

## **ELECTRONIC SUPPLEMENTARY INFORMATION**

### **(ESI)**

#### **Calcination-controlled performance optimization of iron-vanadium bimetallic oxides nanoparticles for synergistic tumor therapy**

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## 1. Supplementary Experimental Section.

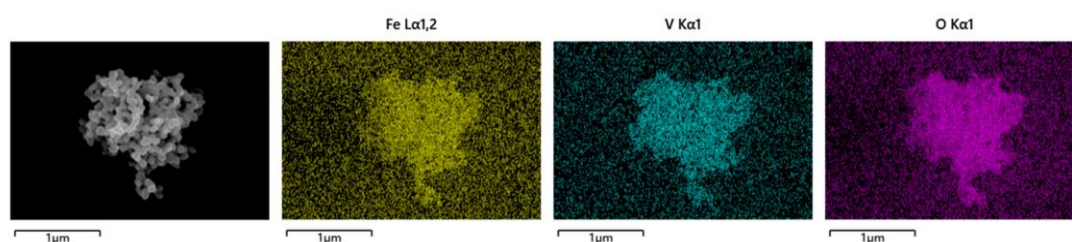
### Chemicals and abbreviations

Iron nitrate nonahydrate ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ), ammonia water, ethanol, and  $\text{H}_2\text{O}_2$  (30%) were purchased from XILONG SCIENTIFIC. Ammonium metavanadate ( $\text{NH}_4\text{VO}_3$ ), 3,3',5,5'-tetramethylbenzidine (TMB) was purchased from Shanghai Aladdin Reagent Co. 5,5-Dimethyl-1-pyrroline N-oxide (DMPO) was purchased from Energy Chemical. Cell Counting Kit-8 (CCK-8), Calcein/PI Live/Dead Viability/Cytotoxicity Assay Kit (Calcein-AM and PI), Reactive Oxygen Species Assay Kit (DCFH-DA), and mitochondrial membrane potential assay kit (5,5',6,6'-tetrachloro-1,1',3,3'-tetraethyl-imidacarbocyanine iodide, JC-1) were purchased from Beyotime Biotechnology Co. Shanghai, China. RPMI 1640 (with 10% serum and 1% penicillin-streptomycin mixture) was from Meilunbio. All chemicals were used without further purification.

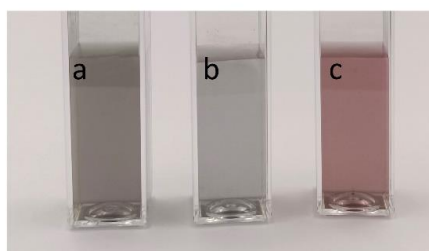
### Characterization

Their crystal phase was determined by X-ray diffraction (XRD) (Bruker, D8 ADVANCE) equipped with Cu-K $\alpha$  radiation ( $\lambda = 0.154 \text{ nm}$ ). Transmission electron microscope (TEM; Hitachi H 9000 NAR) was used to observe the morphologies. Zeta potential was tested on Zetasizer Nano ZS (Malvern Instruments Ltd., UK). X-ray photoelectron spectroscopy was carried out on a Thermo SCIENTIFIC ESCALAB Xi+ X-ray photoelectron spectrometer. UV-vis-NIR spectra were detected on a UV-vis-NIR spectrophotometer (VARIAN CARY 50). Fluorescence imaging was observed by a Confocal Laser Scanning Microscope (CLSM, Leica TCS SP2, Leica Microsystems, Mannheim, Germany).

## 2. Supplementary Figures and Tables.



**Fig. S1** Energy dispersive spectrometer (EDS) analysis of FVO-Ar NPs.



**Fig. S2** FVO-Ar NPs had good dispersibility in deionized water (a), PBS (b), and RPMI 1640 (c) medium.

$$\eta = \frac{hS(T_{max} - T_{surr}) - Q_{dis}}{I(1 - 10^{-A_{808}})} \quad (1)$$

$$Q_{dis} = \frac{\sum mC_p \Delta T}{\Delta t} \quad (2)$$

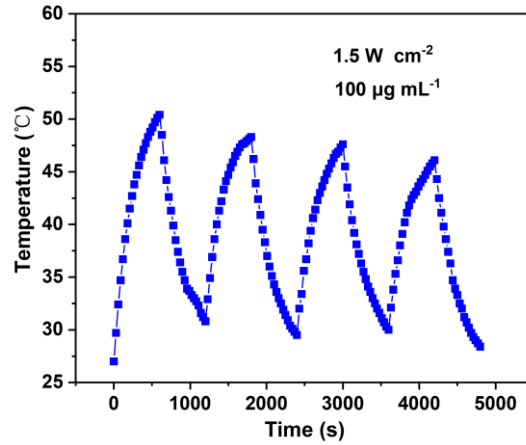
$$hS = \frac{\sum mC_p}{\tau_s} \quad (3)$$

$$\tau_s = \frac{t}{-\ln \theta} \quad (4)$$

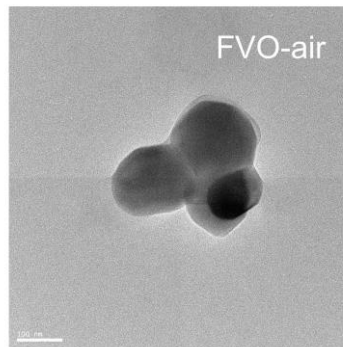
$$\theta = \frac{T - T_{surr}}{T_{max} - T_{surr}} \quad (5)$$

**Fig. S3** Calculation formula of photothermal conversion efficiency.

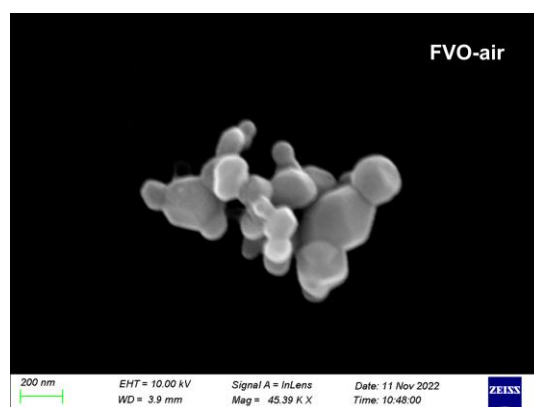
The  $\eta$  is the photothermal conversion efficiency,  $S$  is the surface area of the container, and  $T_{max}$  is the maximum steady temperature for FVO-Ar NPs solution.  $I$  is the incident laser power and  $A$  is the absorbance of NPs solution at 808 nm ( $A_{FVO-Ar} = 1.61$ ,  $A_{FVO-air} = 1.23$ ).  $m$  is the mass (1.0 g) and  $C_p$  is the heat capacity [ $4.2 \times 10^3 \text{ J (kg} \cdot ^\circ\text{C)}^{-1}$ ] of water.  $\theta$  is the dimensionless driving force temperature,  $T_{surr}$  is the surrounding temperature, and  $\tau_s$  is the sample system time constant.



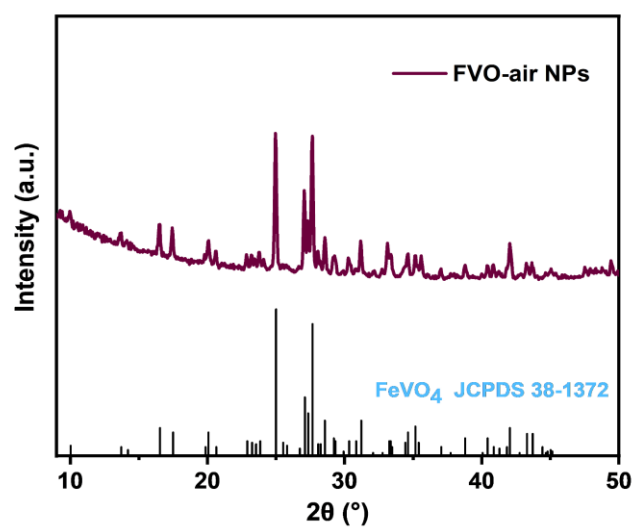
**Fig. S4** Photothermal stability of FVO-Ar NPs aqueous suspension ( $100 \mu\text{g mL}^{-1}$ ,  $1.5 \text{ W cm}^{-2}$ ) for four laser on/off cycles.



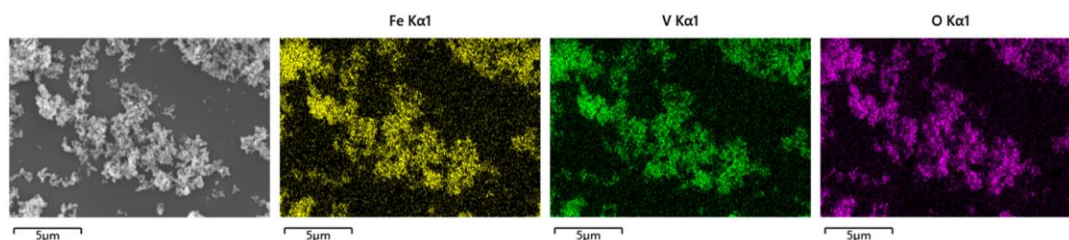
**Fig. S5** TEM image of FVO-air NPs. Scale bar: 100 nm.



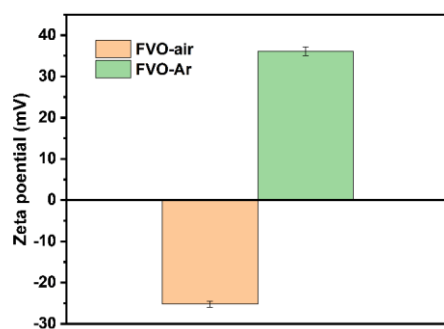
**Fig. S6** SEM image of FVO-air NPs. Scale bar: 200 nm.



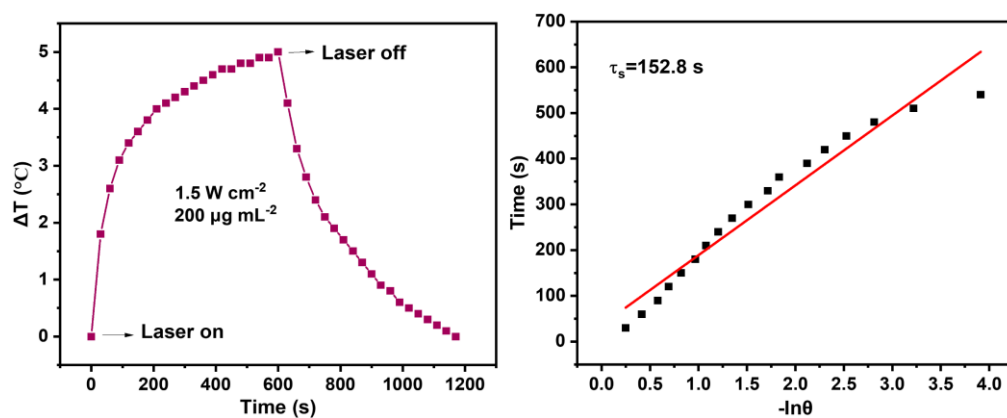
**Fig. S7** XRD patterns of FVO-air NPs.



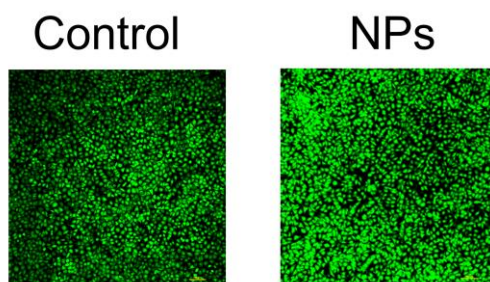
**Fig. S8** Energy dispersive spectrometer (EDS) analysis of FVO-air NPs.



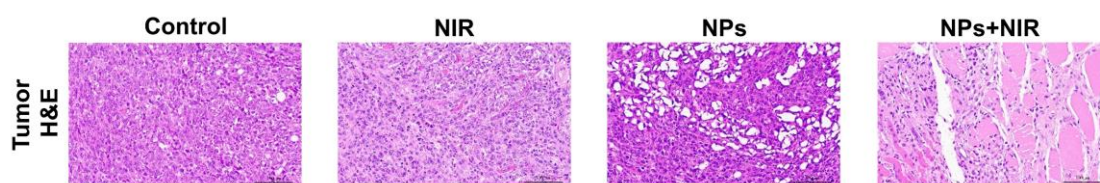
**Fig. S9** Zeta potential of FVO-air NPs and FVO-Ar NPs. (n=3)



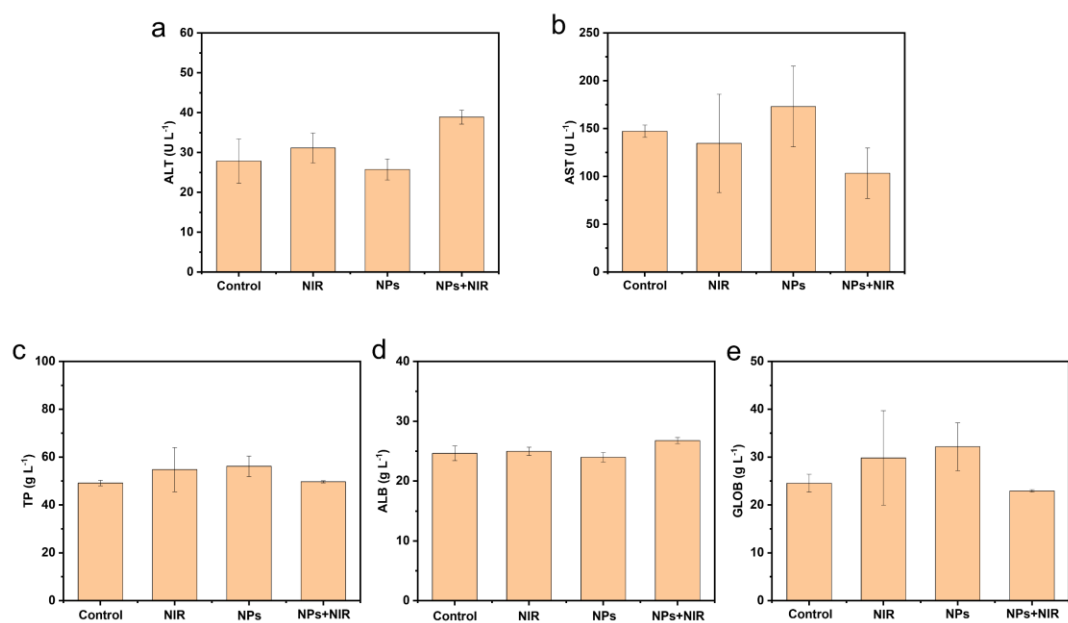
**Fig. S10** Photothermal conversion test of FVO-air NPs. ( $1.5 \text{ W cm}^{-2}$ )



**Fig. S11** With or without the addition of FVO-Ar NPs, L929 cells both showed green fluorescence. Scale bar:  $100 \mu\text{m}$ .



**Fig. S12** H&E stained tumor slices on the 16th day of various treatments (Scale bar:  $100 \mu\text{m}$ ).



**Fig. S13** The liver function indexes of mice after various treatments. (a) Alanine aminotransferase (ALT), (b) Aspartate aminotransferase (AST), (c) Total protein (TP), (d) Albumin (ALB), (e) Globulin (GLOB).