The application of microwave digestion in the testing of elements in Kaoline

1. Introduction

Kaoline also called China clay, a kind of non-metallic mineral with the crystal chemical formula as $2SiO_2 \cdot Al_2O_3 \cdot 2H_2O$. It has been widely used in ceramic industry, papermaking and refractory insulating material. It is an important and common clay mineral in nature. Based on the genesis of kaolin deposits, geographical conditions and mineralization geology, different kaoline occurs various characteristics and can be applied for different usage. The analysis as morphology, composition and ore minerals are vital parameters in determining the usage of kaoline. For examples, the refractoriness is related to the chemical composition of kaoline. The refractoriness of high purified kaolin is generally about 1700℃. When the content as potassium, sodium and iron is high, the refractoriness of the material is compromised. So it is vital to determine the chemical contents inside Kaoline for the related products' quality assessment. Depending on the composition of kaoline, the microwave digestion offers an attracting way for efficient sample preparation in elemental analysis.

2. Instrument and reagent

The digestions were carried out with M6 microwave digestion system and HP16 high pressure digestion vessels.

M6 microwave digestion system

HP16 rotor

Reagent:

HNO_3(GR) ; HF（GR）; saturated boric acid

3. Method

1. Weighed 0.2g sample into sample cup.
2. Add nitric acid and hydrofluoric acid into the sample Then mix the sample by swirling the cup gently.
3. Seal the vessels and set the microwave digestion program as follows:

Table 1: microwave digestion program

Step	Setting temperature(℃)	Ramp time (min)	Temperature holding (min)
1	120	10	2
2	180	8	2
3	220	8	30

4. Take the vessels out of the cavity when the temperature falls under 60 ℃.
5. Add saturated boric acid into the sample cup for complexing the F^-. Under normal condition, the adding volume of boric acid versus hydrofluoric acid should be 6:1.
6. Seal the vessel again then conduct microwave digestion program as shown in Table 2.

Table 2: microwave digestion program

Step	Setting temperature(℃)	Ramp time (min)	Temperature holding (min)
1	120	10	2
2	180	8	15

7. After that, take the vessels out of the cavity when the temperature falls under 60 ℃.
8. Dilute the sample with deionized water for further test.

4. Result and discussion

The final digestion solution is clear and transparent. Here HP16 as digestion rotor was used, the volume of which reaches 100 mL and can withstand up to 4 MPa pressure during the reaction. It can perform high performance digestion in the sample preparation process. Thanks to the high temperature and pressure maintaining ability of M6 microwave digestion system during the experiment, it can improve the reaction efficiency and reduce operation error.