

Supporting Information for

MgAl-LDH nanoflowers as a novel sensing material for high-performance humidity sensing

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Keywords: Humidity sensor; Quartz crystal microbalance; MgAl-LDH; nanoflowers

1. The Chemical Raw Materials

Aluminium nitrate ($\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$), magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), nickel nitrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), potassium bichromate ($\text{K}_2\text{Cr}_2\text{O}_7$), nitric acid (HNO_3), ammonium hydroxide ($\text{NH}_3 \cdot \text{H}_2\text{O}$), EDTA, dimethylglyoxime, triammonium citrate, methyl orange, urea, ethanol and iodine solution were purchased from Sinopharm Group Co. LTD.

2. Synthesis of MgAl-LDH nanoflowers

MgAl-LDHs nanoflowers and microsheets were synthesized using NO_3^- based divalent and trivalent salts as precursor materials. For MgAl-LDHs nanoflowers, 0.015 mol $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 0.005 mol $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were dissolved in boiling deionized water to remove CO_2 . Then, ammonia was added dropwise to the solution at a temperature of 338 K under N_2 atmosphere. The mixed solution was controlled at a pH value of 10. After vigorous stirring for 30 min, both types of suspensions were transferred into 100 mL Teflon-lined autoclaves and maintained at 120 °C for 24 h. The precipitate was then collected by filtration, washed with deionized water and ethanol several times, and finally dried at 80 °C for 12 h.

2. Characterization

The morphology of MgAl-LDH nanoflowers was observed by scanning electron microscope (SEM, Germany ZEISS Sigma 300) and transmission electron microscopy (TEM, Japan JEOL JEM F200). Wide angle X-ray diffraction patterns of the materials were obtained on a Germany Bruker D2 Phaser diffractometer using Cu KR radiation.

Nitrogen adsorption-desorption isotherms were measured at 77 K by using a surface area and porosity analyzer (Micromeritics ASAP 2020). The materials were degassed before the measurements. The BET method was used to extract the specific surface areas. The amounts of various elements in MgAl-LDH nanoflowers were determined by using inductively coupled plasma atomic emission spectroscopy (ICP-AES) Varian 710-ES. FT-IR spectra of the sample was measured on an FTIR-8400s (Shimadzu) spectrometer in the transmission mode. Standard KBr technique was applied. Contact angle measurements of MgAl-LDH nanoflowers modified QCM was measured by Data physics OCA20. The static contact angle was measured in sessile drop mode. The volume of water droplet was set to 5 μL .

3. Fabrication and Test Methods of the QCM humidity Sensor

Prior to measurements, QCM sensors ultrasonic cleaning was successively carried out three times in acetone, ethanol and deionized water for 10 min, respectively. Then the sensors were dried at 40 °C for 30 min. After obtaining clean crystals chips, the corresponding frequency was recorded as the fundamental frequency. To ensure sensor consistency, we employed the widely used drop-coating technique¹⁻³ in the QCM sensor manufacturing field to deposit the as-prepared 20 mg/mL MgAl-LDH nanoflowers dispersion onto the silver surface of the QCM chips by a micropipette. In order to evaluate the effect of the depositing amount of MgAl-LDH nanoflowers on sensor performance, the sensors with 1, 2, and 3 μL of MgAl-LDH nanoflowers deposited on the electrode of QCMs were fabricated. The corresponding sensors were labeled as QCM-1, QCM-2 and QCM-3. After drying at 40 °C for 40 min, MgAl-LDH

nanoflowers modified QCM humidity sensors were successfully made. Then, the frequencies of all sensors were recorded. The details were exhibited as in Table 1. The humidity-sensing experiments were performed at room temperature (298 K). In order to explore the capability of humidity detection, a man-made measure setup is sketched in Fig. S1. The sensor was exposed to relative humidity (RH) level of 11%, 33%, 52%, 75%, and 97% produced by the saturated solution of LiCl, MgCl₂, Mg(NO₃)₂, NaCl, and K₂SO₄, respectively. The superabsorbent P₂O₅ powder was used as a desiccant in a vessel to provide a relative dry atmosphere (ca. 0% RH) as the baseline environment⁴. The real-time resonant frequency of QCM transducer was recorded by a frequency counter. In order to know the deposition amount of MgAl-LDH nanoflowers on the electrode of QCMs, the resonance frequencies and crystal's resistance (R_{crystal}) values before and after films deposition were measured, which was shown in Table 1. The deposition mass of MgAl-LDH nanoflowers on the electrode of QCMs were calculated according to the Sauerbrey equation.

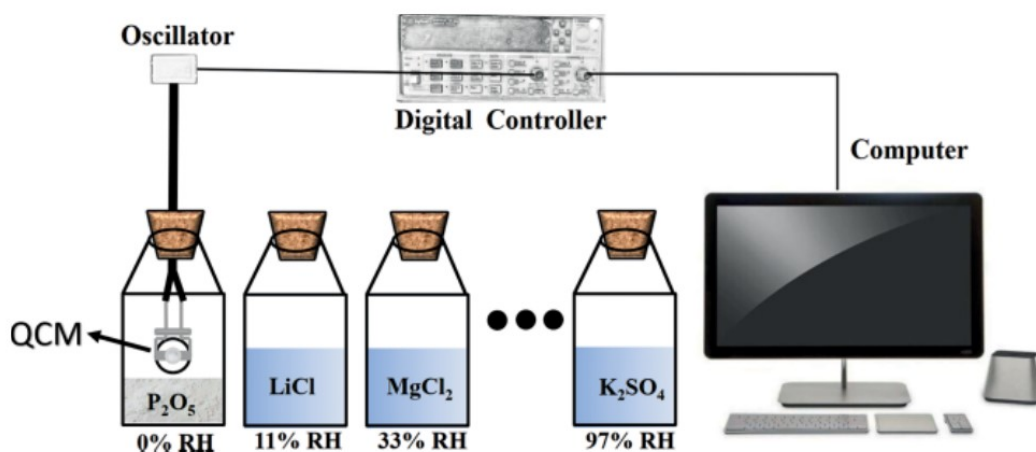


Fig. S1. The sketch diagram of humidity detection system.

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