Chapter 44
Flue Gas CO_2 Sequestration by Turkish Coal Fly Ashes and Anatolian Geothermal Hot Waters

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Introduction

The increasing CO_2 concentration in the Earth's atmosphere, mainly caused by fossil fuel combustion, has led to concerns about global warming. A technology that could possibly contribute to reducing carbon dioxide emissions is the in-situ mineral sequestration (long term geological storage) or the ex-situ mineral sequestration (controlled industrial reactors) of CO_2.

Carbonation of CO_2 gas has many various advantages. Most distinct fact is that carbonates have a lower energy state than CO_2. Mineral carbonation is thermodynamically occurs in nature (i.e., the weathering of rock over geologic time periods). Further, the raw materials such as magnesium based minerals are abundant on earth. The produced carbonates are basely stable and thus re-evolve of CO_2 into the atmosphere is not an issue. However, conventional carbonation reactions are slow under ambient temperatures and pressures.

Forty percent of global electricity is generated in fossil fuel power plants per annum, with emissions of about 33 % (9.5 billion metric tons) of global energy related CO_2 emissions of approximately 28.8 Gt in 2010 (Minchener and McMullan 2007; IEA 2012). Over half of the electricity demand of the Turkey is supplied by coal-fired power plants, with emissions of about 16 million metric tons of CO_2 per annum, representing about 33 % of Turkey energy-related CO_2 emissions (TKI 2009; TTK 2009).

Therefore, developing effective CO_2 sequestration is one of the critical components in addressing global climate change. Note that improving the efficiency of energy production and utilization, and developing renewable energy sources will certainly play a very important role in reducing CO_2 emissions (5), however these

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measures alone cannot address the greenhouse emissions issue mainly because world energy consumption will increase significantly as the living standard improves in many parts of the world.

Three boreholes, one injection well, and two observation wells will be drilled, about 50–100 m (160–330 ft) apart, into the flank of an anticlinal structure. A total of up to 1 t/day of CO_2, in gaseous state at the well head, will be injected at about 700 m (2300 ft) depth into a saline mudstone aquifer. Injection is planned to start at the end of 2013 and last for approximately 2 years, during which the distribution and fate of the injected gas will be monitored by surface geophysical surveys and by downhole instrumentation. Various processes and subprojects and work packages address the science and operational aspects of CO_2 (IPCC 2005; Stangeland 2007; Solomon 2006; O'conner et al. 2000; Siegenthaler and Oeschger 1987; Keeling et al. 1995; Plasynski et al. 1999). The proposed part of the project is restricted to the baseline survey and the injection and basic monitoring of CO_2. The supply of CO_2 will be provided by near Coal Power Station in Şırnak. Other choice will be the purchase of a pure CO_2 originating from flue gas of hydrocarbon production at an oil refinery. The CO_2 will be delivered by truck in liquid phase, temporarily stored at the well site, and conditioned before injection. The project proposed plans to inject the CO_2 at the well head in gaseous phase at a slightly supercritical pressure and slightly supercritical temperature. The anticline at Şirnak-Silopi was used for gas storage in the past in a shallower depth interval in Şırnak-Silopi pit mine field, which also is an mining industry partner in CO_2 sequestration and will be responsible for the CO_2-injection operations.

This paper discussed progress on reactor achieved by tests and search for fast reaction methods using exhaust gas containing waste sulfur and carbon gases at the stack of Power Stations. This experimental study demonstrates that 1 ton of fly-ash could sequester up to near 20 kg of CO_2, i.e. 50 ton of fly ash per ton of CO_2 sequestered. This confirms the possibility to use this alkaline residue for CO_2 mitigation. Other alkaline sources containing calcium, magnesium and magnesium salts, supercritical CO_2, water slurry, and additives were searched for optimum sequestration methods and also in order to enhance mineral reactivity; and in analyzing the structural changes to identify reaction paths and potential barriers. Carbonation liquid and gaseous products may change to near 20 %–45 % yield performances by time increase from 1 to 6 h.

CO_2 Sequestration and Carbonation

Options for storing CO_2 in deep underground geological formations need adequate porosity and thickness for storage capacity and permeability for gas injection are critical. The storage formation should be capped by extensive confining units such as shale, salt caves or anhydrite beds to ensure that CO_2 does not escape into overlying, shallower rock units and ultimately to the surface. Geological storage of CO_2 requires compression of CO_2 to allow injection. This is done by compressing

the CO_2 to a dense fluid state known as 'supercritical'. This supercritical state is achieved by exposing the CO_2 to temperatures higher than 31.1 °C and pressure greater than 73.9 bars. The density of CO_2 will increase with depth, until about 800 m or greater, where the injected CO_2 will be in a dense supercritical state (DOE 1998; Simbeck 2004; Burruss 2004; Oldenburg et al., 2001).

The mineral carbonation, a process of converting CO_2 into stable minerals—mineralization has been studied extensively to capture and store CO_2 (Stevens et al. 2001). However, most of the mineral carbonation studies have been largely investigated at lab scale. Preliminary and pilot scale studies for accelerated mineral carbonation (AMC) were conducted at one of the largest coal-fired power plants (2120 MW) in the USA by reacting flue gas with fly ash particles in a fluidized bed reactor. In the preliminary experiments, flue gas CO_2 and SO_2 concentrations decreased from 13.0 % to 9.6 % and from 107.8 to 15.1 ppm, respectively, during the first 2 min of reaction (Stevens et al. 1999). The flue gas treated by fly ash particles, even mineralization hold high mercury (Hg) concentration of 0.22 mg/kg in flue gas (Myer 2003; Zweigel et al. 2001).

Geothermal brine waters utilization and using CO_2 as feed material in mineral carbonation produce various environmentally benign products. However, many challenges to any solution include technical feasibility, economic viability, environmental soundness and long term sustainability for mineralization of CO_2 (Pacala 2003; Hepple and Benson 2003).

Method of Mineral Sequestration

Mineral sequestration involves the reaction of CO_2 with minerals to form geologically stable carbonates. This mineralization of CO_2 was searched by different studies using various materials (Lindeberg 2003; Rubin and Rao 2003). General and specific global mineral carbonation reaction pathway is shown in Fig. 44.1. The goals of the current work were to design an ash–water suspension carbonation process in a continuous mode laboratory-scale plant and to search for potential means of intensifying the water neutralization process (Davis 2004; Seifritz 1990; Kojima et al. 1997; Gunter et al. 1993; Lackner et al. 1995). The carbonation process was optimized by cascading columns in which the pH progressed from
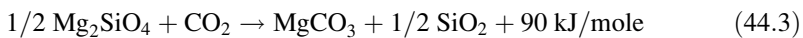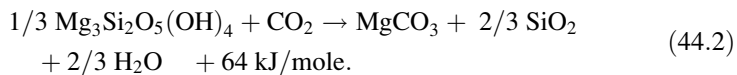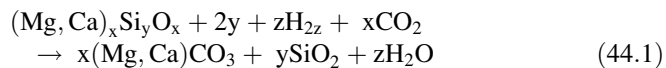

Fig. 44.1 General chemical mineralization path
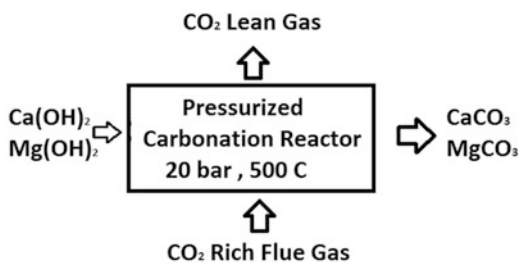
alkaline to almost neutral. The amount of CO_2 captured from flue gases can reach 0,01–0,12 kg/l at the 500 °C temperature and 10 bar pressure level (O'Connor 1998). Laboratory-scale neutralization experiments were carried out to compare the reactor designs. Sedimentation of carbonate particles was observed and their main characteristics were determined.

Mineral carbonation reactions are known to geologists and occur spontaneously on geological time scales. For example, the reaction of CO_2 with common mineral silicates to form carbonates like calcite and magnesite or calcite is exothermic and thermodynamically favored (O'Connor 1998; Butt et al. 1997; Drägulescu et al. 1972).

The family of reactions represented by Reaction 44.1 has the potential to convert naturally occurring silicate minerals to geologically stable carbonate minerals and silica (Goldberg et al. 2000). This process follows natural chemical transformations such as weathering of rocks to form carbonates over geologic time periods. Reaction 44.2 illustrates the transformation of the common silicate mineral serpentine (Goff et al. 1997), $Mg_3Si_2O_5(OH)_4$, and CO_2 into magnesite, $MgCO_3$, silica and water. Using this ideal case, 1 ton of serpentine can dispose of approximately one-half ton of CO_2. Reaction 44.3 illustrates the transformation of forsterite, which is the end member of the common silicate mineral olivine. One ton of olivine can dispose of approximately two-thirds of a ton of CO_2. Again, the reaction is exothermic and releases 90 kJ/mole of CO_2.

$$(Mg, Ca)_x Si_y O_x + 2y + zH_{2z} + xCO_2$$
$$\rightarrow x(Mg, Ca)CO_3 + ySiO_2 + zH_2O \tag{44.1}$$

$$1/3\ Mg_3Si_2O_5(OH)_4 + CO_2 \rightarrow MgCO_3 + 2/3\ SiO_2$$
$$+ 2/3\ H_2O \quad + 64\ kJ/mole. \tag{44.2}$$

$$1/2\ Mg_2SiO_4 + CO_2 \rightarrow MgCO_3 + 1/2\ SiO_2 + 90\ kJ/mole \tag{44.3}$$

A conceptual illustration of the projected Fly ash mineralization process is presented in Fig. 44.2. As illustrated, CO_2 from one or more power plants is transported to a carbonation reactor, combined with fly ash slurry tank and held at the appropriate reaction conditions until the desired degree of carbonation is
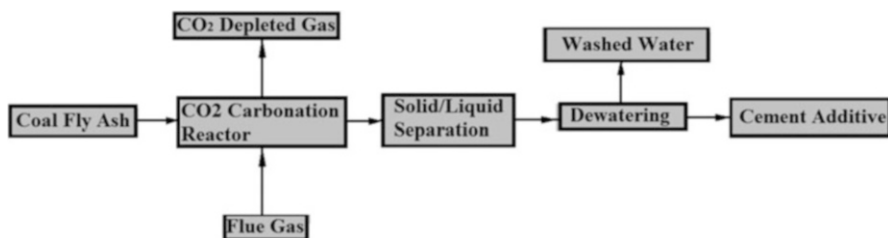

Fig. 44.2 Chemical reaction process of fly ashes for sequestration in the diagram

reached. Careful control of solution chemistry yielded olivine conversions of 90 % in 24 h and 83 % within 6 h. The most recent results show further modifications of the same basic reaction can achieve 65 % conversion in 1 h and 83 % conversion in 3 h. A recent literature review indicated that weak carbonic acid treatments had also been suggested for Mg extraction in the prior literature (O'Connor 1998). Carbonation tests performed at ARC employing heat pretreated serpentine have resulted in up to 83 % conversion in 30 min lower than 115 bars (O'Connor 1998). Indeed, by increasing sodium bicarbonate concentration the carbonation reaction of serpentine can reach 62 % completion under 50 bars. Waters contains various natural elements. Substantial fractions of these elements in springs could be lost during clay rock pores leak. 50–60 % of arsenic, lead, manganese, mercury and selenium could be removed by salt transforming in cooling.

Then products of the reaction, which might be slurry of carbonated minerals and residues and lean gas CO$_2$ in aqueous, are separated. The residual CO$_2$ is recycled. Useful materials are collected for construction works or the carbonated materials and residue are returned to the mine site. Almost calcium and magnesium oxide (MgO) content in the magnesium silicate ore mineral of 40 % and 60–70 % chemical efficiency of the carbonation reaction; mass balance of equation 44.1. A 100 MW power plant in Sırnak-Silopi, generating approximately 200 tons/day of CO$_2$, would require just over 140 tons/day of calcium and magnesium containing fly ash. Several fly ash types in Turkey containing sufficient calcium and magnesium oxide quantity in silicate mineral to provide raw materials for the mineral carbonation.

Experimental Work

In this research, representative specimens the different types of Turkish geothermal hot water sources Sirnak, were classified to calcium content and bicarbonate by chemical analysis. Gas samples of 10–20 kg tubes from each different coal power stations were used. Screen analysis of Turkish lignite samples were made by standard Tyler Screens and particle size distributions and normal distributions of Turkish fly ash samples are respectively illustrated from Fig. 44.3.

Number citations consecutively in alkali waters were reacted by waste gas in the process reactor (Fig. 44.2) and solution distributions were investigated at each stage of sequestration. Specific surface area of fly ash minerals was about 4.76–6.2 m^2/g determined by BET surface analyzer and highly sufficient in order to react with gaseous CO$_2$. 80 % of weights of fly ash were reacted in 10 bar alkali solution. Main reactive salt structure is widely distributed and pores structures are associated with clay minerals. Coarse alkali oxides are also seen (Table 44.1).

From these results, we designed and developed laboratory scale process unit consisting a three consecutive autoclave reactors (1 m $\Phi \times$ 0.7 m) to capture and carbonize flue gas CO$_2$. Flue gas was filtered and withdrawn from the gas tube and was fed to the autoclave with fly ash slurry at about 7 l/s. The cyclone and decanter
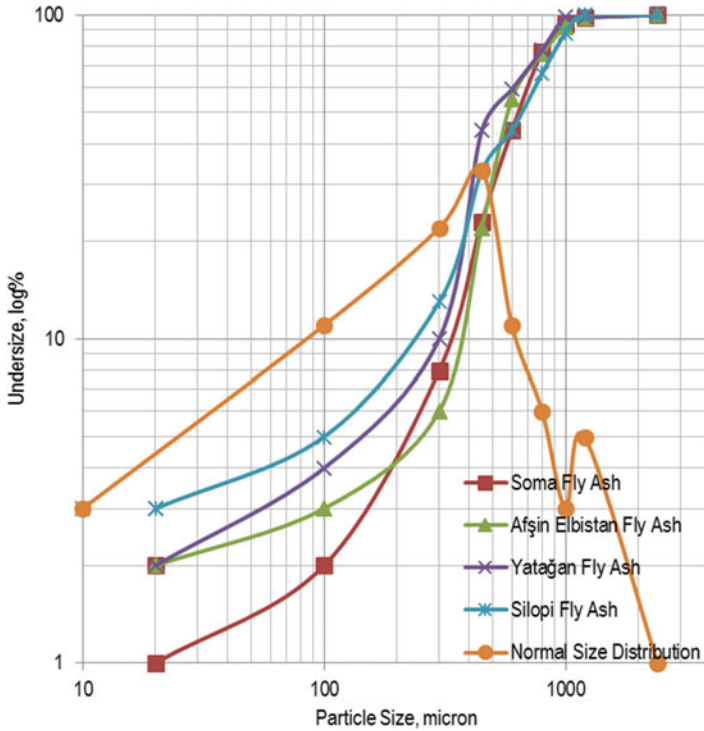
Fig. 44.3 Particle size distribution and normal size distribution of the fly ash used in carbonation process

Table 44.1 Chemical analysis of Turkish fly ash used in the experiment

Chemical composition	Soma fly ash F	Yatagan fly ash F	Silopi fly ash F	Afşin Elbistan fly ash F
SiO_2 (S)	42.14	52.05	40.4	20.4
Al_2O_3 (A)	17.12	23.69	4.1	7.1
Fe_2O_3(F)	4.34	5.94	15.96	4.96
$CS + CAF$	63.26	81.68	81	88
CaO	29.6	19.60	24.7	64.7
MgO	1.53	2.38	2.1	3.1
K_2O	1.15	2.51	1.55	0.55
Na_2O	1.29	2.56	1.25	0.250
Loss of ignition	0.34	0.90	5.22	4.22
SO_3	3.43	1.45	2.2	2.4
Density (g/cm^3)	2.22	2.12	1.9	2.0
Loose unit weight (g/cm^3)	0.72	0.88	0.71	0.69

unit separates ash and aqueous water. The salt particles were fluidized by flow of slurry through a slurry tank and pump. The laboratory scale studies were conducted at a controlled pressure (115.1 kPa) by controlling the flue gas content. The flue gas was continuously monitored to measure flue gas CO_2, SO_2 and NO_x concentrations by an industrial grade gas analyzer, while the fresh and spent ash were analyzed for calcium carbonate ($CaCO_3$), sulfur (S), and mercury (Hg) content.

Results and Discussion

The major technical challenge now hindering the use of minerals to sequester CO_2 is theirs low reaction rate. Weathering of rock is extremely slow. The highest priority is given to identifying faster reaction pathways. The optimized process has to be economical. Although many carbonation reactions are exothermic, it is generally very difficult to recover the low-grade heat while the long reaction time and demanding reaction conditions.

The environmental impact from mining, mineralization and carbonation processes must be considered in sequestration. We succeeded in achieving shortened carbonation reaction times employing fly ash containing lime and calcium magnesium silicates such as gehlenite, mehlenite. Reaction took 4 h to reach 40–50 % completion of carbonation. The reaction required temperatures of 450–550 °C, pressures of 10–20 bar, and mineral particles in the −100 μm size range. Because the high pressure requirement of the carbonation reaction will certainly lead to high process costs, the team is modifying solution chemistry to allow reaction to proceed at a lower pressure and temperature. The research showed that the concentration of HCO_3^- in the solution is critical to the reaction rate. The high CO_2 pressure will lead increased CO_2 absorption in the solution and thus enhance the HCO_3^- concentration. Adding bicarbonate such as sodium bicarbonate in the solution will significantly increase the HCO_3^- concentration even at a relatively lower CO_2 pressure.

In experiments with 3 h reaction period at reactor temperature of 500 °C and fly ash slurries were kept under changing CO_2 pressure ranged 5 bar to 40 bar. Products were subjected to analysis for carbonate yield determination. Test results of yields for different Turkish fly ash sources were seen in Fig. 44.4.

That quantity in the carbonation amounts were determined for different source evaluation and reduce the effect of gas content of waste gas in order to optimize carbonation rates. As given in Fig. 44.4 gas conversion yield for steam and ash samples were greatly differed. Yatağan and Elbistan Fly ash showed high carbonation yields reaching 32 % and 30 % respectively. Because of active lime amounts were over 20 % and even alkali Na and K contents were over totally over 5 %, the higher carbonation yields were sufficiently provided by three consecutive carbonation columns at 20 bar.

In the carbonation experiments with addition alkali, reactor temperature changed between 200 and 650 °C and ash slurries were mixed with active lime additionally by 10 % rate. Products received from carbonation of ash specimens were subjected
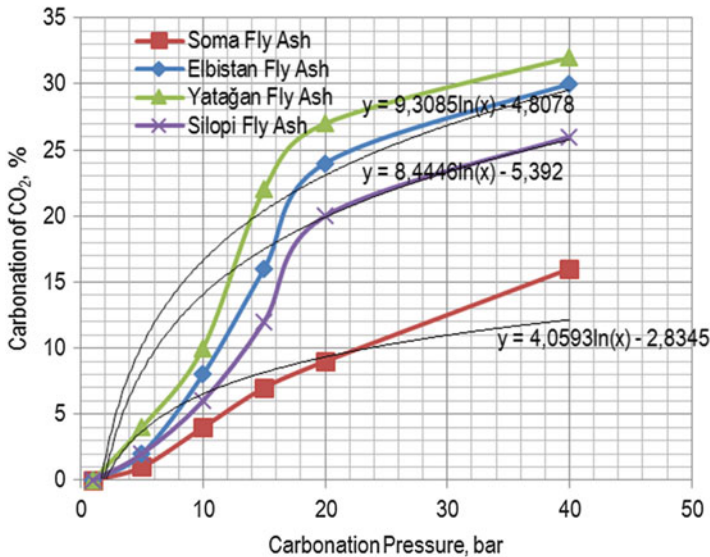
Fig. 44.4 Effect of carbonation time over conversion yield rates in hot 500 °C steam used

to analysis for gas hold-up determination. Test results of carbonation by fly ash and also alkali lime contained over active lime at 10 % weight rate were investigated.

In experiments with 3 h reaction period of ash specimens, at reactor temperature changed between 200 and 600 °C and all ash samples contained over active lime at 10 % weight rate. Products were subjected to analysis for carbonate yield determination. Test results of carbonation yields of Turkish fly ash sources and additional 10 % lime were seen in Fig. 44.5.

As given in Fig. 44.5 carbonation yield for steam and ash samples were linearly changed between 200 and 650 °C. Yatağan and Elbistan Fly ash showed high carbonation yields reaching 44 % and 24 % at 650°C, respectively. Because of low reaction kinetics below 500 °C and even the effect 10 % over dosage of active lime content in the reactors were ranged over 3 % carbonate yield per hour, the higher carbonation yields were sufficiently provided over 500 °C at 20 bar.

In the carbonation experiments, the experimental conditions were controlled on the basis of the gas composition in the ambient state. So neither the contained water vapor nor the others, slurry was taken into account. In order to determine the effect of reaction period on carbonation yield for Turkish fly ash samples, the tests were carried out in three consecutive carbonation columns at 500 °C and 20 bar between1min to 6 h. Yatağan and Elbistan Fly ash showed high carbonation yields reaching 33 % and 26 % at 500°C at 3 h reaction period, respectively. Because of low reaction kinetics below 30 min reaction period in the reactors, yield rates were ranged over 1 % carbonate yield per hour, the higher carbonation yields were sufficiently provided over 2 h reaction period at 20 bar. Carbonation of slurries and yield products changed to 37 % and 29 % for Yatağan and Elbistan Fly ash, respectively, by time increase from 1 to 6 h (Fig. 44.6).
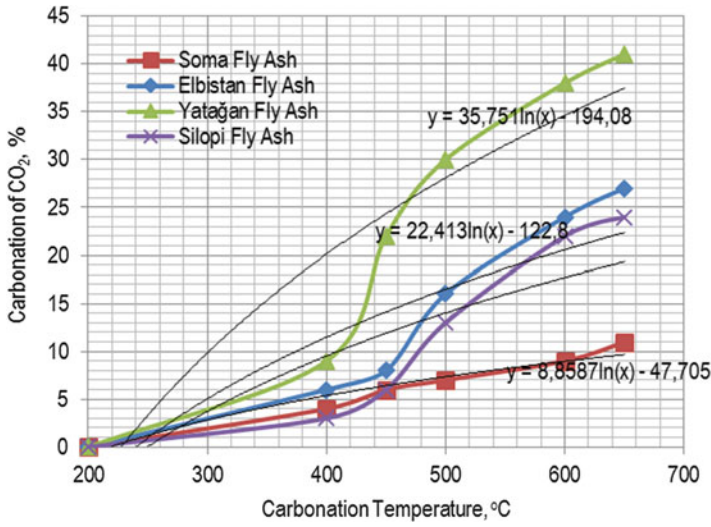
Fig. 44.5 Effect of time of carbonation over conversion yield rates in steam with 10 % additional lime used at 20 bar
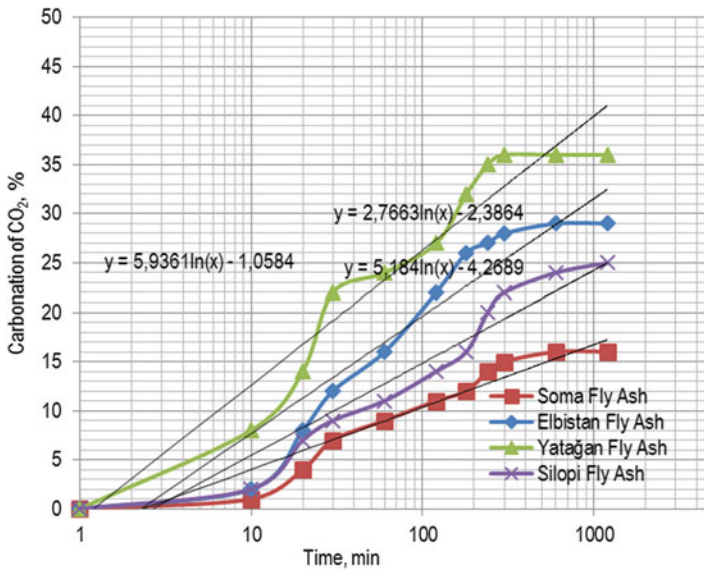
Fig. 44.6 Effect of time of carbonation over conversion yield rates in hot waters used at 500 °C and 20 bar

Conclusion

The qualitative comparison of the carbonation for three different Turkish fly ash reactants and products revealed a complete MgO, CaO–MgO to $MgCO_3$-$CaCO_3$ conversion. The carbonation efficiency of CaO was dependent on the initial pressure of CO_2 of 20 bar. This was significantly affected by reaction temperature 500 °C and by the fly ash dose 100 g/l. The kinetic data demonstrated that the initial rate of CO_2 transfer was enhanced by carbonation process for our experiments. The precipitate calcium carbonate was characterized by isolated micron sized particles and micron agglomerates of calcite.

This study reveals suitable large scale operating units in order to achieve the carbonation method as a viable sequestration tool at industrially relevant scales by using Turkish fly ashes in 500–600 °C waters. Carbonation liquid and gaseous products may change to near 20–40 % yield performances by time increase from 1 to 6 h.

While there is a potential to utilize other types of flue ashes in mineralization, lime or similar alkali can be evaluated to sequester CO_2 allowing clearly significant amounts. Even there are researches succeeded usage of serpentine and olivine. Consequently, the flue gas should be continuously monitored to measure flue gas flow at depleted gas outlet in order to reprocess it.

Carbonation of the ash slurries and carbonation yield products changed to 37 %, 29 %, 25 % and 19 % for Yatağan, Elbistan, Silopi and Soma Fly ash, respectively, by time increase from 1 to 6 h.

Other harmful emissions caused by flue gas containing high sulfur (S), and mercury (Hg) content can be eliminated by this method. In that study, results suggested that an appreciable amount of flue gas CO_2 and significant amounts of SO_2 and Hg can be directly captured and mineralized by the fly ash particles.

Even with progress made so far, to develop an economical method to sequester CO_2 with minerals is still a challenging task, because the process is still relatively slow, and most reactions require high pressure and moderately elevated temperature.

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