



Hydrothermal processing of MSWI Fly Ash-towards new stable minerals and fixation of heavy metals

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ABSTRACT

A hydrothermal processing strategy of MSWI fly ash is presented for obtaining stable minerals with low toxic potential. Different hydrothermal conditions were tested to obtain high yields of new stable minerals. Experimental parameters including temperature, nature and molarity of alkali reagents, and reaction time were evaluated. The chemical stability of hydrothermal products was examined by the toxicity characteristic leaching procedure (TCLP) test and subsequent XRD for the leached residue. The significant amounts of Al-substituted 11 Å tobermorite and katoite in addition to minor amounts of zeolites were formed under experimental conditions at 0.5 M NaOH, 180 °C for 48 h, however KOH treatment in a similar regime resulted in smaller amounts of Al-substituted 11 Å tobermorite and katoite. Similarly, a product of mixed Al-substituted 11 Å tobermorite and katoite could be formed from the washed fly ash treated in 0.5 M NaOH at 180 °C for 48 h. Under the acidic condition, the treated fly ash exhibited an excellent stability of the mineral assemblage and less release of heavy metals relative to the untreated parent materials.

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1. Introduction

MSWI residues represent a by-product generated from municipal solid waste incineration and they are comprised primarily of: (i) bottom ash and (ii) fly ash [1,2]. Bottom ash is not classified as a hazardous waste according to the European Waste Catalogue, and it is explored frequently for constructional applications. It has been recycled commonly in Germany as a secondary building material for civil engineering applications. Utilization of fly ash, however, is at present realized in low technology applications such as additive in cement and construction materials. In Germany, disposal of fly ash is realized by wetting with water which leads to a puzzolanic solidification of the ash in the waste disposal site. Disposal or low level of ash utilization is inevitable due to the potential leachability of heavy metals (Cd, Zn, Pb, Hg, Cu, Cr and Ni) and toxic organic compounds to the environment [3–6]. In addition, fly ash is very highly reactive and subject to mineralogical alteration when it is in contact to the environment. The potential instability in the environment

makes it difficult for fly ash to compete with alternative raw materials. Hence for providing a possible utilization of fly ash, chemical stabilization of this material is required. This makes eventually a positive impact on the waste minimization and the reduction of waste accumulation at landfills [5].

Various technological options such as thermal treatment, physical/chemical separation, and stabilization/solidification (S/S) techniques are now available for treatments of fly ash in view of their reuse or final disposal [7–10]. The use of thermal treatment via vitrification has been tested and practised on a small scale. However, the method is considered more costly than other solutions because it requires a significant amount of energy and an expensive capital apparatus. It could also lead to a subsequent environmental destructive impact as a result of volatilisation of heavy metals from fly ash during vitrifying and melting processes.

As an alternative to the thermal treatment, physical/chemical separation methods employed for extracting heavy metals (Cd, Pb and Zn) from fly ash have been attempted but with limited success [11–13]. More recently, the stabilization and solidification (S/S) methods have emerged as a viable alternative for MSWI fly ash treatment by solidification with Portland cement into stable complexes [14]. In this context, toxic components are incorporated in a cement matrix through either physical or chemical immobilization mechanisms, depending on the particular contaminant to be fixed and type of binder being used. This technique has long been known as an economical option for the waste management strategy [14].

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However, the solidification with Portland cement presents some disadvantages. Specifically protection against humidity is required to prevent breakdown and leaching of heavy metals [15]. In addition, salts present in fly ash will interfere with basic hydration reactions of cement, leading to an inadequate set and/or deterioration of the waste form over time.

Pozzolanic solidification of MSWI fly ash in a solution enriched with $\text{Ca}(\text{OH})_2$ in order to take an advantage of its pozzolanic property has yet to be fully realized [16,17]. An interesting potential for generating a pozzolanic property of MSWI fly ash is that ettringite and calcium-silicate-hydrate (C-S-H) may be formed using an activator of $\text{Ca}(\text{OH})_2$ giving a good degree of stabilization of the hardened material. This stabilized material becomes, in principle, more compatible for storage, landfill or reuse as a resource.

Among other ash treatments, a hydrothermal method for processing fly ash with improved chemical stability could be considered as a promising technology, which offers considerable advantages in terms of economic, technical and environmental performance [5]. This method has been employed successfully for treating coal fly ash in alkali solutions (NaOH and KOH), [18], and may be also beneficially applied for MSWI fly ash. The value in using MSWI fly ash in the synthesis of zeolites is due to its high specific surface and high content of aluminosilicate glass [5], although this glass may be less abundant in MSWI fly ash than in coal fly ash. Further, the efficiency of hydrothermal processing depends upon the mass ratio of SiO_2 and Al_2O_3 -components in the glassy phase which has the greatest influence over the composition and type of stable minerals produced [19].

Recent publications on the hydrothermal treatment of MSWI fly ash have improved understanding considerably for new potential applications of fly ash as resources [20–22]. The use of MSWI fly ash for the synthesis of zeolite compounds (e.g., zeolites A and P and Sodalite) by the hydrothermal method in the NaOH solution has been demonstrated previously [20,22]. Here water washing was previously employed for the treatment of fly ash and followed by heating of fly ash at 800°C . This pre-treatment procedure is suggested to be an important part for the preparation of reproducible zeolitic compounds. Yao et al. [23] have reported the synthesis of tobermorite [$\text{Ca}_5\text{Si}_6(\text{OH})_2\text{O}_{16}\cdot 4\text{H}_2\text{O}$] from fly ash by hydrothermal treatment in NaOH solutions at 180°C , and those products revealed the uptake behavior for Cs^+ and NH_4^+ . More recently, a mixture of Al-substituted tobermorite-11 Å [$\text{Ca}_5(\text{Si},\text{Al})_6(\text{OH})_2\text{O}_{16}\cdot 4\text{H}_2\text{O}$] and katoite [$\text{Ca}_3\text{Al}_2(\text{SiO}_4)(\text{OH})_8$] has been synthesized from newsprint recycling residues under hydrothermal condition at 100°C in NaOH, LiOH and KOH solutions [24]. The resulting products of Al-substituted tobermorite-11 Å and katoite facilitated the immobilization of heavy metal ions and also exhibited an important ion exchange property for nuclear and hazardous wastewater conditioning. However, characteristics of MSWI fly ash, which are essential to provide a scientific basis and establish confidence in the technology of hydrothermal processes, remain poorly understood and lack quantification. This is probably because of complicated chemical and mineralogical character of fly ash. A detailed knowledge of mineralogical phase compositions (crystalline and glassy phases) of fly ash is necessary in order to be able to assess the correlation between microstructure and process parameter and thus, to optimize the stabilized products.

The present study was undertaken to access the benefits of hydrothermal processing of MSWI fly ash to stable phase minerals. The starting fly ash was hydrothermally treated in 0.5 M NaOH and KOH solution at temperatures ranging 90 – 180°C for 48 h. Parallel experiments were also conducted with the washed fly ash. These experimental conditions were chosen on the basis of our study [25] indicating that the best crystallinity of stable mineral phases (i.e., tobermorite-11 Å, and katoite) would be formed at such experimental conditions. Effects of temperatures on the processing

of amorphous and crystalline phases to stable mineral phases were analyzed by the Rietveld refinement method from X-ray diffraction (XRD) data. The resulting products were also characterized by scanning electron microscopy (SEM) for microstructure and morphology observation, and by the toxicity characteristic leaching procedure (TCLP) for testing of hazardous materials.

2. Experimental procedure

2.1. Materials

Approximately 5 kg of MSWI fly ash were collected from the electrostatic precipitator (ESP) facility at an incinerator plant of the town of Iserlohn close to the Ruhr industrial area, Germany. This sampled fly ash was then homogenized within 24 h after sampling with an agate vibratory disc mill for 30 min, until all material had passed a $100\ \mu\text{m}$ sieve. LOI was determined by heating the raw fly ash to constant weight at 750°C .

A pre-treatment of fly ash was conducted by water-washing in a glass beaker equipped with a magnetic stirrer for mixing at 300 rpm. Batches of 70 g of the fly ash powder were prepared in this way. They were initially placed in the glass beaker containing distilled water. The water to solid mass ratio (ml/g) was adjusted to be 10. Subsequently, this mixture was stirred to stand for 24 h

Table 1
Chemical composition of raw and washed fly ashes.

Element	Raw fly ash Wt.%	Washed fly ash
Si	5.63 (20) ^a	6.95 (25)
Ti	1.02 (02)	1.53 (05)
Al	2.43 (02)	3.73 (06)
Fe	3.71 (05)	3.99 (08)
Ca	12.41 (22)	18.97 (26)
Mg	1.08 (02)	1.57 (04)
Mn	0.12 (0)	0.18 (02)
K	6.11 (08)	0.99 (01)
Na	10.38 (28)	3.09 (07)
P	0.40 (0)	0.55 (0)
Cl	8.32 (14)	0.56 (0)
Pb	1.36 (07)	2.17 (09)
S	4.12 (0)	5.49 (0)
Zn	4.91(03)	8.09(08)
^b LOI	8.51(28)	
Element	Raw fly ash ppm	Washed fly ash
As	307 (25)	1029(30)
Ba	3470 (93)	5365(75)
Bi	204 (2)	364(5)
Cd	456 (6)	751 (7)
Co	262 (18)	265 (19)
Cr	2026 (29)	2924 (39)
Cs	114 (3)	184 (5)
Cu	3513 (32)	5660 (24)
F	1 (0)	5131 (28)
Ga	3042 (229)	43 (0)
Mo	30 (4)	609 (4)
Ni	9 (1)	799 (21)
Rb	34 (1)	27 (2)
Sb	3 (0)	2678 (2)
Sn	489 (7)	557 (8)
Sr	24 (4)	744 (6)
V	614 (19)	122 (14)
Y	6 (3)	1436 (24)
Zr	265 (3)	336 (7)

^a Figures in parentheses indicate the least-squares estimated standard deviation (esd) referring to the least significant figure to left, a zero indicates an esd < 0.05%.

^b LOI: loss on ignition.

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