## **XPS analysis**

## **Experimental section**

To obtain dried nanoparticle, 50 mL sol was diluted with ethanol (99.9%) at ratio (v) 1:1. The diluted sample was centrifuged at 10000 rpm for 30 min and the precipitated solid was collected and dried in vacuum dryer at 80 °C for 6 h. XPS data were acquired using a PerkinElmer PHI 5400 X-ray photoelectron spectrometer. This system was equipped with Mg X-ray source operated at 300 W (15 kV, 20 mA). The carbon (C 1s) line at 285.0 eV was used as the reference line.

## **Results and discussion section**

A typical XPS spectrum of Copper nanoparticle was measured and the spectrum of Cu  $2p_{3/2}$  is depicted in Fig. S1a. The Cu  $2p_{3/2}$  spectrum shows that the Cu<sup>2+</sup> peak lies at 932.98 eV<sup>1</sup>, with two shake-up satellites at higher binding energy (BE 942.35 and 940.85 eV)<sup>2, 3</sup>. The Cu<sup>1+</sup> peak was appeared at 931.55 eV which attributed to Cu<sub>2</sub>O<sup>1, 4</sup>. Apart from that, Fig. S1b shows O 1s core level spectrum is broad and fitted by 2 different peaks. The peak located at lower energy (BE 529.50 eV) is attributed to O<sup>2-</sup> in CuO<sup>3, 5, 6</sup> while the other peak at higher energy (BE 530.30 eV) is ascribed to O<sup>1-</sup> in Cu<sub>2</sub>O<sup>5, 6</sup>. The binding energy, FWHM and area of Cu  $2p_{3/2}$  are summarized in Table S1. The XPS results indicate that the sample is composed of CuO and Cu<sub>2</sub>O. The XPS result was not in agreement with XANES result, where 57.6% of CuO and 42.4% of Cu<sub>2</sub>O was found in the solids separated from sol; while, 47% Cu<sup>0</sup>, 23% of CuO and 30% CuCl<sub>2</sub> was acquired in the sol via XANES fitting. The concentration of oxide species increased, it might be due to oxidation happened during the XPS sample preparation.

Binding energy	Cu phase	FWHM	Area	Percentage (%)
(eV)				
931.55	Cu <sub>2</sub> O	2.76	1852.811	42.43
932.98	CuO	3.39	860.6919	19.71
940.85	CuO	3.90	1074.291	24.60
942.35	CuO	2.36	579.304	13.26

Table S1 The results of deconvolution of XPS Cu  $2p_{3/2}$  peaks





Fig. S1 Typical deconvolution of Cu  $2p_{3/2}(a)$  and O 1s (b) main peak of 6 h sample.

## References

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