Application of Microwave Digestion in the Determination of Trace Elements in Graphite Anode Materials

1. Introduction

Lithium-ion battery technology offers outstanding advantages in the eco-economic and new energy's development. As the core component of lithium-ion batteries, anode material plays an important role in batteries performance behavior as cell energy density, working potential etc.. Nowadays, graphite is considering as the most state-of-the-art anode material for lithium batteries. It has the merits as low lithium insertion potential to ensure high output voltage; stable structure during charging and discharging process and has a long cycle life; high electronic conductivity; abundant in natural resources etc. While, the impurities content in graphite material can cause battery degradation and affect the stability and life cycle greatly. So that the determination of impurities is the vital for the Lithium battery producer for their QA/QC requirement. However, graphite material can withstand high temperature, highly corrosion resistant, stable in structure which makes sample preparation as a demanding challenge. Here, a microwave digestion method is presented for preparing graphite materials for further elemental analysis as ICP-OES or ICP -MS. With high-performance air-tight high pressure vessel coupled with M6 microwave digestion system, the graphite can be digested thoroughly.

2. Instrument and reagent

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

M6 microwave digestion system

HP 16 rotor

G-160 hot block

Reagent:

HNO_3(GR) ; H_2SO_4 (GR) ; $HClO_4$ (GR)

Sulfuric acid (H_2SO_4) was used due to its high boiling point. It can maintain the high temperature at a reduced vapor pressure.

Limited perchloric acid ($HClO_4$) was used (1mL) in the acid mixture to provide a higher oxidation potential which helps to degrade carbon above 200 ^{0}C.

3. Experiment method

1. Weigh 0.1 g sample into sample cup.
2. Add nitric acid, sulfuric acid and perchloric acid into the sample in the order mentioned above. Then mix the sample by swirling the cup gently.
3. Seal the vessel and set the microwave digestion program as follow:

Table 1: microwave digestion program

Step	Setting temperature(℃)	Ramp time (min)	Temperature holding (min)
1	140	10	5
2	180	8	5
3	220	8	20

4. Take the vessels out of the cavity when the temperature falls under 60 ℃. Open the vessels and place them on the hot block to evaporate acid.
5. Dilute the sample to 50 mL with deionized water when the temperature of the vessels cools to room temperature.

4. Result and discussion

The final solution is clear and transparent as shown in the figure below.

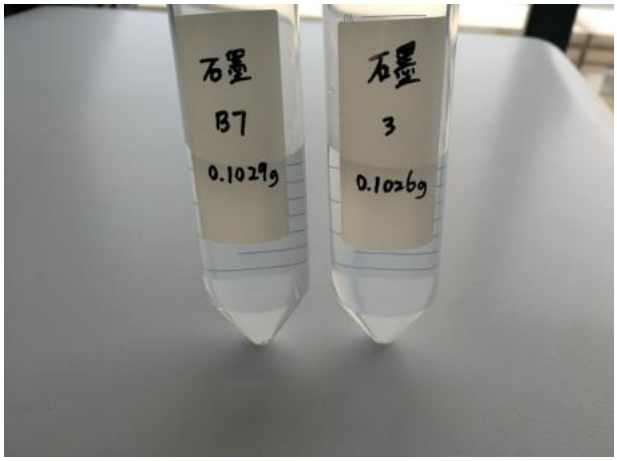

Fig.1 Graphite digestion solution

The accuracy of a measurement can be affected by matrix effect due to incomplete digestion. In order to verified the method, the sample solution was spiked during the post-digestion process. The concentration of copper, iron, nickel as spiked elements were shown in the following table.

Table 2: ICP-OES measurement spike recovery

Element	Spiked (mg/L)	Found value (mg/L)	Recovery (%)
Copper	5	5.03	101
Iron	10	10.49	105
Nickel	15	14.968	99.8

The result shown in table 2 demonstrates excellent recoveries for the spiked element, which suggests a minimized matrix effect due to the digestion procedure. At the other hand, the

high pressure rotor HP 16 can withstand the limited adding amount of perchloric acid, which generate high oxidation potential above 200 ^{0}C. The unique venting technology ensures the pressure control and safe digestion during the experiment.

5. conclusion

Preekem's M6 microwave digestion system coupled with HP 16 rotor can be applied in the digestion of graphite anode material. The digestion method presents here represent as simple start for different kinds of graphite material. Due to the different composition and structure of graphite, the digestion method and parameter as sample weight, reaction temperature, acid composition should be altered to adapt different situation. Thanks to the advanced full vessel real-time temperature monitor and pressure control technique, the digestion unit not only ensures the safe and precise sample digestion but also improves the accuracy and reproducibility during the experiment.