Supporting Information

New Nickel(II) Cluster-Based Mixed Cation Coordination Polymer Constructed from 2-Mercaptobenzoic Acid and Its Application

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Experimental

Materials

The following chemicals were used directly as received from the suppliers without further purification: 2-mercaptobenzoic acid(mba) (Alfa Aisha; 98 at.%), sodium hydroxide (NaOH; Zhengjiang Zhongxing; 99 at.%), anhydrous ethanol (CH_3CH_2OH; Anhui Ante; 96 at.%), and nickel chloride hexahydrate ($NiCl_2.6H_2O$; Aladdin Shanghai; 99 at.%).

Sample preparation

We added 0.12 g (0.003 mol) of sodium hydroxide to 10 ml of anhydrous ethanol and placed the solution in an ultrasonic bath for 30 min until all sodium hydroxide was dissolved in the anhydrous ethanol. Next, mba (0.003 mol, 0.4626 g) was added to the solution and stirred on a constant-temperature magnetic stirrer for 30 min at 27 °C until complete dissolution. Thereafter, $NiCl_2.6H_2O$ (0.0015 mol, 0.3565 g) was completely dissolved in 5 ml of deionized water, and the solution was slowly added drop-wise into the ethanol solution and stirred for an additional 30 min. The homogeneous solution was then poured into a 30 ml Teflon-lined stainless-steel reactor, sealed, and allowed to react at 120 °C for 96 h in an electric blast oven. The reactor was allowed to cool to 27 °C, and the resulting product was washed several times with deionized water and anhydrous ethanol in a sequential manner before being dried in an electric blast oven at 90 °C to obtain a black fibrous product (yield ~0.37 g, 45.4 wt.% based on $NiCl_2.6H_2O$ and mba).

Structure characterization

Powder X-ray diffraction (PXRD) measurements were performed using a Bruker D8 ADVANCE diffractometer at 40 kV and 40 mA with Cu *Kα*(λ= 1.5406Å) radiation, with a step size of 0.01° and a scan speed of 0.1 $s.step^{-1}$. Thermogravimetric Analysis (TGA) was performed under N_2 at a heating rate of 5 $°C.min^{-1}$ using a TA-Q600 system (TA Instruments, USA). The thermal stability of **1** was investigated from room temperature to 800 °C. Fourier Transform Infrared Radiation (FT-IR) spectra were recorded on an EQUINOX55 spectrophotometer (Bruker) in the range 4000–400 cm^{-1} with the KBr disk technique. X-ray photoelectron spectroscopy (XPS) was performed on a spectrometer from Kratos Axis Ultra DLD, using Mono Al *Kα* radiation at a power of 120 W (8 mA, 15 kV). The nitrogen

adsorption/desorption data were recorded at the liquid nitrogen temperature (77 K) using micromeritics apparatus (ASAP 2020 M). The specific surface area was calculated using the Brunauer–Emmett–Teller (BET) equation. Ultraviolet−Visible (UV) Diffuse Reflectance Spectroscopy (DRS) was measured with a Shimadzu 3600 UV−vis−Near Infrared Radiation (NIR) spectrophotometer with $BaSO_4$ as an internal standard. Elemental analysis(EA) (H) was carried out on a Carlo-Erba 1112 Elemental Analyzer.

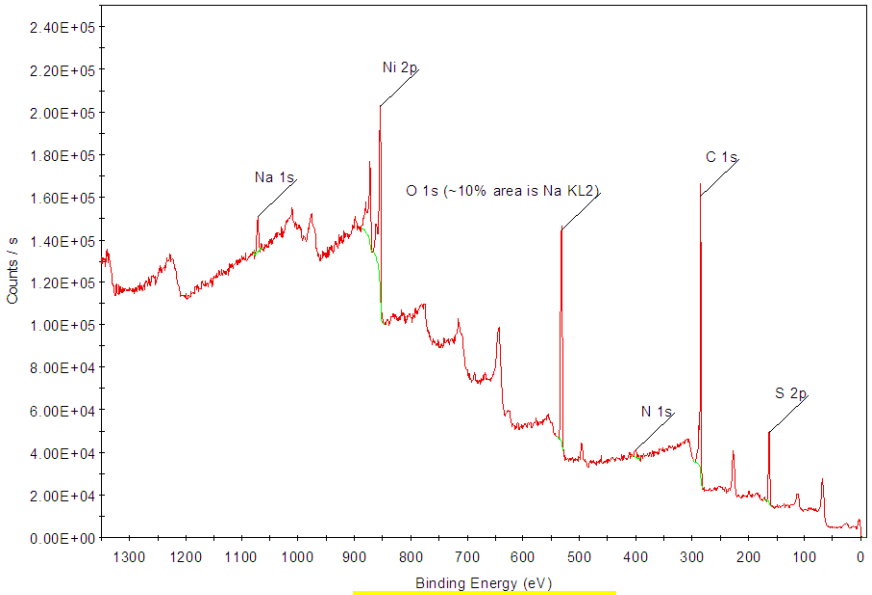

Figure S1. The XPS spectrum of **1**.

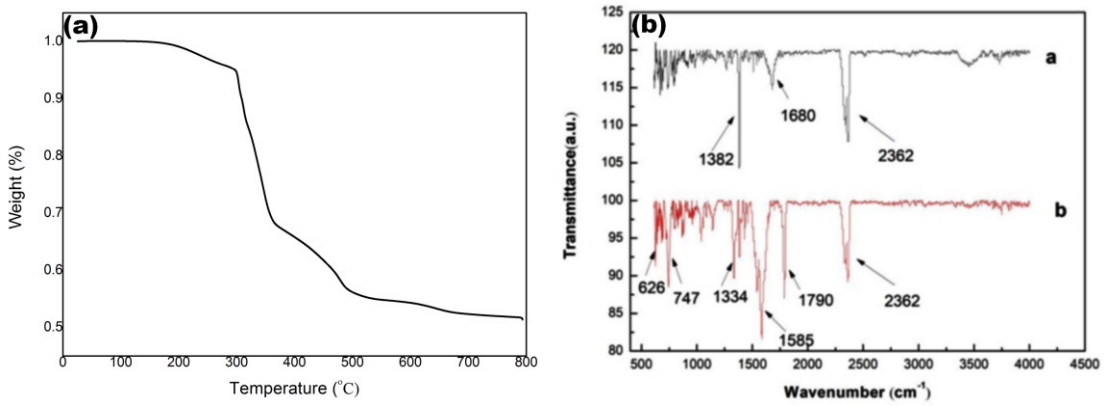

Figure S2. (a)TGA plot showing the relative weight loss of **1** as a function of temperature. (b) FT-IR spectra of mba (top) and the complex sample (bottom).

送检单位	温州大学				
送 检 人	祝精燕	送检日期	2019 年 5 月 7 日	联系电话	
样品名称	Ni 已洗			登记编号	1889
检测项目	H				

检测结果

样品质量		2.332 毫克	2.661 毫克	毫克
百分含量	H	3.23 %	3.17 %	%
		%	%	%
		%	%	%

附 注：

方法简述：

检测依据：☑ JY/T 017-1996 元素分析仪方法通则
☐ 其他：

仪器型号：☑ Elementar 公司 Vario EL III 型全自动元素分析仪
☐ Elementar 公司 Vario Micro Cube 型全自动元素分析仪
☐ Thermo Flash 2000 型元素分析仪

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检 测 员：

核 验 员：

中国科学院上海有机化学研究所
公共技术服务中心
2019 年 5 月 8 日

通讯地址：上海市零陵路 345 号， 联系人：戴钰婷，电话：021-54925315，E-mail: daiyt@sioc.ac.cn
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Anal. calcd for $C_{42}H_{27}Cl_2NaNi_8O_{15}S_6$ (%): H, 1.78. Found: H, 3.17.

Figure S3. EA result(H) of **1**.

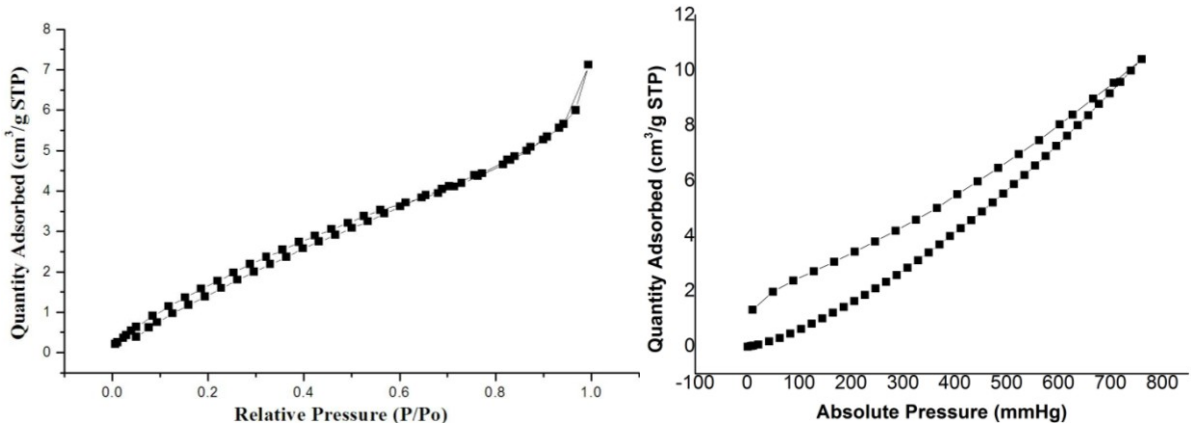

Figure S4. N_2(left) and CO_2(right) adsorption/desorption isotherms curve of **1**.

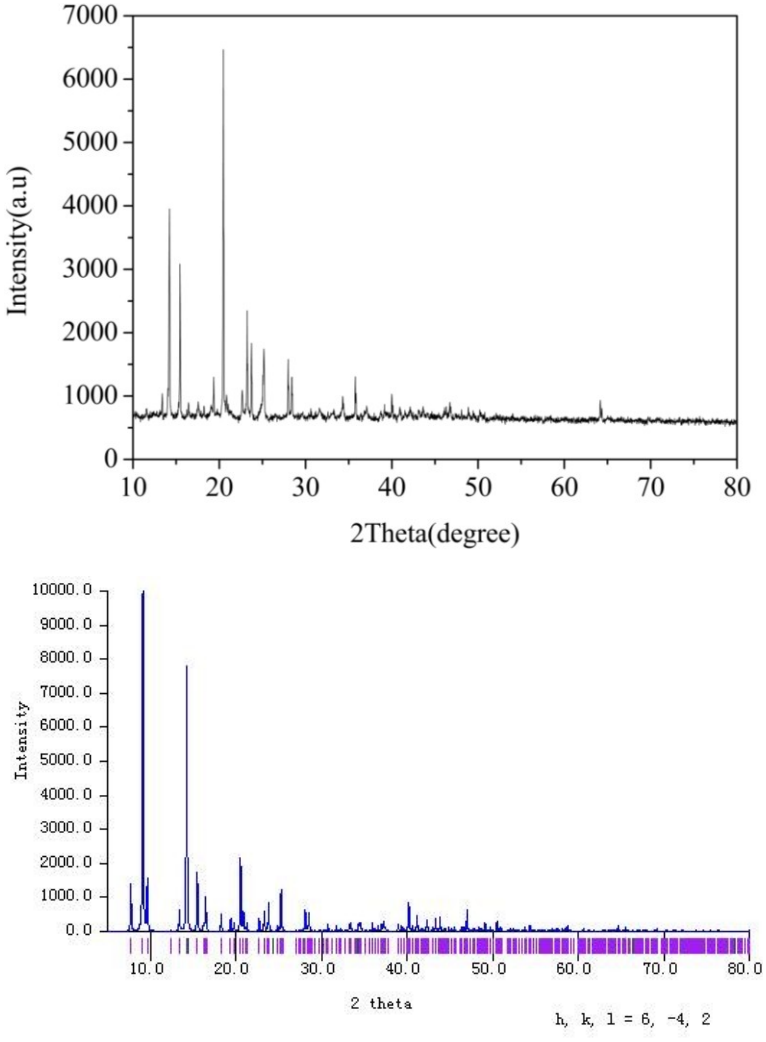

Figure S5. PXRD pattern(top) and simulated PXRD pattern(bottom) of **1**.

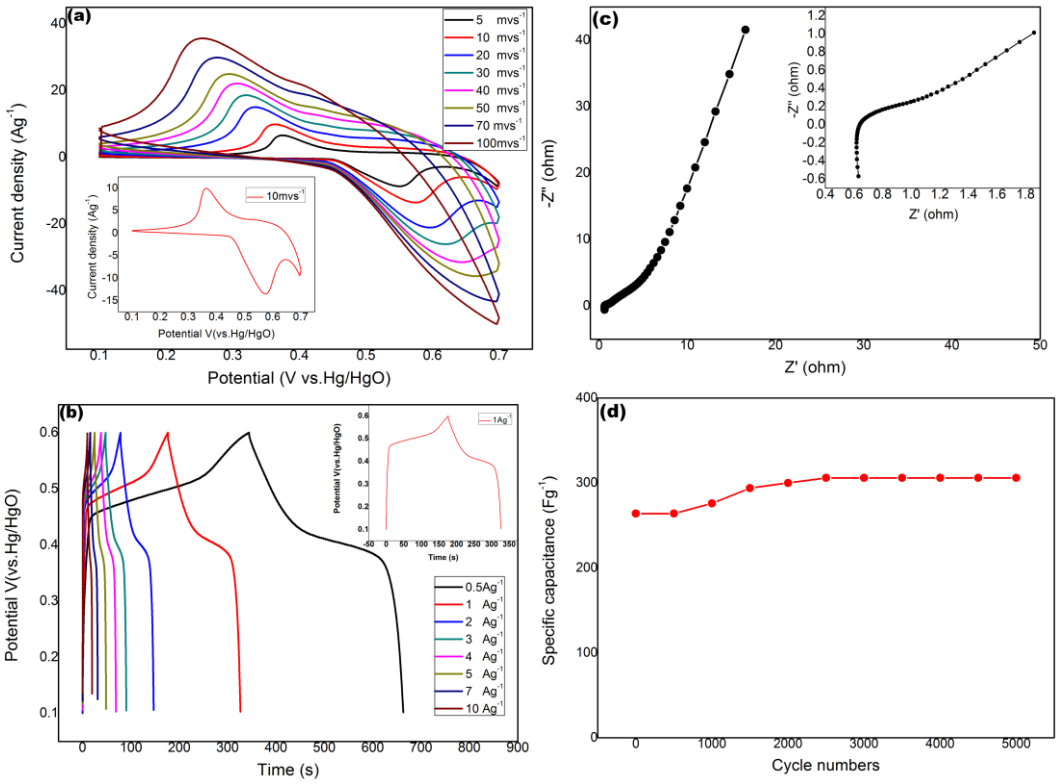

Figure S6. Electrochemical properties of NiO/C&S composite. (a) Cuclic voltammogram (CV) curves (current vs. potential) at different scan rates; the inset shows only the CV curve at 10 mVs^{-1} for clarity. (b) Galvanostatic charge/discharge curves of at different current densities; the inset shows only the galvanostatic charge-discharge curve at 1 Ag^{-1} for clarity. (c) Electrochemical impedance spectrum (the inset shows the impedance spectrum for a limited range between 0.5 and 1.8 Ω on the x-axis). (d) Cycle-life performance during 5,000 cycles at a specific current of 3 Ag^{-1}.

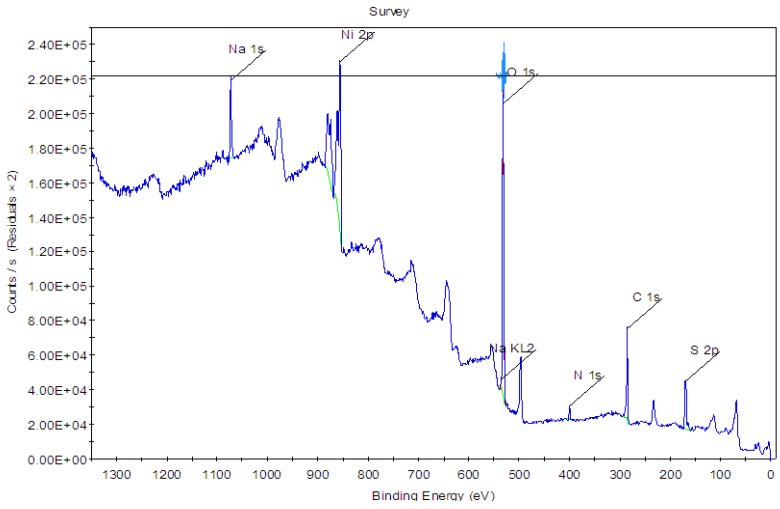

Figure S7. The XPS spectrum of NiO/C&S composite.

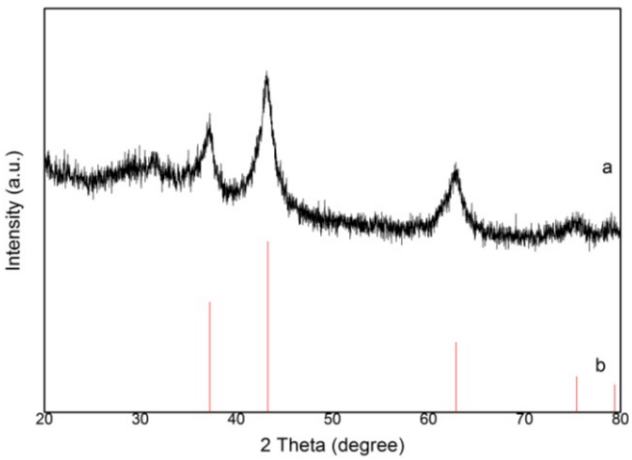

Figure S8. PXRD patterns of (a) NiO/C&S composite, (b) NiO(JCPDS No. 04-0835).

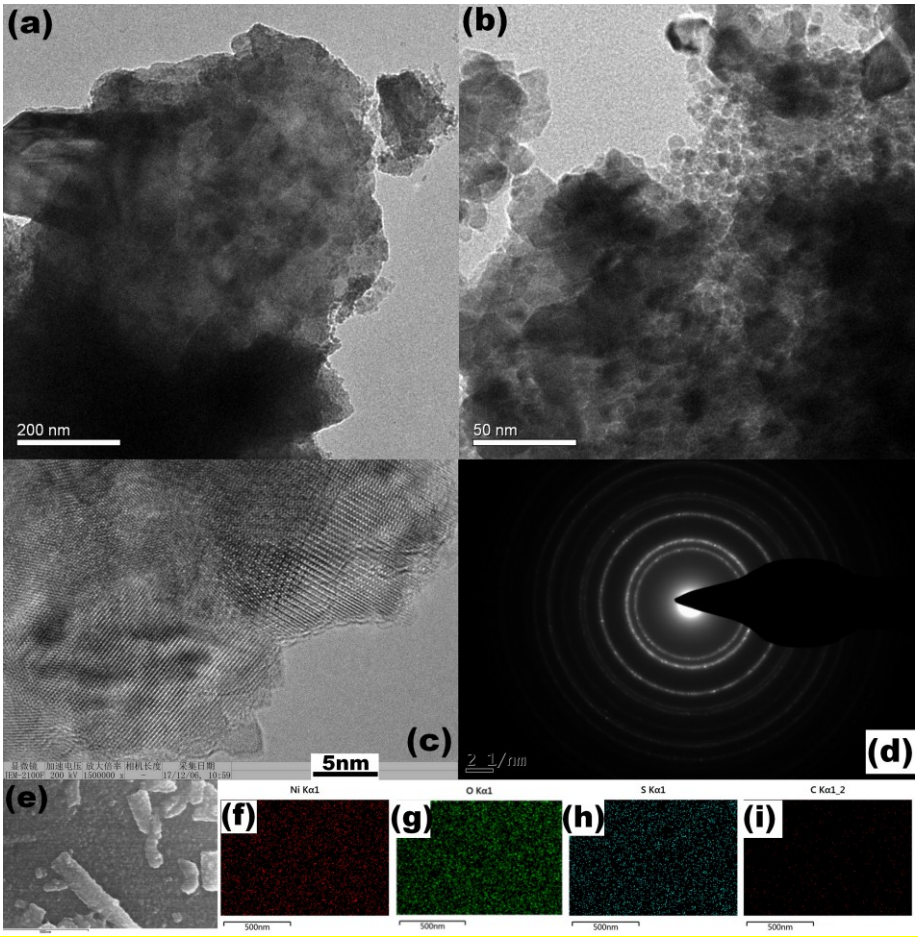

Figure S9. (a, b)TEM images at different magnification, (c) high-magnification TEM image, (d) electron diffraction pattern of NiO/C&S composite. The rings in the SAED pattern are consistent with (111), (200), (220), (311), and (222) reflections of cubic NiO, (e) The image of the NiO/C&S composite sample was used for elemental mapping that qualitatively indicates the presence of Ni, O, S, and C elements in the sample, (f-i) The mapping images in different color clearly show that various elements are uniformly distributed in the particle sample.