbacks of mineral carbonation are due to slow kinetics (especially under ambient conditions), large amounts of minerals that have to be employed and high cost, which severely limit the application of this process on a large scale. A natural source of alkali metal oxides are

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*Corresponding author, e-mail: jjaschik@iich.gliwice.pl minerals containing silicates of calcium and magnesium, such as wollastonite, serpentine and talc. The necessary minerals are available in amounts far exceeding the known fossil fuel reserves (Lackner et al., 1995; Sanna et al., 2014). Commonly, two options of mineral carbonation are taken into account: *in situ*, in which the process is carried out underground (in a gas-solid system), and *ex-situ*, where the carbonation occurs in overground installations. In the latter case minerals have to be mined and processed (ground and calcined), and the carbonation product has to be transported to a suitable storage site; this obviously increases the cost of the process.

The simplest approach to mineral carbonation would be to react gaseous CO_2 with a metal-oxide bearing phase. Unfortunately, such direct gas-solid reactions are too slow to be practical. Moreover, it is likely to require a higher pressure, temperature and/or suitable pretreatment (Lackner, 1997). In order to conduct carbonation at rates that may enable practical implementation of the process, the extraction of a metal from the solid is required. This can be done by suspending the solid material in an aqueous solution (in overground industrial reactors) and letting it dissolve and release metal ions. Dissolution may be the rate-limiting step for the overall carbonation process and thus requires careful analysis.

One of the ways to lower the cost of carbonation process is the use of alkaline industrial waste (Huijgen et al., 2005a), such as fly and bottom ash, concrete waste and slag, residues from municipal waste combustion or steel production. Waste commonly occurs in a pulverised form and thus does not require additional mechanical processing. Calcium and/or magnesium oxides are usually unbounded and their release to the solution is much faster than that of natural minerals. The industrial waste does not have to be mined and, moreover, a relevant installation can be placed close to a waste source, thus lowering the overall cost. Additionally, hazardous waste can be deactivated through pH-neutralisation and mineral transformation. On the other hand, the storage capacity of industrial waste is limited and dependent on developments of technology. The process of neutralising CO_2 with industrial waste has been presented in literature (Back et al., 2008; Bauer et al., 2011; Mun and Cho, 2013; Sanna et al., 2014; Stolaroff et al., 2005; Uibu and Kuusik, 2009). Possibilities of using domestic fly ashes were also examined (Jaschik et al., 2013; Uliasz-Bochenczyk, 2011; Uliasz-Bocheńczyk et al., 2012). The studies presented so far lead to a clear conclusion that industrial residues have a potential for quick CO_2 binding with acceptable yield under ambient conditions. The total cost of carbonation using industrial waste is generally lower than that for the sequestration based on natural minerals, but is still unacceptably high. The comparison of costs for different methods of CO_2 storage is presented in Table 1. Currently, research is focused on lowering the overall cost and maximising the storage of CO_2 by optimising the operating conditions (Sanna et al., 2014).

Table 1. Costs of storage in USD per tonne of CO_2 avoided

Geological storage[1]	0.5 – 8
Ocean storage[1]	5 – 30
Mineral carbonation:	
natural minerals[1]	50 – 100
cement, concrete residue[2]	22 – 35
steel slag, concrete waste[3]	8

[1]Metz et al., (2005); [2]Huijgen et al., (2005a); [3]Stolaroff et al. (2005)

In this work the potential of fly ashes from Polish power plants for binding CO_2 has been examined. The aqueous route was chosen because of its markedly higher reaction rates compared to the gas-solid system. Hence our experimental study focused on the kinetics of dissolution as the rate-limiting step for the aqueous carbonation process.

2. SAMPLE CHARACTERISATION

Three types of ash were used: fly ash from conventional pulverised hard coal fired boilers (PC), fly ash from pulverised coal fired boilers with desulphurisation products (PCD), and fly ash from lignite fluidised bed combustion (FBC). PCD ash was collected from flue gas desulphurisation plants based on the dry flue gas desulphurisation (FGD) method with wetting. The chemical composition and phase composition of the samples were determined using a Thermo iCAP 6500 Duo ICP spectrometer (Thermo Fisher Scientific) and an Empyrean X-ray diffractometer (PANalytical), respectively. Particle size distribution was measured by a Mastersizer 2000 laser particle size analyser (Malvern Instruments) using 2-propanol as a dispersant. BET surface area as well as total and micropore volumes were determined using ASAP 2020 physisorption analyser (Micromeritics).

The main characteristics of the ashes are presented in Tables 2 and 3. A chemical analysis showed that FBC ash contained 29.1 wt. % of calcium (expressed in terms of CaO), i.e. most of all the ashes studied. Approximately 41.8 wt. % of calcium in FBC ash occurs as free oxides, whereas magnesium is present solely as a component of the vitreous phase. The content of free calcium oxide in FBC ash, 12 wt. %, is also the highest among all the waste studied. The content of free magnesium oxide is generally low in all the ashes.

Table 2. Chemical and phase composition of the ashes

	Chemical composition, wt. %				Phase composition, wt. %		
ash	PC	PCD	FBC	ash	PC	PCD	FBC
SiO_2	50.8	42.8	27.0	Quartz SiO_2	6.9	5.3	1.9
CaO	2.97	13.9	29.1	Lime CaO	0.4	1.7	12.0
MgO	2.52	2.18	2.02	Portlandite $Ca(OH)_2$	-	0.5	0.2
Al_2O_3	25.2	21.0	20.2	Anhydrite $CaSO_4$	-	3.5	12.4
Fe_2O_3	6.08	5.15	4.54	Calcite $CaCO_3$	-	2.7	6.4
Na_2O	1.06	0.93	1.27	Periclase MgO	1.0	0.9	-
K_2O	3.35	2.64	1.01	Mullite $3Al_2O_3{\cdot}2SiO_2$	12.9	8.8	-
SO_3	0.32	2.94	8.75	Anorthite[a] /Albite[b]	-	1.7[a]	1.5[b]
P_2O_5	0.74	0.53	0.20	Hematite α-Fe_2O_3	0.6	0.8	1.6
TiO_2	1.03	0.86	1.68	Magnetite Fe_3O_4	1.4	0.9	-
C (TOC)	5.04	3.87	0.14	Amorphous	76.8	73.1	63.0

Table 3. Particle size distribution, surface and porosity of the ashes

	Particle size distribution, $m \cdot 10^{-6}$				Surface and porosity		
ash	PC	PCD	FBC	ash	PC	PCD	FBC
$D_{0.1}$	5.50	4.53	3.96	BET, m^2/g	1.78	4.35	6.66
$D_{0.5}$	56.1	41.8	24.6	Micropore area, m^2/g	0.33	1.04	0.59
$D_{0.9}$	237.4	170.9	87.4	Total pore vol., mm^3/g	0.58	0.57	3.71
D_{32}	13.0	10.7	8.94	Micropore vol., mm^3/g	0.018	0.055	0.026
D_{43}	97.8	68.0	36.8	Aver. pore size, $m \cdot 10^{-9}$	6.67	4.35	14.24

A laser diffraction analysis found that the median particle size was between 25 and 60 μm, and the size of the largest particles did not exceed 250 μm. Thus, although the three types of dust were finely comminuted, the FBC ash was the finest. The BET surface area was also the highest for the lignite fluidised ash. However, due to the small porosity of ashes, the values of BET surface are regarded as approximate.

The morphology of ash particles was examined using scanning electron microscopy (SEM). Their characteristic shape and texture are shown in Fig. 1. As can be seen, ash particles from fluidised bed boilers (Fig. 1c) have an irregular shape, porous and uneven surface, while particles from conventional pulverised coal fired boilers (both with and without desulphurisation products, Figs. 1a, 1b), have a regular spherical shape and smooth surface, caused by the formation of the glassy phase.

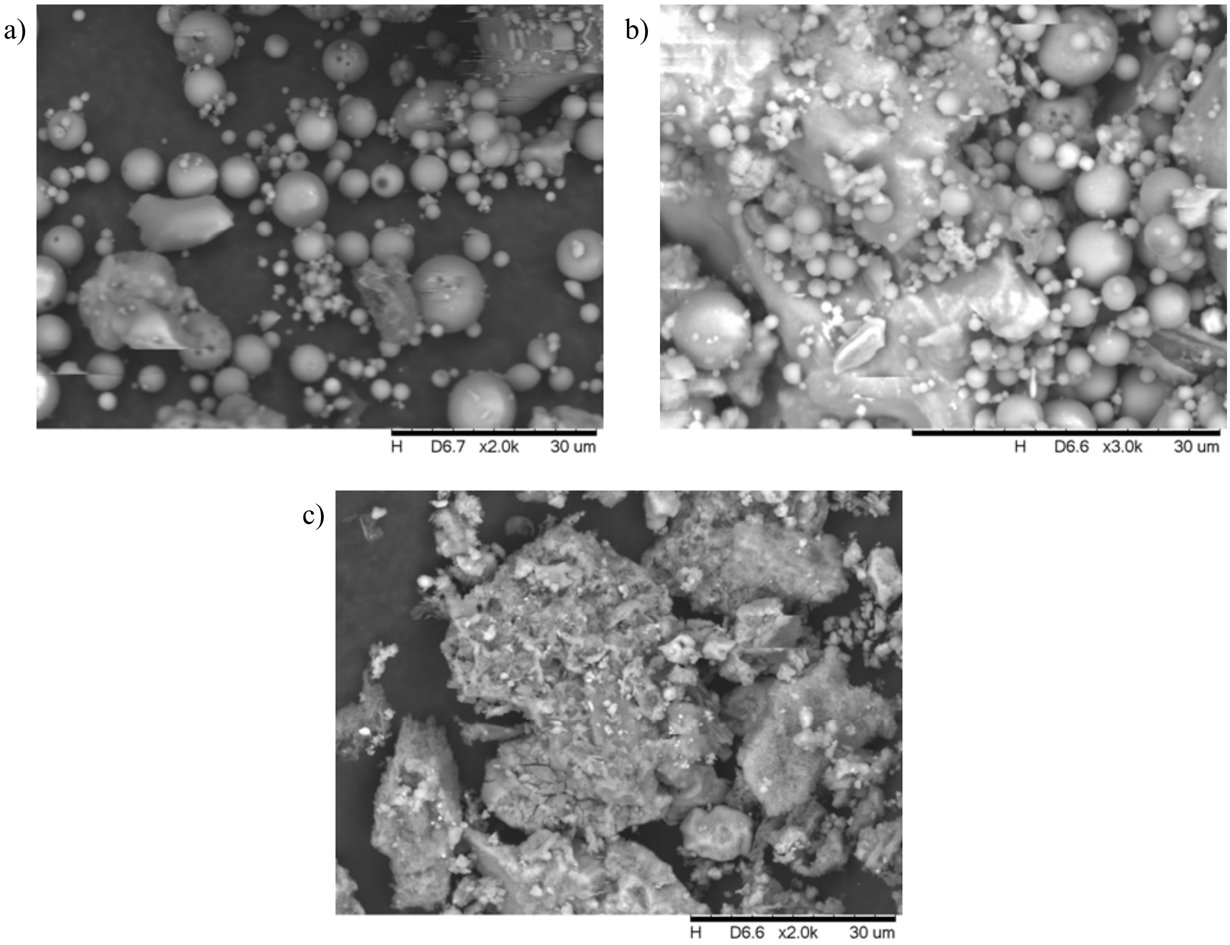

Fig. 1. SEM images of ash particles; a) – pulverised hard coal fire boilers (PC), b) – pulverised coal fire boilers with desulphurisation products (PCD), c) – lignite fluidised bed combustion (FBC)

3. EXPERIMENTAL CONDITIONS

All experiments concerning the dissolution of fly ash were conducted in a laboratory installation that included a 1 dm^3 reactor with a heating jacket. The reactor was made of borosilicate glass (QVF/Normag), and had a draft tube with 4 baffles and a propeller mixer. A schematic diagram of the experimental setup is shown in Fig. 2.

The reactor worked periodically. Ash suspensions with a solvent (water or acid solution) of a given solid-to-liquid mass ratio were mixed in the reactor at a fixed temperature and stirrer speed. During a single experimental run suspension samples (around 20 cm^3 each) were withdrawn using a pump. The suspension was immediately filtered, then the concentrations of Ca^{+2}, Mg^{+2} and SO_4^{-2} ions were determined in the filtrate by complexometric titration using EDTA (Ca^{+2}, Mg^{+2}) and by a spectrophotometer DR2800 using Hach Lange cuvette tests (Mg^{+2}, SO_4^{-2}). These concentrations made it possible to determine the extraction degree of calcium and magnesium (defined as the ratio of an ion concentration in the solution to a hypothetical maximum concentration after complete dissolution of the fly ash).

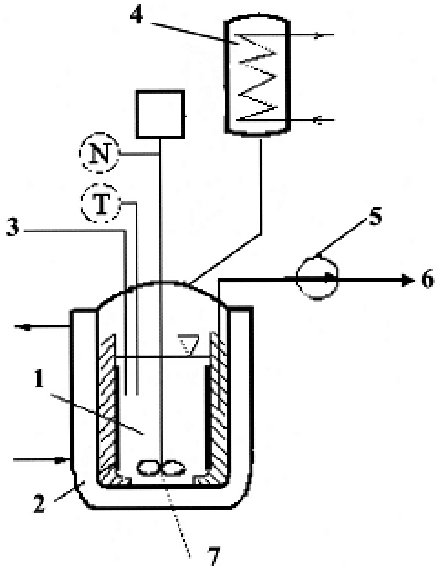

Fig. 2. Schematic diagram of the experimental setup;
1 – reactor, 2 – heating jacket, 3 – inlet of solution and solid phase, 4 – cooler, 5 – peristaltic pump,
6 – sample withdrawal, 7 – mixer, T – temperature control, N – mixer speed control

Both the temperature of the suspension and the mixer speed were controlled during the experiments. The reactor was equipped with a cooler to prevent solvent losses due to evaporation. Dissolution was carried out for about 4.5 - 5 hours. At the end of an individual measurement the reactor was emptied and its contents filtered and analysed.

The rate of dissolution was measured in water and in different acid solutions under atmospheric pressure, over a range of temperatures (20 - 80°C), for the weight ratio of the fly ash to the solvent varied between 1:100 and 1:4 and for a stirrer speed of 300 to 1,100 min^{-1}.

4. RESULTS AND DISCUSSION

Based on the experiments of dissolution of PC and PCD ashes in water it is concluded that the degree of calcium extraction from PCD waste depends only slightly upon the temperature and stirrer speed, as shown in Figs. 3 and 4. On the other hand, the effect of the amount of waste on both Ca^{2+} concentration and the degree of extraction was quite well marked, and reached 52% and 26% in 20ºC for the ash-to-water ratio equal to 1:100 and 1:10, respectively. PCD waste dissolution rate rapidly dropped after 30 minutes, and the equilibrium was reached after about an hour. The concentration of Mg^{+2} ions in water after dissolution was very low, so it seems that the magnesium extraction from PC and PCD ashes to water is of no practical significance.

Only a few experimental runs were made for the dissolution of PC ash in water. Because of the low content of calcium oxide, slow dissolution kinetics and low extraction, experiments were made only for high ash-to-water ratios. Even for the highest waste-to-solvent ratio (1:4) and the highest temperature used in the experiments (80ºC), after five hours the concentration of Ca^{2+} in the solution remained very low (0.35 g·dm^{-3}, extraction ratio: 6.6%). The effect of ash-to-water ratio on the concentration of Ca^{+2} during the dissolution of PC and PCD ashes in water is presented in Fig. 5.

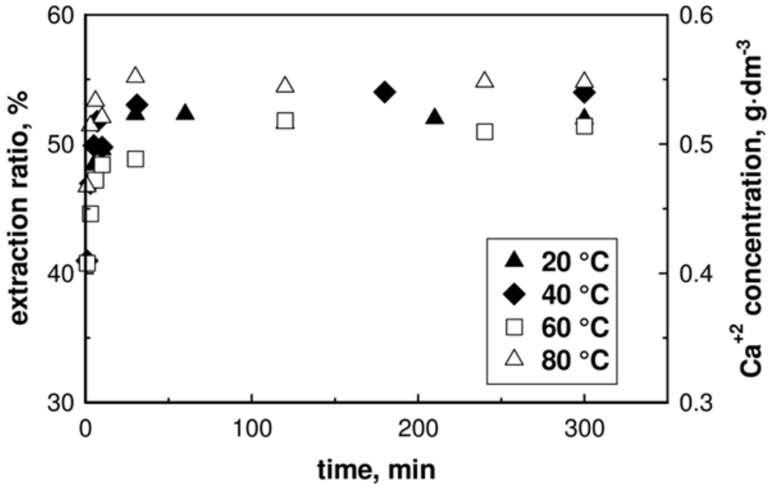

Fig. 3. The effect of temperature on Ca extraction from PCD ash during dissolution in water; stirrer speed = 600 min^{-1}, ash-to-water ratio = 1:100

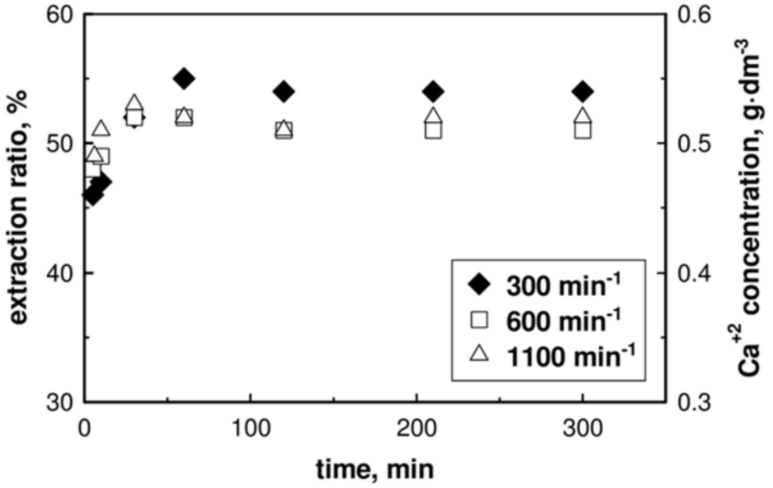

Fig. 4. The effect of stirrer speed on Ca extraction from PCD ash during dissolution in water; temperature = 20ºC, ash-to-water ratio = 1:100

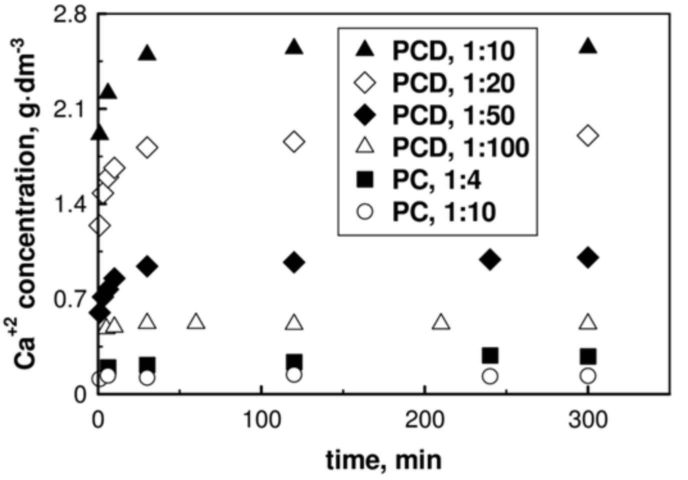

Fig. 5. The effect of ash-to-water ratio on Ca extraction during dissolution of PCD and PC ashes in water; temperature = 20ºC, stirrer speed = 600 min^{-1}

The extraction of alkali metals from industrial waste may be facilitated by the presence of acids in aqueous solutions, similarly as in the case of natural minerals (Huijgen et al., 2005; Sanna et al., 2014). The effect of acid concentration on the kinetics of dissolution was studied using PCD ash (with acetic acid and citric acid) and PC ash (acetic acid). Results for PCD ash are presented in Fig. 6.

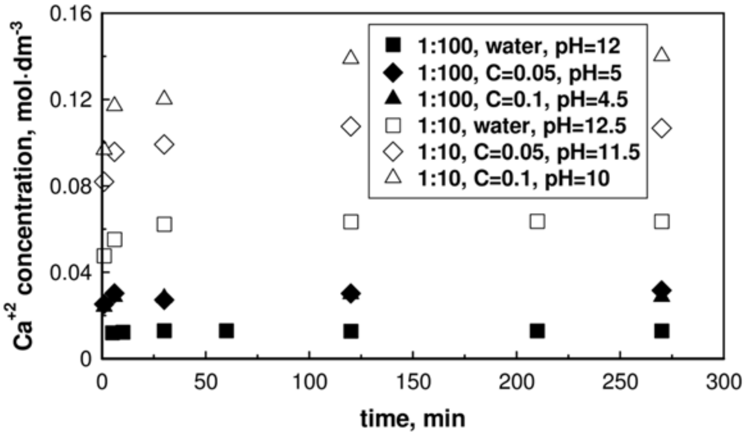

Fig. 6. The effect of acid concentration on Ca^{+2} concentration during dissolution of PCD ash in acetic acid solutions; temperature = 20°C, stirrer speed = 600 min^{-1}

As can be seen from Fig. 6, even a small amount of acetic acid ($C = 0.05$ mol·dm^{-3}) was sufficient to increase considerably the solubility of ash compared with its solubility in water. For the ash-to-solvent ratio equal to 1:100, the concentration of calcium ions after 4.5 hour dissolution increased more than twice, from 0.012 mol·dm^{-3} in water to 0.029 mol·dm^{-3} in 0.05 M solution of acetic acid, and almost two times for the ash-to-solvent ratio of 1:10 (0.064 mol·dm^{-3} in water, 0.107 mol·dm^{-3} in 0.5 M solution). A further increase of the acetic acid concentration did not produce such an increase in solubility. Similar results were obtained with citric acid solutions, until a supersaturated solution of calcium citrate was reached and the solid crystallised. This happened for a high content of ash or citric acid.

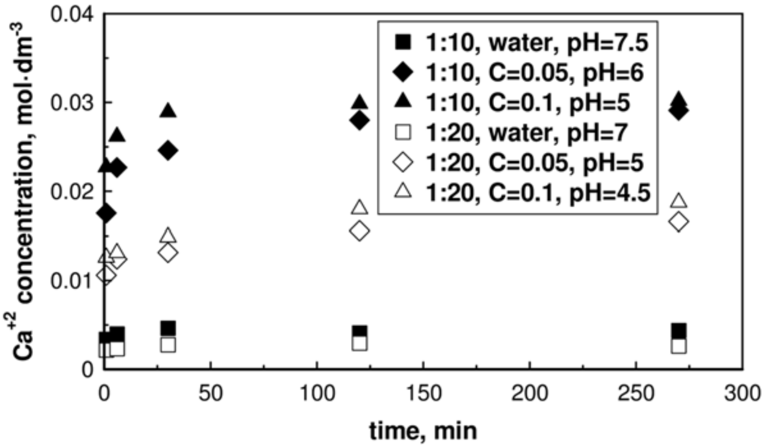

Fig. 7. The effect of acid concentration on Ca^{+2} concentration during dissolution of PC ash in acetic acid solutions; temperature = 20°C, stirrer speed = 600 min^{-1}

The influence of acid on the solubility was even greater in the case of PC ash, as can be seen from Fig. 7. For the ash-to-solvent ratio equal to 1:20, using an acetic acid solution as a solvent caused an increase in Ca^{+2} concentration from 0.0026 mol·dm^{-3} (water) to 0.017 mol·dm^{-3} (0.05 M solution) and 0.019 mol·dm^{-3} (0.1 M solution). These values correspond to the extraction degrees of 10%, 62.6% and 70.7%; for the ratio of 1:10 the concentration of Ca^{+2} after dissolution was 0.0044 mol·dm^{-3} (water), 0.029 mol·dm^{-3} (0.05 M solution) and 0.030 mol·dm^{-3} (0.1 M solution), and the corresponding values of the extraction degree were 8.3%, 54.9% and 57.1%. Even though the acid considerably improved the extraction of calcium from PC ash, the concentration of calcium ions in the solution was still much lower than that of PCD ash. This was quite obvious as PCD ash contains much more calcium oxide than PC ash does. Moreover, high extraction degree of PC ash was obtained only using an acid solution as the solvent, which resulted in a significant drop in pH level. For PCD ash, when acetic acid was used, the final solution was still alkaline for a large content of ash, as opposed to solutions obtained after the dissolution of PC ash. In all the experiments with acid solutions, the dissolution equilibrium was reached after about an hour, similarly as in the case of water, but pH levels of final solutions were obviously lower than those for dissolution in water. This decrease in pH will hinder the absorption of CO_2 in the next step of carbonation.

The rate of FBC ash dissolution was measured only in water, because in all the cases high extraction degrees of calcium or saturated solutions were obtained. It was therefore concluded that there was no need to use acid solutions as solvents. The fast kinetic dissolution of FBC ash is due to a high content of free calcium oxide and structural characteristics of particles, conducive to dissolution process. Similarly to the previous cases the content of magnesium oxide in FBC ash was low, and the amount of magnesium released into the solution was probably also too low to have any measurable impact on carbonation. Because of the high content of calcium sulphate, special attention was paid to the sulphate extraction from the ash to water.

The studies of FBC ash dissolution reveal that the content of Ca^{+2} as well as that of SO_4^{-2} ions in the filtrate obtained after the dissolution of ash is almost independent of the stirrer speed (Fig. 8), decreases with temperature (Fig. 9) and increases with the rise in the ash-to-solvent ratio (Fig. 10), until it reaches the saturation level at a ratio of 1:20. A further increase in this ratio does not produce any increase in the Ca^{+2} and SO_4^{-2} content, as the state of saturation has already been attained. Simultaneously, the extraction degree of calcium drops with an increase in the ash-to-solvent ratio from around 64% (at a ratio of 1:100) to 10.5% for a ratio of 1:10 (both for 20°C). The extraction degree of sulphate drops in the same cases from around 69.5% (1:100) to 16.7% (at a ratio of 1:10).

In all the cases the equilibrium of Ca^{+2} ions was established after about 30 minutes, whereas for the dissolution of calcium sulphate the time necessary to reach the equilibrium was as long as 2 hours. As can be seen, the rate of dissolution of calcium sulphate was slower than that of calcium oxide, but after 4.5 hours of dissolution the final degree of extraction of sulphate ions was larger than that of calcium ions. Obviously the concentration of SO_4^{-2} was always lower than Ca^{+2} content. In all the cases the pH of the solutions obtained after 4.5 hours of dissolution was about 12.5. However, after one minute the pH of filtrate was at least 12. It is thus obvious that from the moment dissolution started leachates were highly alkaline.

If mineral carbonation proceeds in an aqueous solution, two routes can be distinguished (Huijgen et al., 2005; Sanna et al., 2014: a) direct scheme - the extraction of reactive components from the ash and the precipitation of carbonates take place simultaneously in the same reactor, and b) indirect scheme - the extraction of reactive components from ash and the precipitation of carbonates take place in two separate steps (reactors).

When FBC ash is used in the direct aqueous route, sulphate ions will be present in the suspension. Sulphate ions, alongside carbonate ions (created in the reaction of H_2O with CO_2) will produce insoluble layers of $CaSO_4$ and $CaCO_3$, which will probably coat the surface of ash particles, thus preventing further dissolution of calcium oxide, as observed in the experiments reported elsewhere (Huijgen et al., 2005; Uibu and Kuusik, 2009). Therefore, the content of SO_4^{-2} and CO_3^{2} ions in the liquid phase has to be closely monitored, as they lead to the formation of insoluble layers on the surface of particles and thereby strongly inhibit dissolution. The coating of precipitated solids building on ash particles makes recycling of unconverted feedstock impossible.

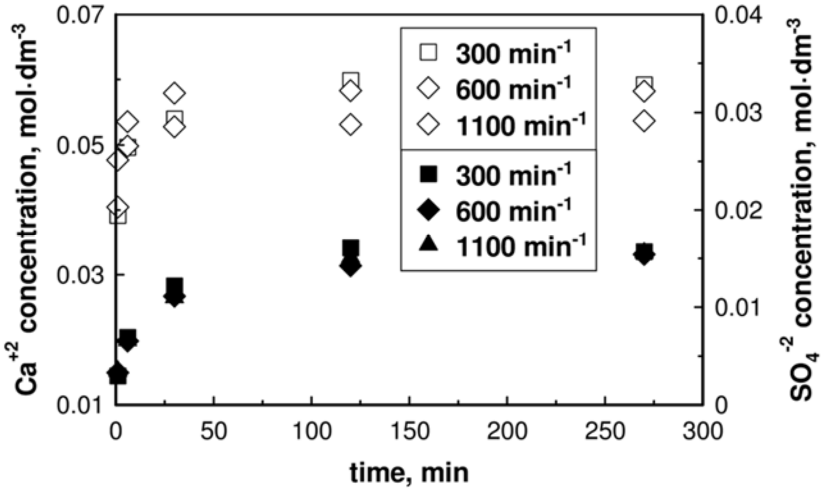

Fig. 8. The effect of stirrer speed on Ca^{+2} (empty symbols) and SO_4^{-2} (solid symbols) concentration during dissolution of FBC ash in water; temperature = 20°C, ash-to-water ratio = 1:20

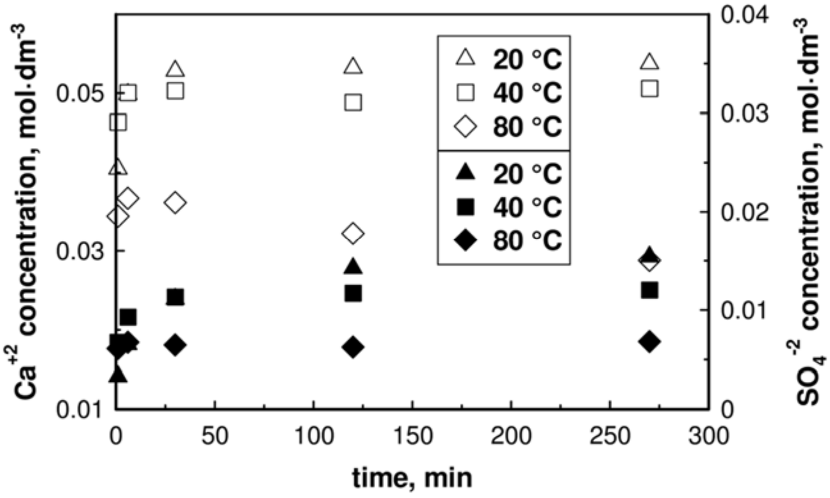

Fig. 9. The effect of temperature on Ca^{+2} (empty symbols) and SO_4^{-2} (solid symbols) concentration during dissolution of FBC ash in water; stirrer speed = 600 min^{-1}, ash-to-water ratio = 1:20

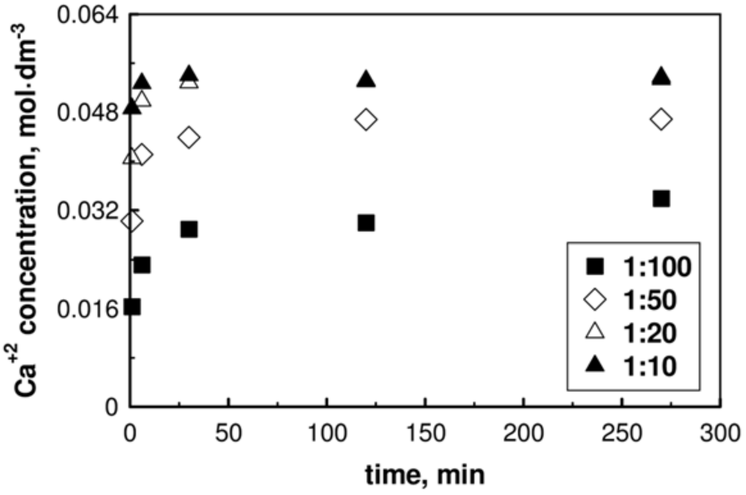

Fig. 10. The effect of ash-to-water ratio on Ca^{+2} concentration during dissolution of FBC ash in water; stirrer speed = 600 min^{-1}, temperature = 20ºC

When FBC ash is used in an indirect process, an alkaline solution containing mainly calcium and sulphate ions will be obtained after the first step and directed to the next reactor. However, in this case the sulphate ions, while not hindering the dissolution of ash, will affect the precipitation of calcite (Huijgen et al., 2005a). Moreover, the final product of carbonation, calcium carbonate (PCC), has a high commercial value. This approach is thus advantageous and is proposed for the future study of carbonation process using ash from lignite fluidised bed combustion.

5. INDIRECT AQUEOUS CARBONATION PROCESS

A few preliminary measurements of CO_2 absorption in a solution obtained after the dissolution of FBC ash in water were made. The idea of the two-step aqueous carbonation process using fly ash is shown in Fig. 11.

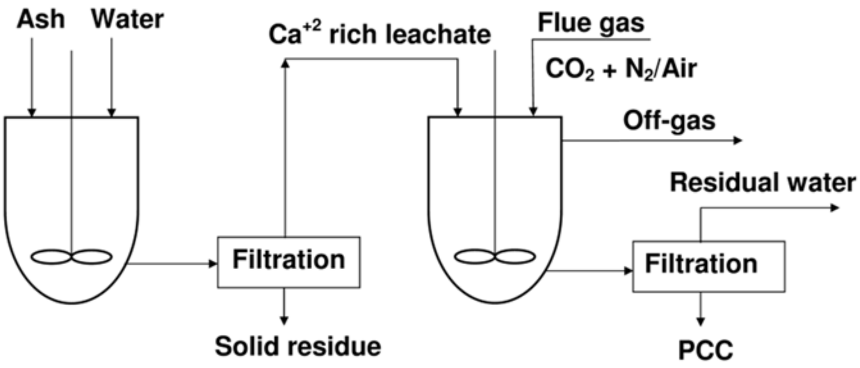

Fig. 11. Scheme of indirect aqueous carbonation

The solution directed to the second reactor was obtained after 30 min. dissolution of FBC ash in water under ambient conditions with an ash-to-solvent ratio of 1:20. The leachate contained 0.0535 $mol \cdot dm^{-3}$ of Ca^{+2} and 0.0131 $mol \cdot dm^{-3}$ of SO_4^{-2}. The inlet gas stream was a mixture of nitrogen and carbon dioxide, with about 15% of CO_2 (corresponding to its content in the flue gas). The absorption process was conducted for 5 minutes. After this time a 50% conversion of calcium was attained, and almost 100% of carbon dioxide supplied to the reactor was captured. The solid phase contained only calcium carbonate, and no crystallisation of calcium sulphate was observed. The median size of $CaCO_3$ particles was about 12 μm.

Studies on CO_2 absorption and the precipitation of calcium carbonate in mineral carbonation are underway.

6. CONCLUSIONS

The results clearly show that the studied fluidised lignite fly ash has the highest potential for carbonation and can be employed in CO_2 capture from flue gas. The high content of calcium oxide and the morphology of ash particles lead, over a short period of time, to alkaline solutions saturated with calcium ions of pH of about 12.5. Therefore, the next step of carbonation with the presence of carbon dioxide should occur with a sufficiently high yield. The fly ash from pulverised hard coal fired boilers (PC) due to a much lower content of CaO and slow dissolution kinetics has to be ruled out as a candidate for the fixation of CO_2. The fly ash containing desulphurisation waste (PCD) has also potential to capture CO_2, but its use requires an acid to be used as a solvent, leading to a decrease in pH and adversely affecting the absorption of CO_2 in the next step of carbonation.

SYMBOLS

C	concentration, mol·dm^{-3}
D_{32}	Sauter mean diameter, m
D_{43}	de Brouckere mean diameter, m

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