

## Full Length Research Paper

# Behaviours of $\text{MeCl}_2$ (Me: Pb and Cd) during thermal treatment of kaolin-lime mixture

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Accepted 1 August, 2011

**This paper reports the emission behaviours of Pb and Cd during heat treatment of their chloride salts with kaolin-lime and kaolin-lime-silica mixtures. The leaching behaviours of Pb and Cd from the heat-treated products are also assessed. The results showed that the addition of lime into kaolin increased the emitted amounts of Pb and Cd during heating. Addition of silica into the mixtures further increased the emissions of both elements at low temperature ( $\leq 1000^\circ\text{C}$ ) and inhibited their emissions at high temperature ( $\geq 1100^\circ\text{C}$ ). Crystallinity of silica had profound effect on the emissions of Pb and Cd from the mixtures. The heat-treated product containing anorthite as the major crystalline phase not only retain more amounts of Pb and Cd in its composition but also emit lesser amounts of Pb and Cd into solution than the product containing gehlenite as the major crystalline phase during subjected leaching test.**

**Key words:** Toxic element, kaolin, lime, silica, heat-treatment, leaching.

## INTRODUCTION

Thermal treatment such as incineration and combustion is generally adopted to decrease volume of various types of wastes as well as to produce energy from fossil fuels. However, incineration of industrial wastes and combustion of fossil fuels often create problem due to emissions of large amounts of gases containing particulate matters and toxic elements such as Cd, Pb into the atmosphere. Different devices like scrubbers, electrostatic precipitators, bag-filters are generally used in incinerator plant or combustion unit to control the emissions of toxic gases and fine particulates. Several solid sorbents are also used to capture different toxic elements produced during thermal treatment (Ubroy and Shadman, 1991; Yao et al., 2004; Diaz-Sumoano and Martinez-Tarazona, 2005; Linak et al., 1995; Gullet and Ragnunathan, 1994). Both physical and chemical adsorptions processes are responsible for stabilization of toxic elements in solid sorbents (Chen et al., 2001).

Improvement on the control of emissions of toxic components into atmosphere however increases the concentrations of toxic components in ashes, obtained after incineration or combustion. Nowadays, a large volume of ash containing high amounts of toxic elements including Pb and Cd is produced all over the world. Treatment of incineration or combustion ashes containing high amounts of toxic component has significant importance for controlling the leaching of toxic components present in these ashes. One of the effective and commonly used treatment method to neutralize the toxic component induce toxicity is the thermal treatment of these ashes. However, major drawback of thermal treatment is the consumption of high amount of energy. Any low temperature process, which can effectively immobilize toxic components of waste, will be more attractive from economical as well as environmental points of view.

The heat-treatment of ash at low temperature ( $\leq 1050^\circ\text{C}$ ) is therefore proposed to decrease the energy consumption (Matssuzawa et al., 2006). Presence of some components in low temperature heat treated product such as gehlenite and some calcium

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**Table 1.** Different mixtures used for heat treatment.

Name	Amount (%)					
	Kaolin	Lime	Silica	Quartz	Pb	Cd
K	97	-	-	-	2	1
KL2	77	20	-	-	2	1
KL3	67	30	-	-	2	1
QKL2	67	20	-	10	2	1
QKL3	57	30	-	10	2	1
SKL2	67	20	10	-	2	1
SKL3	57	30	10	-	2	1

aluminosilicate based amorphous products can take several elements in its structure and therefore they can reduce the emission of toxic components into the environment (Matssuzawa et al., 2006; Okada et al., 2003; Jha et al., 2004, 2005). Moreover, several types of wastes containing very high amounts of toxic elements including Pb and Cd as well as waste derived ashes are often used to prepare various ceramic products like bricks and tiles (Saikia et al., 2000, Sengupta et al., 2002, Ediz and Yurdakul, 2009, Menezes et al., 2009). These materials also contain minerals like anorthite and gehlenite. Not much has been reported on the emission behaviour of toxic elements during production of toxic waste containing ceramics and also leaching behaviour of toxic components from the heat-treated product that may contain minerals like anorthite and gehlenite.

In the present study, we report the emission behaviour of Cd and Pb during heat-treatment of kaolin-lime and kaolin-lime-silica mixtures at the temperature range of 800 to 1200°C. The temperature chosen for heat-treatment is generally in the range that considered for low temperature heat treatment methods for incinerated ashes as well as the temperature regions that can be suitable for synthesis of minerals those present in various ceramics. The chloride salts of Cd and Pb are considered because chloride is one of the major anionic constituents of MSWI fly ash. Finally the leaching behaviours of Pb and Cd present in the heat-treated products are also assessed.

## EXPERIMENTALS

### Materials

Kaolin sample used in this investigation was collected from Sigma-Aldrich Co, USA. High quality, analytical grade  $PbCl_2$  and  $CdCl_2 \cdot 2.5H_2O$  were used as a Pb and Cd sources respectively. Other chemicals used to prepare solid mixtures were reagent grade and collected from Kanto Chemical Co. (Japan). Details about the characterization of kaolin were presented elsewhere (Saikia et al., 2004). Ultra pure double distilled water with a conductance of  $1 \times 10^{-7}$  S/cm was used in all experiments. All the glass and plastics wares, used in this investigation were also immersed in a 0.1 M  $HNO_3$  acid solution for overnight and then washed several times by

ultra pure water.

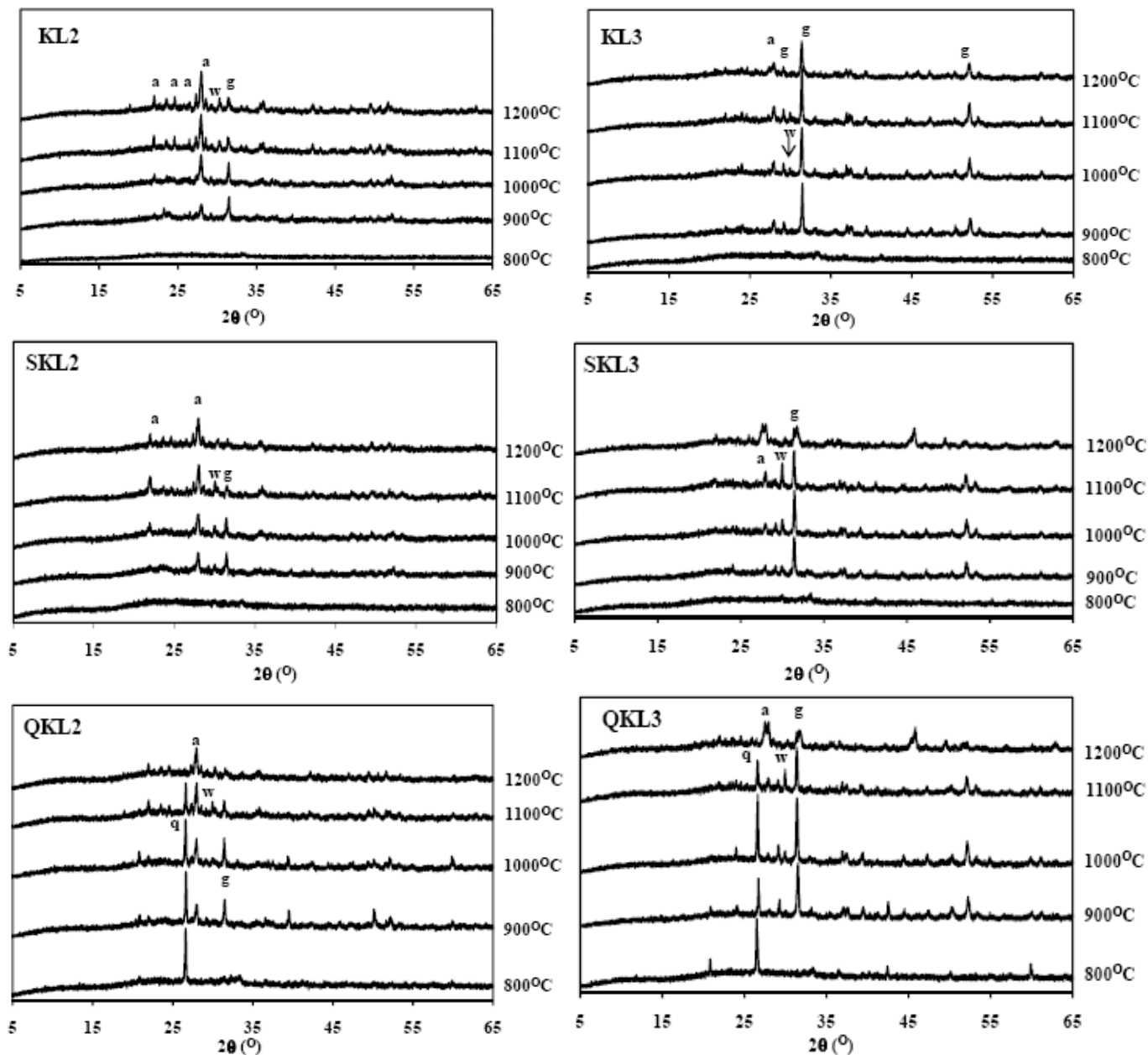
The different mixtures prepared in this investigation along with their names are presented in Table 1. Initially, mixtures containing all the components except Pb and Cd salts were mixed and homogenized in a ball mill. In these homogenized mixtures, about 2% Pb as  $PbCl_2$  and about 1% Cd as  $CdCl_2 \cdot 2.5H_2O$  were then added and again homogenized the mixtures. A metal chloride mixed kaolin mixture was also homogenized. The mixtures were covered tightly and stored in a desiccator to protect from moisture. Amorphous silica and crystalline quartz were used to study the effect of silica sources on the emission behaviours of Pb and Cd.

### High temperature experiment

To study the emission behaviours of Pb and Cd, precisely weigh 5 g of mixture was taken in a platinum crucible and heated in an electric muffle furnace in air atmosphere for 24 h at 800, 900, 1000, 1100 and 1200°C. After cooling, the heat-treated products were removed from the furnace, measured the weight change during heating, ground in an agate mortar and stored in air tight plastic bags and in vacuum desiccator. The XRD patterns of the powdered products were recorded by an X-ray diffractometer using Cu K- $\alpha$  radiation (model Miniflex, MJ 14848 B01, Rigaku, Tokyo, Japan). To determine the amounts of Pb and Cd in the raw and the heat-treated products, the samples were digested successively by using  $HNO_3$ , HF,  $HClO_4$  acid on a hot plate. The concentrations of Pb and Cd present in the solutions obtained after digestion were measured by using a sequential-type inductively coupled plasma atomic emission spectrophotometer, model ICPS-7500 (Shimadzu, Tokyo, Japan).

### Leaching experiment

Toxicity characteristic leaching procedure (TCLP) leaching test after a slight modification was adopted to study the leaching behaviour of Pb and Cd from the heat-treated products (EPA SW-846, 1992). Due to the alkaline nature of the product, the leachate solution was prepared at  $2.89 \pm 0.05$  by mixing analytical grade glacial acetic acid with distilled water. To generate TCLP leachate, 2.5 g of the powdered heat-treated product was mixed with 50 ml of TCLP fluid (liquid to solid ratio: 20) in a plastic beaker and shaken for 19 h in an electric shaker at 24°C. After completion of the leaching test, the pH of the suspension was measured. The liquid fraction (leachate) was then collected by using a 0.45  $\mu m$  cellulose acetate membrane filter (Advantec Toyo, Tokyo, Japan) and kept in plastic container at pH 2.5 using a few drops of concentrated  $HNO_3$ . The Pb and Cd concentrations in the leachate were determined by using an inductively coupled plasma atomic emission spectrophotometer.



**Figure 1.** XRD patterns of different Pb and Cd containing mixtures after heat-treatment at various temperatures [a: anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ ); g: gehlenite ( $\text{Ca}_2\text{Al}_2\text{SiO}_7$ ); w: wollastonite ( $\text{CaSiO}_3$ ); q: quartz ( $\text{SiO}_2$ )].

## RESULTS AND DISCUSSION

### Analysis of X-ray diffraction patterns

Figure 1 show the XRD patterns of the products obtained after heat-treatment of various mixtures. Details about the different compositions are indicated in Table 1. The XRD patterns of 800°C heat-treated kaolin-lime (KL) and amorphous silica containing kaolin-lime (SKL) mixtures are X-Ray amorphous with a broad halo in the  $2\theta$  range of 20 to 35°. However, quartz-containing mixtures (QKL) exhibit characteristic peaks of quartz in this region. The

amorphous nature of the products as well as the complete absence of peaks due to kaolinite, lime and calcite in the XRD patterns of these mixtures indicate the formation of calcium aluminosilicate based amorphous compounds due to reaction of lime with kaolinite at 800°C. Okada et al. (2003) also reported the formation of an amorphous calcium aluminosilicate,  $\text{CaAl}_2\text{Si}_2\text{O}_8$  during the reaction of kaolin and calcite at 800°C.

Heat-treatment of KL2 to 900°C shows the formations of both gehlenite ( $\text{Ca}_2\text{Al}_2\text{SiO}_7$ ) and anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ ) as the crystalline products. Heating of this mixture to high temperatures increases the intensities of anorthite peaks

and decreases the intensities of gehlenite peaks in their XRD patterns, possibly due to conversion of gehlenite into anorthite and other products (Unsworth et al., 1988). The peak due to the formation of wollastonite ( $\text{CaSiO}_3$ ) in the XRD patterns of KL2 mixture is appeared after heat-treatment at 1100 °C. On the other hand, XRD patterns of KL3 mixture obtained after heat-treatment at 900 °C shows the intense peaks due to the formation of gehlenite with weak peaks due to anorthite. Heating of this mixture to high temperatures shows the formation of more amounts of gehlenite and anorthite by showing the increased peak intensities of these compounds. Formation of wollastonite in the KL3 mixtures is also started from 900 °C. Formation of high amount of gehlenite in KL3 mixture is due to the presence of relatively high amount of lime than that present in KL2 mixture.

On the other hand, both amorphous and crystalline silica containing mixtures show the formations of anorthite and gehlenite from 900 °C. With increasing heating temperatures, the intensities of anorthite peaks are increasing and the intensities of gehlenite peaks are decreasing in all the XRD patterns of silica containing mixtures. Strong peak of wollastonite is also observed due to the presence of additional amount of silica in these mixtures. The intensities of the peaks due to quartz are almost similar in the various XRD patterns of QKL mixtures, obtained after heat-treatment in the temperature range of 800 to 1000 °C but started to diminish after heat-treatment at 1100 °C and almost disappeared after heat-treatment at 1200 °C. Thus the reaction of quartz with other constituents starts after 1000 °C and almost completed after heat-treatment at 1200 °C. The width of major peaks of all the crystalline phases in the XRD patterns of all the products obtained after heating at 1200 °C are increased due to the formation of amorphous glassy materials.

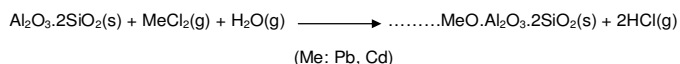
### Behaviour of Pb and Cd during heat-treatment

Table 2 shows the amounts of Pb and Cd present in different heat-treated products. The amounts of Pb and Cd in the heat-treated products as presented in the Table 2 are normalized data with respect to the amounts present in the raw mixtures. For this purpose, the amount loss from a mixture during heat-treatment at a particular temperature is determined.

The amounts of Pb and Cd present in 800 °C heat-treated all products are almost similar to the amount that present in untreated samples. This is due to the higher boiling points of  $\text{CdCl}_2$  and  $\text{PbCl}_2$  than the treated temperature. The boiling points of  $\text{CdCl}_2$  and  $\text{PbCl}_2$  are respectively 950 and 960 °C. If some amounts of them are volatilized during heating, they can be physically and chemically adsorbed on the surfaces of the products, formed during heat-treatment (Chen et al., 2001; Ubroy and Shadman, 1991). The reduction in concentrations of

Pb and Cd after heat treatment at 900 °C is unknown; however one possible reason is the lowering of boiling point of  $\text{CdCl}_2$  and  $\text{PbCl}_2$  due to increasing vapour pressure in the complex mixture containing chloride salts (Osada et al., 2009). Physically adsorbed Cd and Pb may also be volatilized at this temperature as the physical adsorption process is less effective at the temperature above 800 °C. Heating of all the mixtures to higher temperature further decreases the concentrations of both elements in the products due to their volatilizations.

Figure 2 shows the emitted percentages of Pb and Cd from the different heat treated products of KL2 and KL3 mixtures. The amounts of both elements emitted from KL2 mixture are higher than those emitted from KL3 mixture, which indicates that the increasing content of lime increases the emissions of Cd and Pb from the KL mixture. To confirm the effect of lime, a kaolin-metal chlorides mixture was heat-treated at various temperatures and emitted amounts of Pb and Cd were determined. The amounts of Pb and Cd emitted from kaolin-chloride mixture are presented by broken lines in Figure 2. The amounts of Pb and Cd emitted from the kaolin only mixture is considerably lower than those emitted from KL mixtures at the experimental temperature region. It was reported earlier that the chloride salts of Cd and Pb can reacts with kaolin during heat-treatment and form  $\text{CdAl}_2\text{Si}_2\text{O}_8$  and  $\text{PbAl}_2\text{Si}_2\text{O}_8$ , which can be represented by following chemical reaction (Holland et al., 1976; Ubroy and Shadman, 1991; Yang et al., 2001):

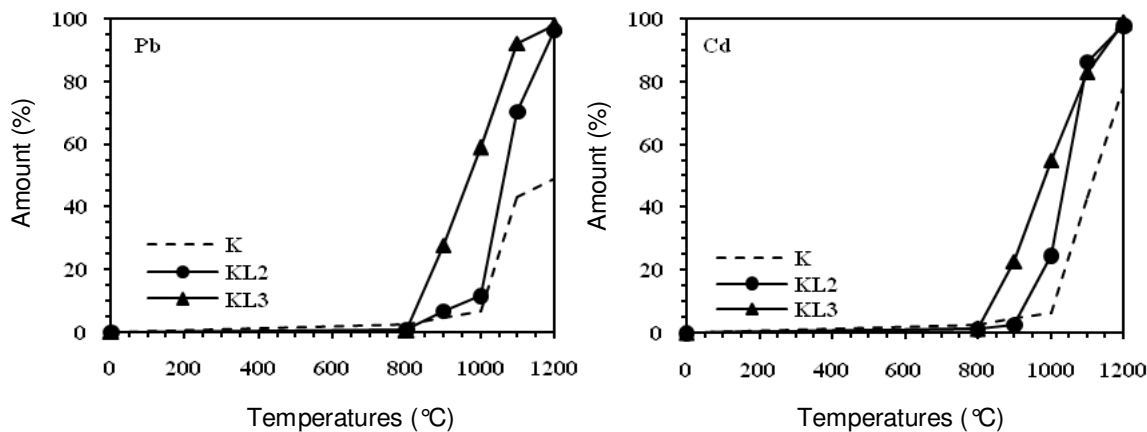


Addition of lime into kaolin possibly favours the formation of calcium aluminosilicate based products such as gehlenite and anorthite, which decreases the availability of aluminosilicates to reacts with metal chlorides and therefore favours their (metal chlorides) evaporations. However, these calcium aluminosilicates can entrap Pb and Cd in their crystal structures too.

Figure 3 shows the emitted percentages of Pb and Cd from the different kaolin-lime-silica mixtures during heat-treatment at various temperatures. In most of the cases, the amounts of Pb and Cd emitted from the mixtures containing amorphous and crystalline (quartz) silica during heat-treatment at 1100 and 1200 °C are lower than that emitted from the mixtures prepared without silica. On the other hand, compared to KL mixtures, higher emissions of both elements are observed from silica containing mixtures except SKL3 due to the heating of these mixtures up to 1000 °C. Furthermore, the emitted amounts of Pb and Cd from the SKL mixtures are higher than those emitted from the QKL mixtures after heat-treatment at or above 1100 °C. In case of heat-treatment at or below 1000 °C, the amounts of both elements emitted from the QKL mixtures are higher than the amounts emitted from SKL mixtures. Thus the presence and nature of silica has considerable effect on the emission behaviour

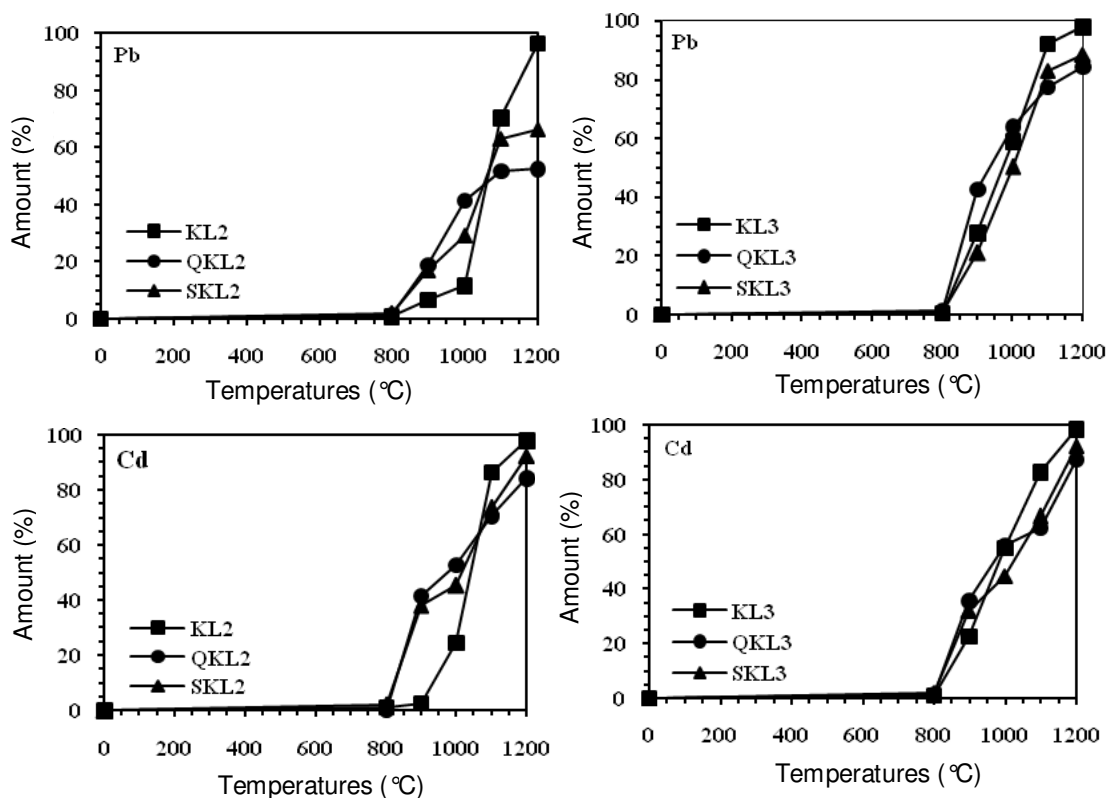
**Table 2.** Concentration of Pb and Cd in different products obtained after heat-treatment at different temperatures.

Temperature (°C)	Concentration (mg/g)			
	Pb		Cd	
	K		K	
0	17.14		7.60	
800	16.69		7.40	
900	16.33		7.24	
1000	16.01		7.10	
1100	9.74		4.32	
1200	8.72		1.65	
	KL2	KL3	KL2	KL3
0	16.95	15.74	7.77	7.95
800	16.80	15.64	7.68	7.85
900	15.81	11.38	7.57	6.13
1000	14.99	6.45	5.86	3.57
1100	5.04	1.25	1.07	1.37
1200	0.62	0.35	0.17	0.12
	QKL2	QKL3	QKL2	QKL3
0	16.62	16.68	7.86	7.88
800	16.43	16.43	7.83	7.78
900	13.46	9.59	4.58	5.07
1000	9.72	6.00	3.71	3.47
1100	8.03	3.79	2.32	2.96
1200	7.92	2.62	1.24	0.99
	SKL2	SKL3	SKL2	SKL3
0	16.73	16.56	8.60	7.77
800	16.44	16.40	8.42	7.59
900	13.90	13.13	5.33	5.27
1000	11.86	8.27	4.72	4.29
1100	6.17	2.82	2.29	2.60
1200	5.63	1.91	0.67	0.59

**Figure 2.** Amounts of Pb and Cd emitted from the kaolin-lime mixtures at different temperatures.

of Pb and Cd during heat-treatment of silica containing lime-kaolin mixtures.

Besides aluminosilicates, both elements can also form some stable aluminosilicates, silicates like  $PbO \cdot SiO_2$  and



**Figure 3.** Amounts of Pb and Cd emitted from the kaolin-lime-silica mixtures at different temperatures.

CdO.SiO<sub>2</sub> at high temperature due to the addition of silica into KL mixtures (Folguerus et al., 2007). The emission of lower amounts of Pb and Cd from the SKL mixtures during their heating at low temperature ( $\leq 1000^{\circ}\text{C}$ ) is possibly due to the formations of higher amounts of Pb and Cd aluminosilicates and silicates. At high temperature ( $\geq 1100^{\circ}\text{C}$ ), possibly no more silica is available in SKL mixtures to react with remaining amounts of Pb and Cd and therefore cannot control their emissions. However, it was not possible to detect the silicates and aluminosilicates of Pb and Cd in the XRD patterns, possibly due to their minimal concentrations in the products.

On the other hand, the emission behaviours of Pb and Cd from the QKL mixtures can be related to the reactivity of quartz during heat-treatment. The XRD patterns of QKL mixtures (Figure 1) show that the intensities of quartz peaks are almost similar up to  $1000^{\circ}\text{C}$  and started to decrease after heat treatment at  $1100^{\circ}\text{C}$ , which indicates that the crystalline silica converted into amorphous reactive phase at  $1100^{\circ}\text{C}$ . Therefore the presence of high amounts of reactive or amorphous silica in the QKL mixtures after heat-treatment at or above  $1100^{\circ}\text{C}$  possibly hampers the emissions of Pb and Cd by forming some stable aluminosilicates like PbO.SiO<sub>2</sub> and CdO.SiO<sub>2</sub>.

### Leaching behaviour of Pb and Cd from the different heat-treated products

Table 3 shows the pH of the leachates and the concentrations of Pb and Cd in the leachates generated after TCLP leaching tests of the mixtures after heat-treatment at various temperatures. Very small amounts of Pb and Cd are liberated during acidic leaching of all the products obtained after heating at  $800^{\circ}\text{C}$  although almost all amounts of them are present in the products. Possibly the formed metal aluminosilicates and silicates are insoluble at the experimental leaching conditions or amorphous compound formed in all the mixtures during heat-treatment at  $800^{\circ}\text{C}$  can incorporate Pb and Cd in its structure and restricts their liberations during leaching. Past investigations showed that amorphous calcium aluminosilicates, CaAl<sub>2</sub>Si<sub>2</sub>O<sub>8</sub> and Ca<sub>2</sub>Al<sub>2</sub>SiO<sub>7</sub> produce from calcite and kaolin mixtures could take high amounts of transition elements like Ni<sup>2+</sup> from the solution by various physico-chemical processes (Okada et al., 2003; Jha et al., 2004, 2005). It was also reported that the uptake behaviour of this amorphous compound was even better than zeolites. These amorphous compounds also showed the higher selectivity for the uptake of transition metal ions than for the alkali and alkaline earth ions.

The range of pH of the leachates (7.10-9.34) generated from 30% lime containing mixtures is somewhat in the

**Table 3.** TCLP leaching data of heat-treated samples.

Temperature (°C)	pH		Concentration (mg/g) of			
	KL2	KL3	Pb		Cd	
			KL2	KL3	KL2	KL3
800	5.36	8.63	0.0012	0.0038	0.0068	0.0078
900	4.28	5.33	0.0178	0.0662	0.0423	0.0529
1000	4.03	5.15	0.0178	0.0422	0.0457	0.0401
1100	3.68	4.94	0.0272	0.0352	0.0148	0.0170
1200	3.65	5.05	0.0020	0.0022	0.0018	0.0009
	QKL2	QKL3	QKL2	QKL3	QKL2	QKL3
800	5.74	7.10	0.0070	0.0022	0.0090	0.0115
900	4.99	7.14	0.0201	0.0421	0.0280	0.0656
1000	4.81	5.23	0.0290	0.0355	0.0325	0.0868
1100	4.55	5.10	0.0177	0.0142	0.0117	0.0136
1200	3.83	7.60	0.0030	0.0050	0.0018	0.0024
	SKL2	SKL3	SKL2	SKL3	SKL2	SKL3
800	5.88	9.34	0.0014	0.0096	0.0050	0.0104
900	4.85	5.81	0.0597	0.0752	0.0418	0.0745
1000	4.75	5.08	0.0583	0.0563	0.0355	0.0366
1100	4.44	4.94	0.0217	0.0173	0.0192	0.0184
1200	3.79	6.54	0.0012	0.0016	0.0012	0.0017

range, where Pb and Cd can form some insoluble complexes during aqueous and acetic acid based leaching test. On the other hand, Pb and Cd are generally present in soluble form at the pH range of the leachates (5.36-5.88) generated from 20% lime containing products. However, in most of the cases, the concentrations of the Pb and Cd in the leachates generated from the product prepared from 20% lime containing mixture are lower than that present in the leachates generated from the products prepared from 30% lime containing mixture. Similar behaviour is also observed in almost all of the mixtures heat-treated up to 1000°C. On the other hand, compared to the amounts leached from the products generated after heat-treatment of 30% lime containing mixtures at or above 1100°C, higher amounts of Pb and Cd are dissolved from the majority of products obtained after heat-treatment of 20% lime containing mixtures.

Since the XRD patterns of 20 and 30% lime containing compositions show the formation of two structurally different types of compounds, anorthite and gehlenite respectively. Therefore the amount of Pb and Cd dissolve from the product at low temperatures (that is, 900 and 1000°C) can be related with the formation of these compounds during heating of the mixtures. The more amounts of Pb and Cd are dissolved from the product, where gehlenite is the major product in the mixtures. The structure of gehlenite is layered type while anorthite has a framework structure. Possibly,  $Pb^{2+}$  and  $Cd^{2+}$  substituted gehlenite and anorthite are formed during the heat-treatment of the mixtures. The release of  $Pb^{2+}$  and  $Cd^{2+}$  present in host materials may occur more readily from the

layered structured gehlente than the framework structured anorthite and therefore high amounts of Pb and Cd are leached from the gehlenite containing products. Thus compared to gehlenite containing heat-treated product, the product containing anorthite as the major crystalline phase can not only retain more amount of Pb and Cd during heat-treatment but also it emits lesser amounts of these elements during subjected leaching test.

## Conclusions

The study revealed that:

1. Addition of lime into metal chloride containing kaolin mixture increases the emissions of Pb and Cd during heat-treatment.
2. Pb and Cd chloride salts containing lime-kaolin mixtures form some calcium aluminosilicate based amorphous products during heat-treatment at 800°C. The amounts of Pb and Cd leached from this product are very low compared to total content.
3. The heat-treated product containing anorthite as the major crystalline phase can retain more amount of Pb and Cd during heat-treatment than the product containing gehlenite as the major crystalline phase. Dissolution of lesser amounts of Pb and Cd is observed from the products, where anorthite is the major mineral phase.
4. Compared to the addition of amorphous silica into the kaolin-lime mixture, addition of crystalline silica (quartz) enhances the emission of Pb and Cd during the heat-treatment of mixtures at low temperature ( $\leq 1000^\circ\text{C}$ ) and

decreases their emission during heating of mixtures at or above 1100°C.

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