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RESEARCH ARTICLE

Mechanochemical treatment of fly ash and de novo testing of milled fly ash

Yaqi Peng[1] · Alfons Buekens[1] · Minghui Tang[1] · Shengyong Lu[1]

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Abstract
Mechanochemical (MC) treatment has been widely proposed to degrade chlorinated organics in various matrix materials. In this study, fly ash from municipal solid waste incineration was grinded without any addition, using an all-dimensional planetary ball mill. The treated fly ash samples were characterised using X-ray diffraction, Raman spectra, scanning electron microscopy and X-ray energy-dispersive spectroscopy. The residual content of polychlorinated dibenzo-*p*-dioxins and dibenzofurans (PCDD/F) was monitored, as well as polycyclic aromatic hydrocarbons (PAH), a potential precursor of PCDD/F and amorphous carbon or graphite. Finally, de novo formation experiments were performed to test the chemical reactivity of the treated fly ash. The PCDD/F in milled samples was compared to those resulting from de novo tests on these same samples. The results suggest that both milling as well as de novo tests significantly alter the PCDD/F signature, suggesting substantial differences in the mechanisms of formation and destruction.

Keywords PCDD/F · PAH · Fly ash · Mechanochemical treatment · De novo

Introduction

Polychlorinated dibenzo-*p*-dioxins (PCDD) and dibenzofurans (PCDF) are a well-known threat to the environment (Stockholm Convention). They are generated in municipal solid waste incineration (MSWI) and other thermal processes. More than 80% of the total PCDD/F released from incineration are adsorbed onto fly ash (Weber et al. 2002). According to the material life cycle in a recent research, the treatment and reuse of fly ash can provide significant natural resources savings (Bontempi 2017). Thus, complete decomposition of the dioxins in ash has attracted lots of attention. Methods such as microbial degradation and UV irradiation require long treatment time, show low efficiency and are quite expensive and inefficient (Peng et al. 2010).

Mechanochemical (MC) methods have been proposed to treat halogenated organics. They require no heating or off-gas treatment and do not generate secondary pollution (Wei et al. 2009). Numerous compounds have been studied, including PCDD/F (Yan et al. 2007), chlorobenzenes (Mulas et al. 1997; Tanaka et al. 2003), pentachlorophenol (Wei et al. 2009), dechlorane (Zhang et al. 2010) and DDT (Hall et al. 1996). Some additives such as calcium hydride (CaH_2) and quartz (silica sand) have been tested to augment the reaction rate (Cao et al. 1999; Zhang et al. 2001). Many metal oxides (CaO, MgO) have been proved effective for the degradation of chlorinated compounds (Nomura et al. 2005; Tanaka et al. 2003). However, some researchers (Peng et al. 2010; Yan et al. 2007) milled fly ash directly, proving that metal compounds in the ash are also effective in dioxins degradation.

Nomura et al. (Nomura et al. 2005) studied the degradation of OCDD/F by MC treatment and found that dechlorination is an important pathway in the degradation mechanism; the dibenzo-*p*-dioxin and dibenzofuran structures dechlorinate, fracture and degrade further. A weight ratio of 200:1 (CaO/OCDD/F) was chosen for MC treatment and OCDD and OCDF reached a removal efficiency of 99.3 and 99.7% after 2 h (Nomura et al. 2005). A kinetic model was developed in a recent research, which showed that MC destruction of chlorinated compounds is nearly ten times faster than their

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✉ Shengyong Lu
lushy@zju.edu.cn

[1] State Key Laboratory of Clean Energy Utilization, Institute for Thermal Power Engineering, Zhejiang University, Hangzhou 310027, China

dechlorination (Lu et al. 2017). It is evident that MC destruction is the most relevant process.

In our previous work, the process and mechanism of trichlorobenzene degradation during MC treatment were studied; the benzene molecules formed during dechlorination recombined, forming amorphous and graphite-like structures (Lu et al. 2012b). Also, Cagnetta et al. summarised the MC destruction of halogenated organic pollutants and found the carbonisation of pollutant appearing in a lot of research (Cagnetta et al. 2016c). Though numerous studies focused on the effect and mechanisms of dechlorination and destruction by MC treatment, few considered the chemical reactivity of the carbon structures formed during the MC treatment. Earlier work has shown that the MC-degradation of PCDD/F may be jeopardised by the presence of inorganic chlorides in fly ash, so that their preliminary washing out would be necessary to ensure PCDD/F elimination and destruction (Chen et al. 2017b). In our work, the atomic ratio of Cl to Ca was 0.26, based on the characterisation results and measurement of the original fly ash. This seems sufficient for reacting with the Cl to be eliminated, so no more CaO was added.

In the present study, the MC treatment of fly ash was carried out without any additive or preliminary treatment and the destruction efficiency of both PCDD/F and PAH was estimated. The physical and chemical properties of the raw fly ash and MC treated fly ash were comprehensively characterised, and the evolution in fly ash composition during milling is considered.

Moreover, de novo synthesis experiments were performed to explore the chemical reactivity of the residual carbon structures in the treated fly ash.

Materials and methods

Fly ash and MC reactor

The fly ash used in this experiment was sampled from the baghouse filter of an externally circulating fluidised bed municipal solid waste incineration plant (Cixi, Ningbo), in which hydrated lime was injected into the flue gas, to neutralise the acid gases, as well as activated carbon, to adsorb PCDD/F. The original fly ash was dried at 80 °C for 24 h.

The MC reactor is an all-dimensional planetary ball mill (QXQM-2, Changsha Tencan Power Technology Co., Ltd., China) with four stainless steel pots (500 cm^3); each pot contained 330 g stainless steel balls and 15 g fly ash to be milled. The pots are fixed in a single planetary disc, and the milling pots not only rotate together with the disc, but also around their own axis. The direction of the disc revolution and the autorotation of pots are opposite. Compared to the conventional planetary ball mill, all-dimensional unit can make the disc and all pots rotate (1 rpm) around a main spindle to avoid the sedimentation of fly ash caused by gravity and making powders to be ground more completely. Revolution speed was set at 350 rpm, and the machine stops every 15 min for 5 min in case the pots are overheated. After running for 0, 1.5, 3 and 6 h, 1 g of the ground fly ash was sampled and denoted as samples FA_0, $FA_{1.5}$, FA_3 and FA_6.

The de novo experiments

A laboratory scale reactor placed in a vertical furnace is used to conduct a de novo test and investigate its PCDD/F reaction product as shown in Fig. 1. The experiments were conducted in a quartz tubular reactor, operating substantially at atmospheric pressure and surrounded by an electric furnace. Both N_2 and O_2 flows were controlled by mass controllers, and water was injected into the tube at a stable rate by a pump and heated at 120 °C and then mixed with the gases at a ratio of O_2, H_2O and N_2 of 10:5:85. About 1 g of either raw fly ash FA_0 or milled fly ash $FA_{1.5}$, FA_3 and FA_6 was positioned onto a silica wool carrier to establish full contact with the mixed carrier gas. The flow rate of the mixed gas was 300 ml/min. The reaction temperature was set at 350 °C. The PCDD/F present in the off-gas was continually collected by toluene in the ice bath for 1 h. Also, after each experiment, the quartz tube and the fly ash were rinsed and Soxhlet extracted with toluene for the determination of residual PCDD/F.

Analytical methods

All original and MC treated fly ash were measured for the following structural and compositional parameters. The crystalline structures were identified using X-ray diffraction (XRD) (X'Pert PRO, PANalytical Co., Holland). The micromorphology of the samples was analysed by a field emission scanning electron microscopy (SEM) (SU-70, Hitachi, Japan). The structure of the carbon was determined by Raman spectroscopy (LabRamHRUV Jobinyvon Co., France). The chemical composition of fly ash was analysed by X-ray fluorescence (XRF) and energy-dispersive spectrometry (EDS) (EDAX-GENESIS4000, USA).

The concentration of PCDD/F in residue was determined after separation by high resolution gas chromatography (Agilent 7890A) combined with high-resolution mass spectrometry (JMS-800D, JEOL). The procedure used for cleanup was as in USEPA 1613. The column information and the temperature program were already described by Zhao et al. (2016). The original, MC-treated and de novo fly ash were all analysed for PCDD/F.

The concentrations of PAH in raw and MC treated fly ash were analysed by gas chromatograph/mass spectrometer (GC-MS, JMS-Q1050GC, JEOL), and the sample pre-treatment was conducted in accordance with the HJ 646-2013 method in China. Detailed information of the GC-MS and the procedure of pre-treatment are shown in Li et al. (2016).

Conversion and chlorination levels

The degradation efficiency of PCDD/F was calculated by Eq. (1) and Eq. (2). $\eta_{S\text{total}}$ and $\eta_{S\text{ITEQ}}$ are degradation efficiency of PCDD/F for total concentration and International Toxic Equivalents (I-TEQ) concentration, respectively. And C (ng/g) and C' (ng-TEQ/g) are PCDD/F total (through tetra-octa chlorinated PCDD/F homologue) and I-TEQ concentrations.

$$\eta_{S\text{total}} = \frac{C_{\text{FA0}} - C_S}{C_{\text{FA0}}} \times 100\%, \quad S = 0,\ 1.5,\ 3,\ 6 \tag{1}$$

$$\eta_{S\text{ITEQ}} = \frac{C'_{\text{FA0}} - C'_S}{C'_{\text{FA0}}} \times 100\%, \quad S = 0,\ 1.5,\ 3,\ 6 \tag{2}$$

To study the chlorination level of PCDD/F in the samples, the degree of chlorination (d_c) was calculated by Eq. (3), which is defined as the sum of the products of the mole fraction f_i and the number of chlorine atoms n_i for each homologue.

$$d_c = \sum_{i=4}^{8} f_i \times n_i \tag{3}$$

Results and discussion

First, the results of MC treatment are discussed for raw and milled samples, before (FA_0) and after treatment for 1.5, 3 and 6 h ($FA_{1.5}$, FA_3, FA_6). The evolution of PCDD/F is monitored in amount (ng/g fly ash), homologue distribution and mole averaged chlorination level.

Note that the amount of fly ash, in grammes, may have evolved during MC treatment, as well as during de novo testing. The first evolution is further examined under elemental composition; the second leads to a partial oxidation of the amorphous carbon and graphite present, with some concomitant weight loss (Stieglitz and Vogg 1987). Next, these four samples FA_0, $FA_{1.5}$, FA_3 and FA_6 are de novo tested. PCDD/F again are examined in amount (ng/g residual fly ash), homologue distribution and mole averaged chlorination level.

Physicochemical characteristics

Element composition The composition of the original and milled fly ash is shown in Table 1.

The main elements are those of the earth crest, i.e. oxygen, also present in silica, alumina and in calcium, magnesium and iron oxides. A second group of elements is associated either with flue gas pollutants, such as salts of Na and K; or P, S and Cl; or with their treatment by injection of hydrated lime, introduced to control acid gases or of activated carbon (AC) to capture PCDD/F. Titanium oxide and titanites derive from the presence of inorganic pigments in plastics, paints and paper. Carbon derives from unburned carbonised matter and the injection of AC. The addition of hydrated lime inflates the initial amount of Ca in fly ash.

Several elements markedly rise during MC treatment: Fe (+ 47%), Si (+ 37%), Na (+ 36%), Al (+ 16%), Cu (+ 25%), Ni (+ 780%), Cr (+ 1258%) and Mn (+ 217%). Conversely, other elements seem to disappear from the fly ash matrix: P (− 46%), Cl (− 37%), C (− 35%), Mg (− 28%), Ca (− 23%), S (− 19%) and also Ti (− 13%). For these evolutions, only partial explanations may be offered:

1. Already in an earlier study, the appearance of Fe, Cu, Ni, Cr, Mn… was established during milling, apparently due to the attrition of mixing balls and vessel. Present results show that milling treatment cannot eliminate heavy metals or other elements efficiently; rather, it can increase their percentage (Cheruiyot et al. 2016).
2. Some elements, such as C, Cl, S, P and O, are apparently capable of generating gaseous effluents, or else reporting to these, following various reactions and heating (Lu et al. 2012a).

At this point of the analysis, two essential elements are still lacking, so that this scrutiny cannot be completed at present: the maximum temperature attained during milling and the gas phase composition being established inside the mill.

The elements constituting PCDD/F are C, Cl, O and H; among these, both C and Cl are dwindling during MC treatment. Another group of essential elements are those exerting a catalytic activity, e.g. Cu, Cr, Ni, Mn… (Peng et al. 2016). Catalytic activity is closely linked to the quality of the contact between the reactants, as well as to the mobility of the catalytic species (Kuzuhara et al. 2003). Milling reduces the average grain size of fly ash and thus enhances the chemical reactivity of its matrix. The latter was scrutinised as raw fly ash and after milling for 1.5, 3 and 6 h, using various instrumental analyses.

Raman spectrum Two peaks appear in all samples at a wavelength of 1580 and 1355 cm^{-1} in the Raman spectrum (Fig. 2). These are characteristic of graphite and amorphous carbon: the peak at 1580 cm^{-1} represents the G bond, corresponding to graphite, and a broad peak at 1355 cm^{-1} corresponds to the characteristic D bond peak of amorphous carbon (Rowlands et al. 1994; Zhang et al. 2010).

Tanaka et al. milled mixtures of CaO and aramid (an aromatic polyamide) and found that amorphous carbon and graphite appeared already after 1 h in the milled sample and

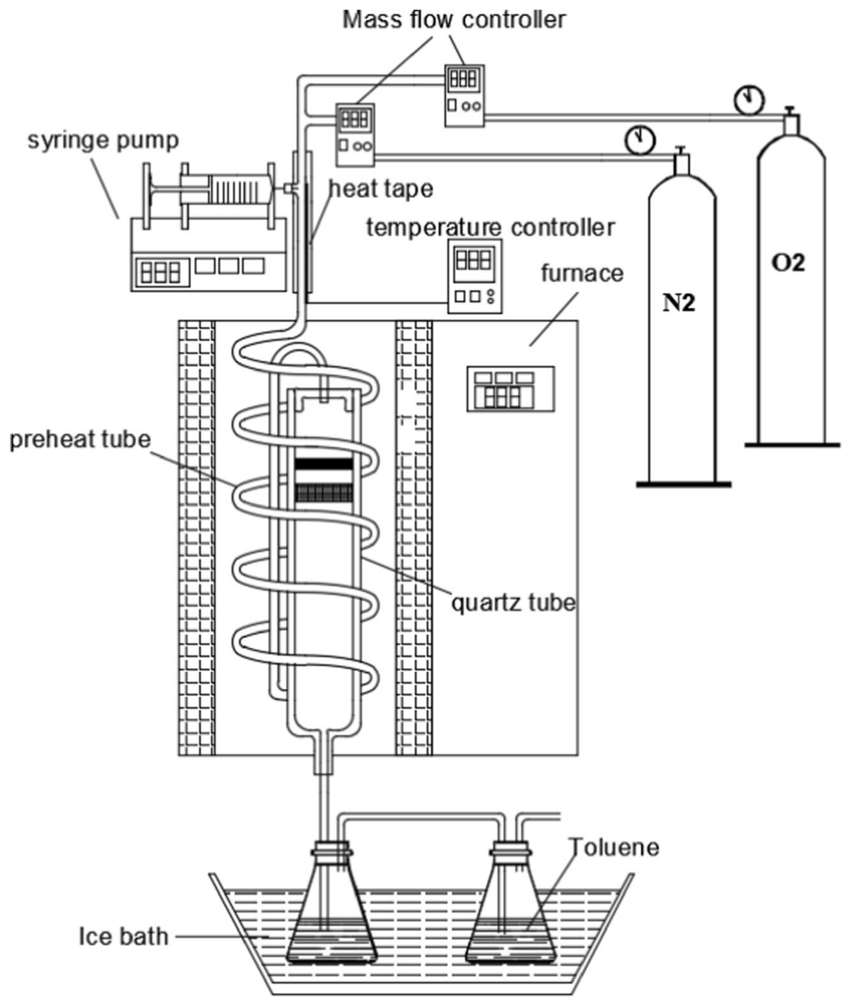


Fig. 1 Schematic diagram of the de novo tests

distinctly intensified with time rising (Tanaka et al. 2003). In the present experiment, the fly ash colour deepened with MC treatment proceeding and finally turned black; therefore, graphite is expected to be the final degradation product. To verify the hypothesis, the I_D/I_G value in the Raman spectrum was calculated. This value decreased from 2.42 to 1.76 with time increasing, indicating that the fraction of graphite augmented.

SEM Figure 3 shows a SEM photography (× 1000 times) of untreated and MC treated fly ash. These are taken to facilitate the understanding and a comparison of processes influencing upon ash surface structure. Clearly, before milling, raw fly ash still consists of many large and diverse particles. After several hours of milling, the overall particle size has decreased markedly, while showing a more uniform distribution of concave surfaces, as well as of aggregation of small particles.

Table 1 Elemental composition (wt.%) of the raw and milled fly ash

	C	O	Na	Mg	Al	Si	P	S	Cl
FA_0	13.24	32.58	1.35	1.82	8.04	13	0.97	1.81	4.04
$FA_{1.5}$	10.95	32.47	1.72	1.7	9.01	16.69	0.73	1.62	3.29
FA_3	9.73	33.34	1.44	1.48	9.18	17.52	0.59	1.46	2.87
FA_6	8.55	32.61	1.83	1.31	9.35	17.8	0.52	1.46	2.55
	K	Ca	Ti	Fe	Cu	Ni	Cr	Mn	
FA_0	1.87	17.28	0.7	3.3	0.052	0.005	0.017	0.076	
$FA_{1.5}$	2.05	15.52	0.62	3.63	0.053	0.016	0.074	0.11	
FA_3	1.98	14.44	0.74	4.44	0.054	0.019	0.081	0.118	
FA_6	1.93	13.31	0.61	4.84	0.065	0.044	0.231	0.241	

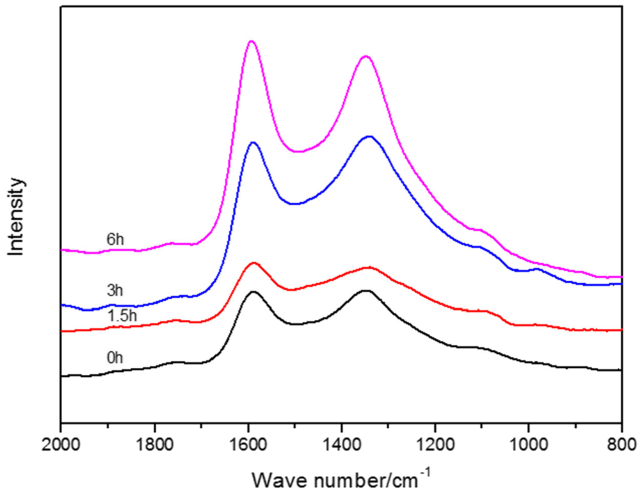


Fig. 2 Raman spectrum of raw and milled fly ash samples

XRD In order to identify possible changes in the presence of crystalline phases, XRD was carried out on both raw and MC treated fly ash (Fig. 4). Raw fly ash includes as major minerals: quartz SiO_2, calcite $CaCO_3$, halite KCl, anhydrite $CaSO_4$, haematite Fe_2O_3, vaterite, a polymorph of $CaCO_3$ and gypsum $CaSO_4·2H_2O$; after MC treatment, the peak intensity of calcite $CaCO_3$ and anhydrite $CaSO_4$ weakened while that of gypsum $CaSO_4·2H_2O$ increased, suggesting hydration to take place, as well as potentially placing a maximum to the temperature attained during milling, since the transition to gypsum takes place only below 120 °C. SiO_2 remains inert, without any phase transition during milling.

In the present tests, the crystalline phase of Ca(OH)Cl remained undetected. In another study on trichlorophenol (TCP) degradation, unobservable formation of crystalline phase Ca(OH)Cl suggests that the inorganic chlorine formed remains amorphous (Lu et al. 2012b). Saeki treated PVC mixed with $CaCO_3$, and a chlorine-containing inorganic phase Ca(OH)Cl was observed with rising grinding time (Saeki et al. 2001). Also, in the present study, it is supposed that Ca(OH)Cl forms a non-crystalline amorphous phase.

MC treatment of PCDD/F and PAH

Destruction of total PCDD/F and I-TEQ The total PCDD/F concentration and I-TEQ of the raw fly ash were 6.45 ng/g and 0.213 ng I-TEQ/g, respectively.

Figure 5 monitors the evolution of PCDD, PCDF, PCDD/F and I-TEQ concentration during the MC treatment. After 6 h of milling, total concentration and I-TEQ finally reduced by 41.7 and 30.6%, respectively. During degradation, different

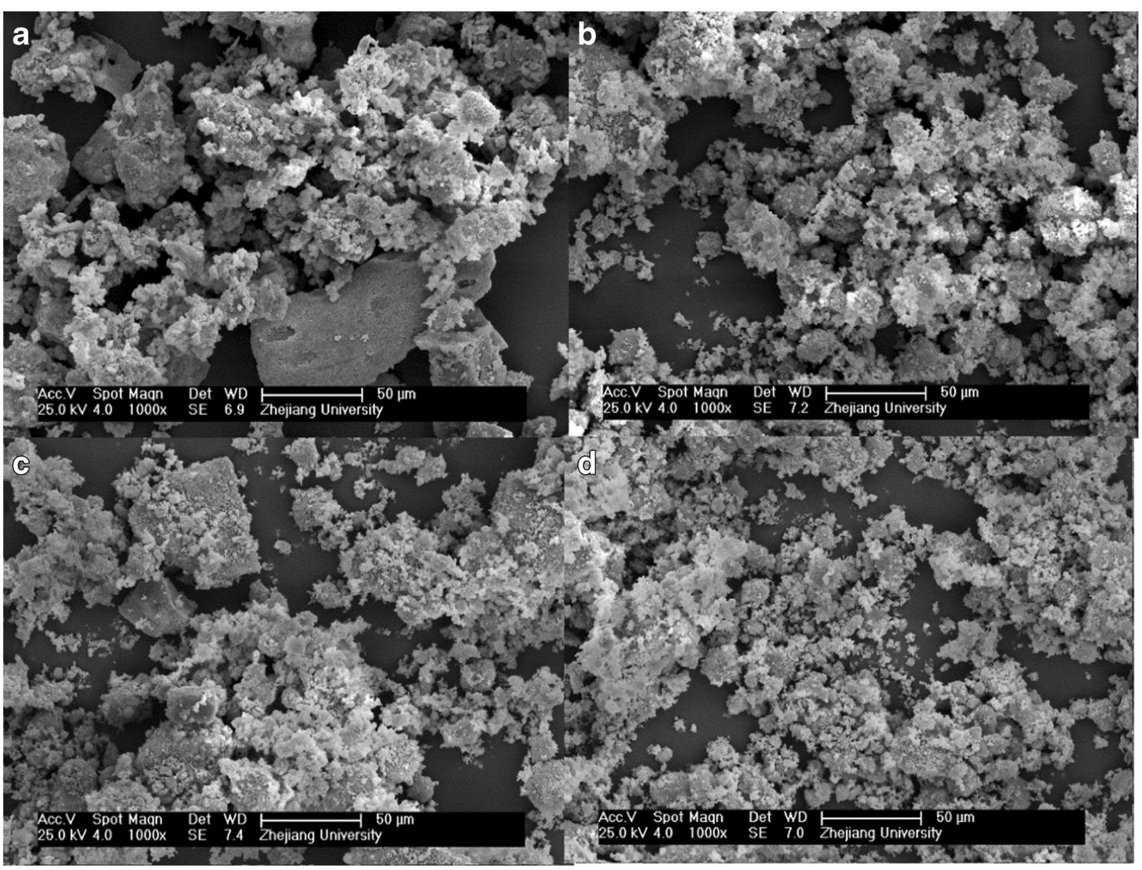


Fig. 3 SEM micrographs for **a** raw fly ash magnified at × 1000 (1 k); **b** FA after 1.5 h milling, also at × 1 k; **c** FA after 3 h milling, at × 1 k; and **d** FA after 6 h milling, at × 1 k

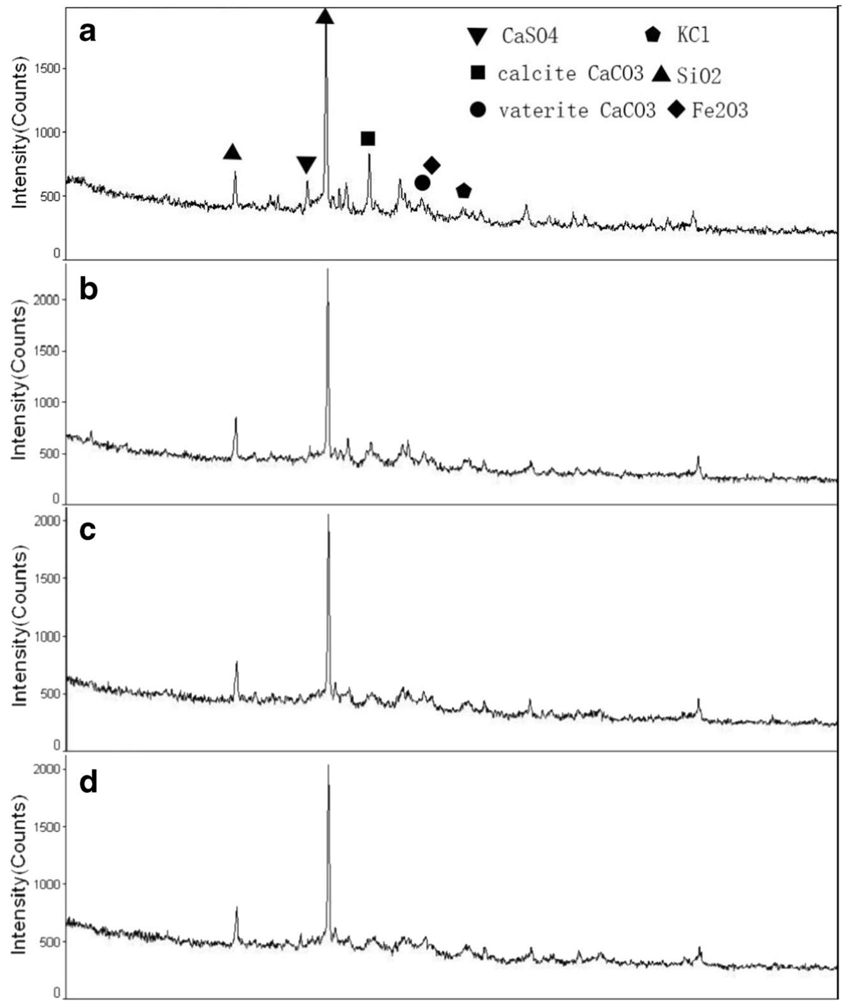


Fig. 4 XRD pattern of **a** raw fly ash and milled fly ash after **b** 1.5 h, **c** 3 h, and **d** 6 h

trends appeared for PCDD/F and I-TEQ. Figure 6 shows the tetra- to octa-CDD/F homologue profiles, both for original and MC treated FA.

Most studies on the MC treatment of chlorinated aromatic compounds showed that calcium oxide is an efficient additive (Nomura et al. 2005; Saeki et al. 2001;

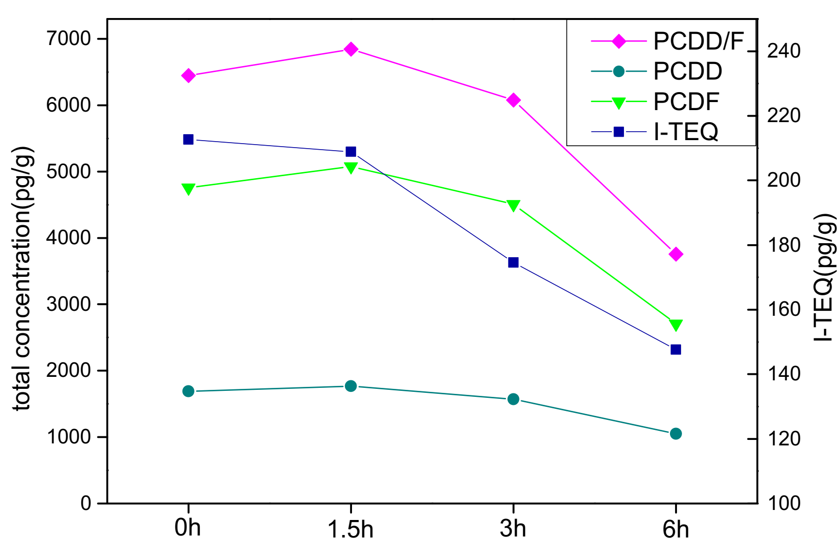
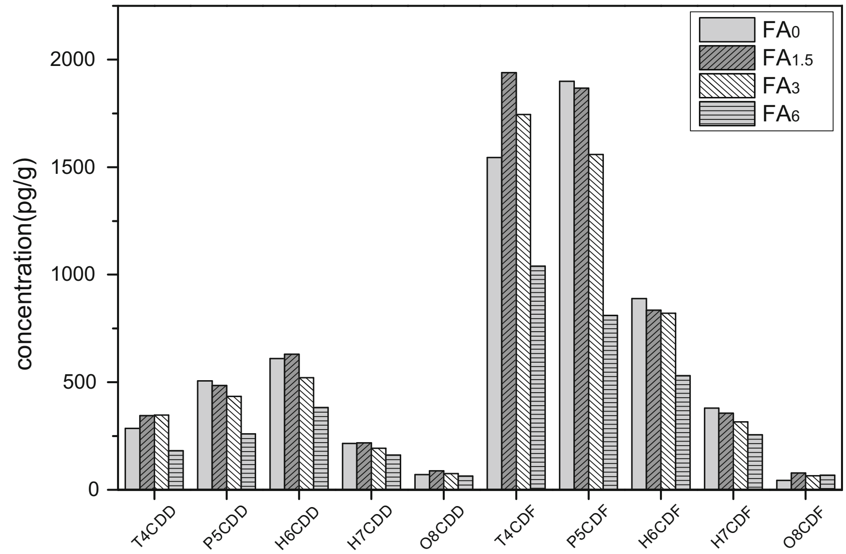


Fig. 5 Time trends for PCDD/F with milling time increasing

Fig. 6 Homologue pattern of PCDD/F for raw fly ash (FA_0) and MC treated fly ash ($FA_{1.5}$, FA_3, FA_6)



Tanaka et al. 2003). As described in a recent work, vacancy formation reaction can even happen to CaO under high energy ball milling (Cagnetta et al. 2017). In this reaction, two electrons were released and transferred to organic molecules, thus inducing carbonisation and dehalogenation (Todres 2006). While in the present experiments, free CaO was not detected, as by Peng et al. (2010). Hence, CaO may act as an intermediate inducing dechlorination.

One of the reasons for degradation of chlorinated organic compounds is the breaking of C-Cl bonds, namely dechlorination (Lu et al. 2012b). Thus, lower chlorinated intermediates are probably produced while higher chlorinated PCDD/F degrade. Nomura et al. (2005) observed tetra- to hepta-CDD/F, formed from MC treatment of OCDD/F, to peak simultaneously and decrease with rising grinding time. The same was also reported by Mitoma et al. (2011), while no additives were added during MC treatment.

In Fig. 6, the amount of TCDD/F first increased from 0 to 1.5 h, which suggests additional formation from PCDD/F precursors in fly ash, such as chlorinated organic compounds (chlorobenzenes, chlorophenols) or PAH, accompanied by dechlorination of high chlorinated PCDD/F. While in 6 h, the destruction efficiency of OCDD/F was much lower than those of tetra- to hepta-CDD/F. Maybe it is due to the low concentration of OCDD/F, which is one or two orders of magnitude lower than others.

Table 2 Degree of chlorination of PCDD/F for raw and MC milled sample

FA_0	$FA_{1.5}$	FA_3	FA_6
4.72	4.67	4.66	4.62

With rising grinding time, the degree of chlorination lessened marginally, from 4.72 for FA_0 to 4.62 for FA_6, as shown in Table 2, which is in agreement with dechlorination mechanism. Based on our previous study (Lu et al. 2012b), Cl radicals have a chance to replace H on the benzene ring, which may be the reason why OCDD/F increased in 1.5 h.

Destruction of PAH PAH could be an important carbon source and precursor for PCDD/F formation, via a variant of the de

Table 3 Amount of PAH (ug/g) in the raw and the MC milled fly ash

	Sample			
	FA_0	$FA_{1.5}$	FA_3	FA_6
Naphthalene	0.32	0.22	0.18	0.18
Acenaphthylene	0.04	0.02	0.02	0.01
Acenaphthene	0.01	0.01	0.01	0.01
Fluorene	0.03	0.03	0.03	0.03
Phenanthrene	0.39	0.27	0.27	0.26
Anthracene	0.37	0.25	0.25	0.25
Fluoranthene	0.51	0.37	0.27	0.21
Pyrene	0.34	0.22	0.16	0.13
Benzo(a)anthracene	0.23	0.09	0.07	0.05
Chrysene	0.09	0.09	0.08	0.08
Benzo(b)fluoranthene	0.23	0.20	0.07	0.07
Benzo(k)fluoranthene	0.21	0.18	0.07	0.07
Benzo(a)pyrene	0.08	0.08	0.06	0.04
Indeno(1,2,3-cd)pyrene	0.05	0.04	0.04	0.04
Dibenzo(a,h)anthracene	0.06	0.06	0.06	0.06
Benzo(g,h,i)perylene	0.04	0.03	0.03	0.03
Total PAH	3	2.16	1.66	1.52
PAH-TEQ	0.219	0.196	0.149	0.127

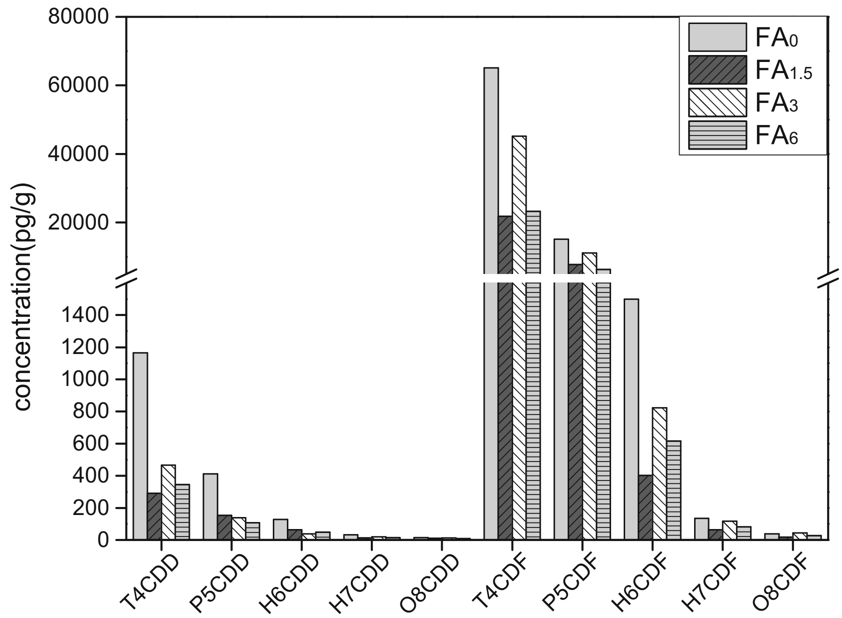


Fig. 7 Homologue pattern of PCDD/F formed from milled residue after de novo reaction

novo synthesis (Peng et al. 2016). Hence, the 16 US EPA PAH were analysed in milled fly ash. With rising grinding time, total PAH and their TEQ concentration diminished obviously, to 49.3 and 41.7%, respectively, after 6 h of treatment as shown in Table 3. The lower degradation rate, compared to the study by Cangialosi et al. (2007), may be due to the different mills used in the experiments. The rate constant and energy dose of monoplanetary are larger than those of all-dimensional planetary mill, which lead to the high conversion efficiency on the base of the kinetic models (Cagnetta et al. 2016b; Chen et al. 2017a).

De novo formation from milled samples

The particle size of fly ash, the amount and species of organic compounds, as well as the structure of the carbon formed all modify during milling; hence, the reactivity of carbon was explored experimentally during four de novo tests at fixed conditions of 350 °C in 1 h.

Figure 7 shows the PCDD/F yield (ng/g) from a de novo synthesis test, conducted on the four samples of fly ash, i.e. the original sample, as well as those obtained after 1.5, 3 and 6 h of milling. Remarkably, the yield from milled fly ash is much lower than that from raw fly ash: after 6 h of milling, the total concentration and I-TEQ value for FA_6 attains just 36.8 and 45.4%, respectively, of that obtained from raw fly ash. As shown in Table 4, the ratio of PCDF/PCDD rises remarkably with grinding time, from 2.6 to 48, 56 and 59, suggesting that de novo reactions starting from carbon or graphite become much more predominant and that precursor routes dwindle in importance. The mole average degree of chlorination of the PCDD/F synthesised from milled fly ash is very low.

According to a recent review (Peng et al. 2016), carbon in fly ash consists of aliphatic and aromatic compounds and macromolecular carbon. Due to the weak C-C bond energy, hydrocarbon chains can be broken easily and the resulting molecules volatilise (Tanaka et al. 2003). Also, some benzene is formed during ball grinding, and condenses, forming amorphous and graphite-like structures, which led to the colour of the samples turning black (Lu et al. 2012b).

Actually, the formation of unintentional persistent organic pollutants (POPs) was also observed by other research when PCDD/F are destroyed during ball milling (Cagnetta et al. 2016a). These POPs can take part in the de novo reactions, increasing the formation of PCDD/F. To decrease the reformation of PCDD/F, prolonged milling is needed to treat the fly ashes.

Table 4 PCDD/F formed after de novo reaction

	d_c	PCDD (ng/g)	PCDF (ng/g)	PCDF/PCDD (−)	I-TEQ (ng/g)
FA_0	4.21	1.75	82	2.6	1.04
$FA_{1.5}$	4.27	0.53	30	48	0.48
FA_3	4.21	0.68	57.2	56	0.51
FA_6	4.24	0.53	30.3	59	0.47

Conclusion

This work investigated the behaviour of the PCDD/F and PAH contained in fly ash during mechanochemical treatment without any addition of reductive or other milling agents. The results obtained demonstrated that the PCDD/F of fly ash decreased after MC treatment for 6 h, both in terms of total and I-TEQ concentration, with lower chlorinated PCDD/F being formed at the initial stages of milling. About half of the PAH were eliminated after MC treatment. The MC treatment also increases the proportion of graphite in the fly ash and reduces the reactivity of the carbon structures present to form PCDD/F through de novo synthesis.

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