

The digestion of cathode material in lithium-ion battery by M6 microwave digestion system coupled with HP 16 rotor

1. Introduction

Lithium-ion batteries are composed of cathode material, anode material, membrane and a suitable electrolyte. Cathode material include the original LiCoO_2 , LiMnO_2 composite; mixed oxide $\text{LiMn}_x\text{Ni}_y\text{Co}_{1-x-y}\text{O}_2$ and the most used LiFePO_4 composite. Nowadays, LiFePO_4 material is widely used in rechargeable batteries due to its unique advantages as extensive source of raw material, low-cost in manufacture, and eco-friendly. With the driven of increasing market, multiple standards have been published for evaluating lithium amount in the cathode. As the material mentioned above, different cathode materials are differing in their chemical properties that require individualized acid composition and digestion method. However, microwave digestion can digest sample in an air-tight vessel which helps to increase the reaction efficiency and has the advantages of low blank value, good parallelism and convenient for method adapting.

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) ; HCl (GR)

3. Experiment method

1. Weigh 0.1 g sample into sample cup.
2. Add nitric acid, hydrochloric acid into the sample Then mix the sample by swirling the cup gently.
3. Seal the vessel and set the microwave digestion method as follow:

Table 1: microwave digestion program

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

4. Take the vessels out of the cavity when the temperature falls under 60 °C. 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 digestion solution for LiFeO_4 is clear and transparent as shown in the figure below. The solution appears yellowish color due to its iron content.



Fig.1 LiFeO_4 digestion solution

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

Table 2: ICP-OES measurement spike recovery

Element	Spiked (mg/L)	Found value (mg/L)	Recovery (%)
Cu	20	19.7	98.5
Zn	60	58.3	97.2
Fe	20	20.4	102
Cr	20	18.8	94
Ni	20	21.4	107
Mo	20	19.8	99
Li	60	60.7	101

The result shown in table 2 demonstrates excellent recoveries for the spiked elements, which suggests a minimized matrix effect due to the digestion procedure. 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 cathode material as LiFeO_4 . Due to the different composition and structure 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.