

Application of Microwave Digestion in the Determination of Elements in Coal Fly Ash

1. Introduction

Coal fly ash is generated during the burning of the fuels. During the process of converting coals to gas, the manufacturer produces large amount of coal fly ash as byproduct which neither have economic values nor can be re-circled for further usage. However, the pollution caused by disposal of coal fly ash is obvious. It will cause sever acid rain, air pollution and soil pollution because of the potential heavy metals, boron composite and other poison components. With the development of recycle technology and the publish of the coal fly ash disposal strategy, the coal fly ash can be regard as a unique material with the potential to be introduce to the concept of circular economy. The research on the migration and transformation of heavy metal inside coal fly ash, the solidify of coal fly ash to construction material or the assessment of environmental health exposal risk need the accurate determination of metal composition and chemical components. However, the complexity of the coal fly ash composition such as containing high amount of silicon-aluminum oxides, clay, metal oxides, or silicon restricts the common digestion method. Here, we present a microwave complete digestion method as sample pre-treatment for metal analysis. The method has the advantages of high efficiency, good digestion effect and low blank value.

2. Instrument and reagents

Instrument:

The digestions were carried out with M6 microwave digestion system and HP16 high pressure digestion vessels. The determination of the trace element was conducted by ICP-OES.



M6 microwave digestion system



HP16 rotor



G-160 hot block

Reagent:

HNO₃(GR) ; HCl (GR) ; HF (GR)

Sample:

Coal fly ashes certified quality control sample

3. Method

1. Weigh 0.1 g coal fly ash quality control samples in to sample cup.
2. Add aqua regia (HCl: HNO₃ =3:1) and HF into the sample cup swirl the cup to mix the sample and acid thoroughly.
3. Add same amount of acid into the sample cup as sample blank, then seal the vessel.
4. Set the microwave digestion program as shown in the following table:

Table1: Microwave digestion program

Step	Setting temperature(°C)	Ramp time (min)	Temperature holding (min)
1	140	10	5
2	180	8	5
3	220		30

5. Take the vessels out of the cavity when the temperature falls under 60 °C.
6. Open the vessels and place them on the hot block to evaporate acid at 180 °C. Dilute the sample to 50 mL with deionized water when the temperature of the sample cools to room temperature.
7. The final solutions were tested by ICP-OES according to HJ 781-2016 Solid Waste Determination of 22 metal elements- Inductively coupled plasma optical emission spectrometry.

4. Result

Table2: ICP-OES measurement for coal fly ash

Element	Certified value (mg/Kg)	Found value (mg/Kg)	Recovery (%)
Cr	60±4	55.9	93.2
Cu	53±2	49.3	93.0
Pb	33.8±2.2	37.8	112

As shown in the table 2, the method presents good accuracy in the determination of heavy metals inside coal fly ash.

5. Conclusion

Preekem's M6 microwave digestion system coupled with HP 16 rotor can digest even "difficult" sample as coal fly ashes to a clear solution, which is an assurance to the accurate ICP-OES analysis. It is a proof that M6 equipped with high pressure rotor can be applied in the study of complete digestion of coal fly ashes. With the advanced full vessel IR temperature monitor system and pressure control unit, M6 can ensure the safe and precise sample digestion during the experiment.