Pre-treatment process for dioxin testing: This study entailed the determination of dioxins in solid samples of fly ash before and after the experiment, as well as the collection and determination of dioxins in gas samples after the experiment. The method of collecting dioxins in gas samples was by absorption using toluene and XAD-2 resin. Before the determination of dioxins in the gas-solid phase, it is necessary to transfer the dioxins from the gas-solid phase to the liquid solvent for detection, and to remove other impurities that may interfere with the detection of dioxins in order to improve the accuracy of the detection, so the samples need to be pretreated. The method of sample pretreatment refers to the "Determination of dioxins in solid waste isotope dilution high-resolution gas chromatography - high-resolution mass spectrometry" (HJ77.3-2008), the use of the U.S. EPA1613 method of operation, mainly including sampling, extraction, purification, concentration and pending detection of the 5-steps, the process is shown in Figure 2.3. The specific steps are as follows: This study requires the determination of dioxins in the solid samples of fly ash before and after the experiment, as well as the collection and determination of dioxins in the gas samples after the experiment. The method of collecting dioxins in the gas samples was to utilize toluene and XAD-2 resin for absorption. Before the determination of dioxins in the gas-solid phase, it is necessary to transfer the dioxins from the gas-solid phase to the liquid solvent for detection, and to remove other impurities that may interfere with the detection of dioxins in order to improve the accuracy of the detection, so the samples need to be pretreated. The method of sample pretreatment refers to "Determination of Dioxin in Solid Waste Isotope Dilution High Resolution Gas Chromatography - High Resolution Mass Spectrometry" (HJ77.3-2008), which is operated by the method of U.S. EPA1613, and mainly includes five steps: sampling, extraction, purification, concentration and pending detection. The specific steps are as follows:

Sampling: accurately weigh 1.00 g of fly ash sample or XAD-2 resin into a glass fiber cartridge, add the extraction internal standard (EPA-1613LCS), place the cartridge over a dispensing funnel fitted with a funnel, treat with hydrochloric acid until no bubbling occurs, and wash the acid-treated sample with tertiary water.

Toluene samples do not require the addition of hydrochloric acid and are treated by adding the ¹³C extraction standard directly.

Extraction: The treated liquid is extracted with liquid-liquid extraction with dichloromethane, and a certain amount of toluene is added after the extraction is concentrated by rotary evaporation, and the dried solid sample after hydrochloric acid treatment is subjected to soxhlet extraction, and the extracted solution is the toluene added by liquid-liquid extraction and concentration.

Purification: The extract was dissolved into n-hexane solvent and concentrated to 1 mL~2 mL by rotary evaporator, and ¹³C purification standard was added, and purified by multilayer silica gel column (sequentially filled with quartz cotton, 1 g of activated silica gel, 4 g of 2% potassium hydroxide silica gel, 1 g of activated silica gel, 8 g of 33% sulfuric acid silica gel, 1 g of activated silica gel, 3 g of 10% silver nitrate silica gel, and 2 cm of anhydrous sodium sulfate), and then rinsed with 150 Then the silica gel column was rinsed with 150 mL of hexane, and the hexane rinsate was collected.

Concentration: The eluent obtained in the previous step was concentrated and transferred to an activated carbon dispersed silica gel column for further purification. The column was eluted with 80 mL of a mixture of dichloromethane and hexane (1:3, v/v) and discarded, and then the activated carbon dispersed silica gel column was inverted and eluted with 40 mL of toluene solvent, and the toluene elution fraction was collected.

To be examined: Concentrate the toluene fraction to less than 1 mL, transfer the concentrated extract into a nitrogen blowing tube, and rinse three times with n-hexane and transfer to a nitrogen blowing tube, use high-purity nitrogen to blow slowly until nearly dry, add ¹³C into the internal standard, and nonane to 20 μ L, vortex mixing, and then transferred to the sample vials, frozen and stored to be analyzed by the instrument.

	Dioxin	Concentration (ng/kg)	I-TEF	Toxicity
	DIOXIII	Concentration (ng/kg)		(ng I-TEQ/kg)
Low-chlorinated PCDF congeners	2,3,7,8-TCDF	396.67	0.1	39.67
	1,2,3,7,8-PeCDF	650.00	0.05	32.67
	2,3,4,7,8-PeCDF	566.67	0.5	283.33
	1,2,3,4,7,8-HxCDF	606.67	0.1	60.67
High-chlorinated PCDF congeners	1,2,3,6,7,8-HxCDF	690.00	0.1	69.00
	2,3,4,6,7,8-HxCDF	523.33	0.1	52.33
	1,2,3,7,8,9- HxCDF	193.33	0.1	19.33
	1,2,3,4,6,7,8-HpCDF	1400.00	0.01	14.00
	1,2,3,4,7,8,9-HpCDF	276.67	0.01	2.77
	OCDF	613.33	0.001	0.61
Low-chlorinated	2,3,7,8-TCDD	48.00	1	48.00
PCDD congeners	1,2,3,7,8-PeCDD	143.33	0.5	72.33
High-chlorinated PCDD congeners	1,2,3,4,7,8-HxCDD	91.67	0.1	9.17
	1,2,3,6,7,8-HxCDD	173.33	0.1	17.33
	1,2,3,7,8,9-HxCDD	146.67	0.1	14.67
	1,2,3,4,6,7,8-HpCDD	1200.00	0.01	12.00
	OCDD	2566.67	0.001	2.57
Total	PCDD/Fs	10286.33	-	750.45

Table S1. Concentrations and toxicity of dioxins in fly ash (FA)

		XRF		XRD		
Ingredients	CaO	Cl	K ₂ O	NaCl	KCl	
Content (%)	29.26±0.66	16.62±0.32	7.97±0.15	21.00±1.41	18.33±1.25	
Ingredients	SO ₃	РЬО	Na ₂ O	SiO ₂	AlO(OH)	
Content (%)	7.12±0.11	4.85±0.20	3.95±0.11	8.33±0.47	9.33±1.25	
Ingredients	SiO ₂	Fe ₂ O ₃	ZnO	Ca(OH) ₂	CaSO ₄	
Content (%)	3.19±0.15	3.12±0.03	1.89±0.10	7.00±0.82	13.67±0.47	
Ingredients	Al ₂ O ₃	TiO ₂	P_2O_5	CaCO ₃	CaClOH	
Content (%)	0.78±0.18	0.62±0.02	0.33±0.01	8.00±0.82	14.33±0.47	

Notes: The components in the left two columns of this table are determined by the XRF method and the data in the right three columns are determined by the XRD method.