

Supplementary information

# Characterisation of Iron Core–Gold Shell Nanoparticles for Anti-Cancer Treatments: Chemical and Structural Transformations During Storage and Use

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## Materials and Methods:

## Characterization of freshly synthesized Fe@Au

Fe@Au nanoparticles were diluted into ethanol and quickly applied onto copper grids coated with a thin carbon support film. The specimens were immediately observed at an accelerating voltage of 300 kV using a 3000F TEM (JEOL, Tokyo, Japan), equipped with a SC1000 11-megapixel CCD camera (Gatan, CA, USA). X-ray diffraction (XRD) analysis of the nanoparticles was done using Cu/K  $\alpha$ -radiation, generated at 40 kV and 30 mA (average wavelength is 0.15418 nm), in a Bruker X-ray diffractometer (RX-III; Shimadzu Corp., Kyoto, Japan). XRD patterns ( $20^\circ < 2\theta < 90^\circ$ ) were collected at a scan rate of 5° per minute.

## Fe/O atomic ratio of reconstituted and aged Fe@Au

Fe@Au nanoparticles were diluted into ethanol and quickly applied onto copper grids coated with a thin carbon support film. The specimens were immediately observed at an accelerating voltage of 200 kV using a 2100F TEM (JEOL, Tokyo, Japan), equipped with an EDXS system. The mean Fe/O ratio of reconstituted and aged Fe@Au is  $0.447 \pm 0.07$  and  $0.314 \pm 0.09$ , respectively, which is significantly different ( $p = 0.0001$ , Student's T test).

## Results:

Table S1. The Fe/O atomic ratio data of reconstituted and aged Fe@Au obtained through EDXS. Each line of the table is the data from a single-field EDXS analysis of the two samples, with the mean  $\pm$  standard error given in the final row in bold.

Reconstituted Fe@Au, atomic %			Aged Fe@Au, atomic %		
O	Fe	Au	O	Fe	Au
64.5	23.5	11.1	63	25.5	11.5
41.1	20.8	38.1	46.4	20.7	33
61.5	26.1	12.4	62.7	20.9	16.4
51.4	26.4	22.2	61.7	21.6	16.7
61.4	23.3	15.2	63.1	17.5	19.4
55.1	22.2	22.8	68.2	20.6	11.2
53.4	30.1	16.4	60.6	23.7	15.8
57.9	25.1	19	48.2	19	32.8
50	24.4	25.6	61	25.6	13.4
40.3	18.7	40.9	60.7	5.5	33.7
65.4	23.5	11.1	65.6	19.6	14.8
45.6	23.8	30.6	69.9	17.8	12.3
63.3	24.4	12.4	66.8	14.7	18.5
			70.6	17.5	11.9
			57.4	23.6	19
			61.7	19.8	18.5
			62	23.6	14.4
			60.8	17.4	21.8
			60.9	18.8	20.3
			75.7	10.3	14
<b>54.7 <math>\pm</math> 2.4</b>	<b>24.0 <math>\pm</math> 0.8</b>	<b>21.4 <math>\pm</math> 2.8</b>	<b>61.4 <math>\pm</math> 1.9</b>	<b>19.4 <math>\pm</math> 1.4</b>	<b>19.2 <math>\pm</math> 2.3</b>

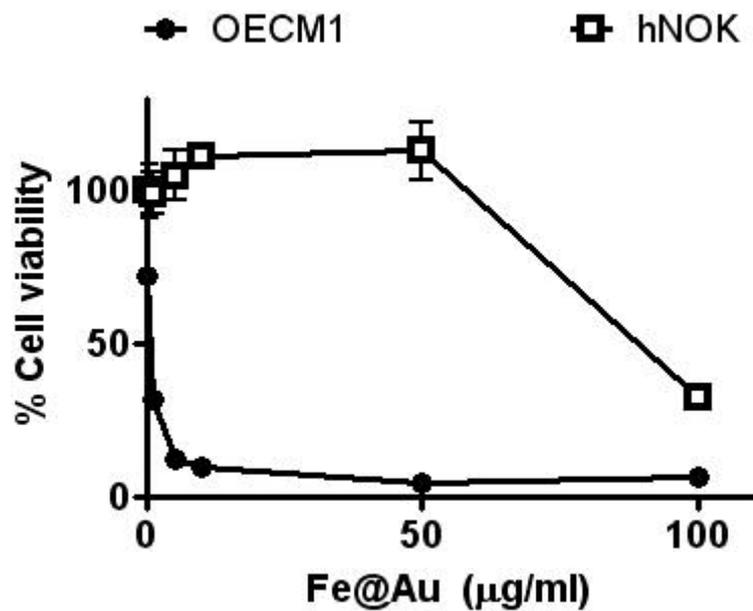


Figure S1. Quality control data on the anti-cancer selective effects of Fe@Au. Prior to further experiments, the cancer specific anti-cancer effect of Fe@Au was estimated by WST-1 assay with 48 hours' treatment. The  $IC_{50}$  of the freshly synthesized Fe@Au in OECM1 is between 1 and 5 µg/mL, while a significant decrease in cellular viability was only observed at doses exceeding 50 µg/mL in hNOK cells ( $p = 0.0053$ , Student t-test; compared to Fe@Au-treated OECM1).

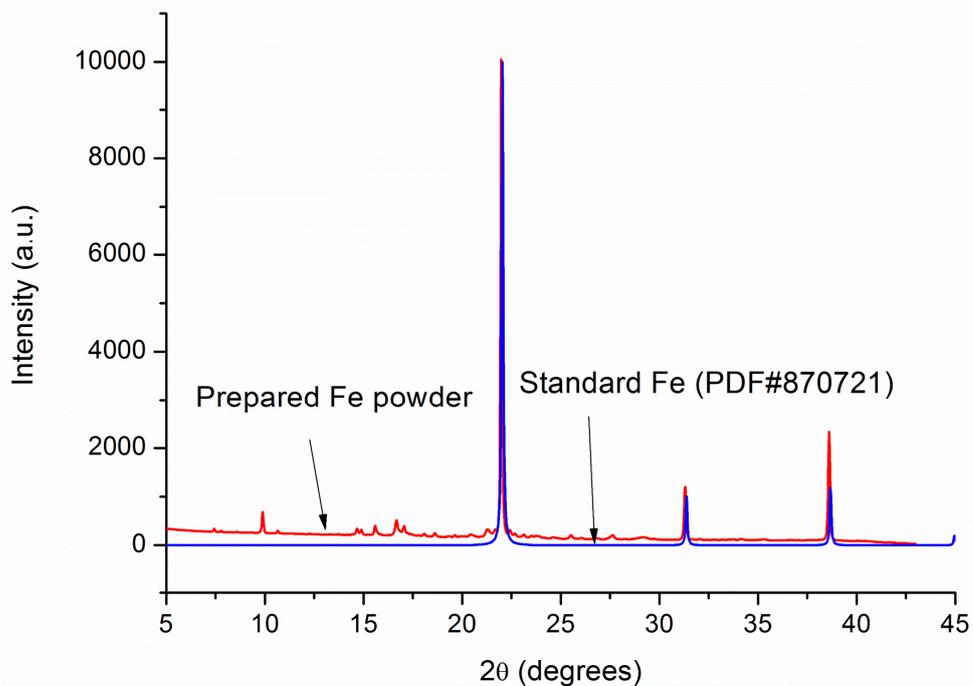


Figure S2. Investigation of synthesized Fe-core-alone particles by synchrotron radiation based XRD. To avoid the problems of overlapping peaks from Fe and Au under XRD of Fe@Au, we synthesized pure Fe core nanoparticles and analyzed them by XRD. This shows that the major peaks are identical with the standard powder diffraction file of elemental Fe.