

**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 H_2O_2 (30%) were purchased from XILONG SCIENTIFIC. Ammonium metavanadate (NH_4VO_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 $\text{Cu-K}\alpha$ radiation ($\lambda = 0.154$ 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.

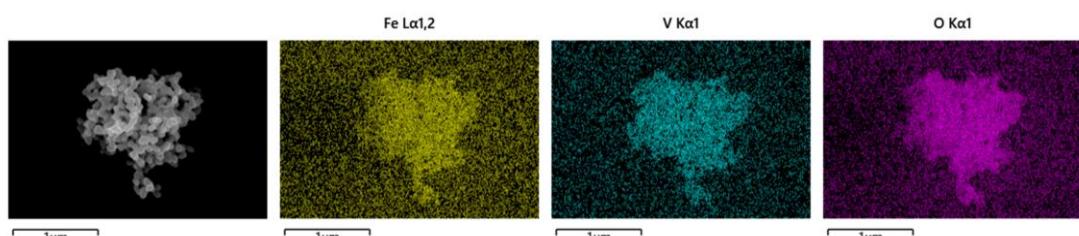


Fig. S1 Energy dispersive spectrometer (EDS) analysis of FVO-Ar NPs.

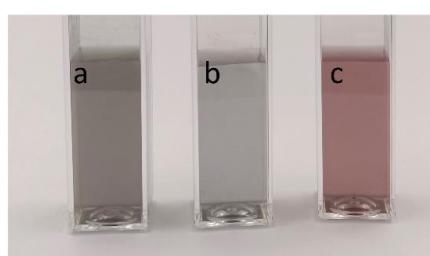


Fig. S2 FVO-Ar NPs had good dispersibility in deionized water (a), PBS (b), and RPMI 1640 (c) medium.

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

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

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

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

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

Fig. S3 Calculation formula of photothermal conversion efficiency.

The η 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 [$4.2 \times 10^3 \text{ J (kg} \cdot \text{}^{\circ}\text{C})^{-1}$] of water. θ is the dimensionless driving force temperature, T_{surr} is the surrounding temperature, and τ_s is the sample system time constant.

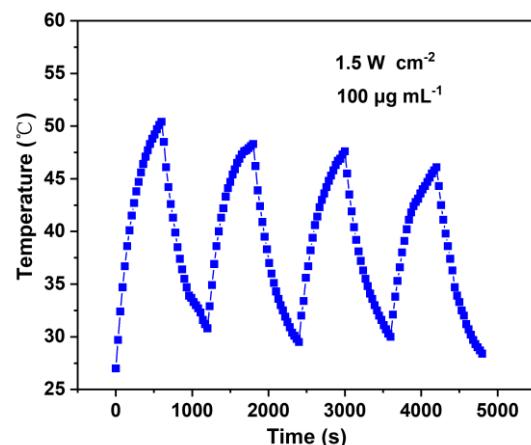


Fig. S4 Photothermal stability of FVO-Ar NPs aqueous suspension ($100 \mu\text{g mL}^{-1}$, 1.5 W cm^{-2}) for four laser on/off cycles.

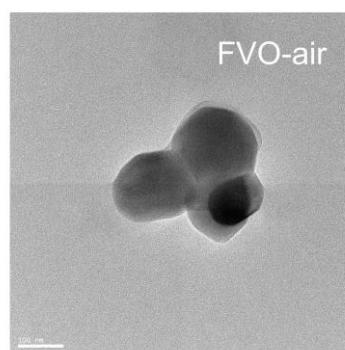


Fig. S5 TEM image of FVO-air NPs. Scale bar: 100 nm.

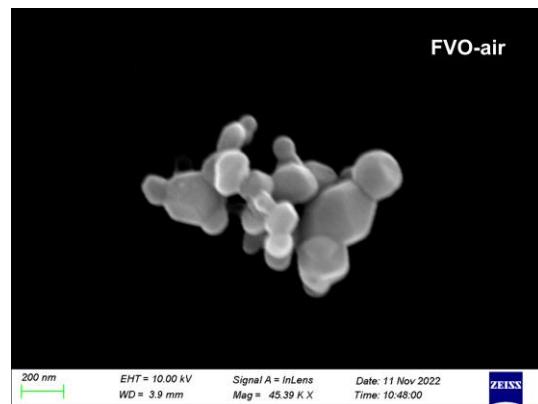


Fig. S6 SEM image of FVO-air NPs. Scale bar: 200 nm.

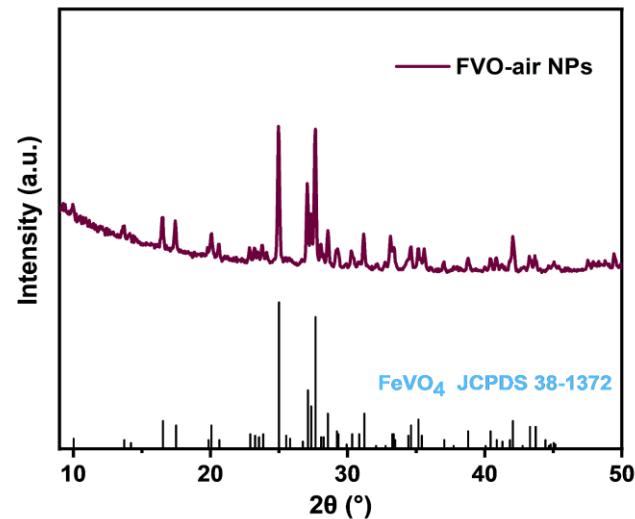


Fig. S7 XRD patterns of FVO-air NPs.

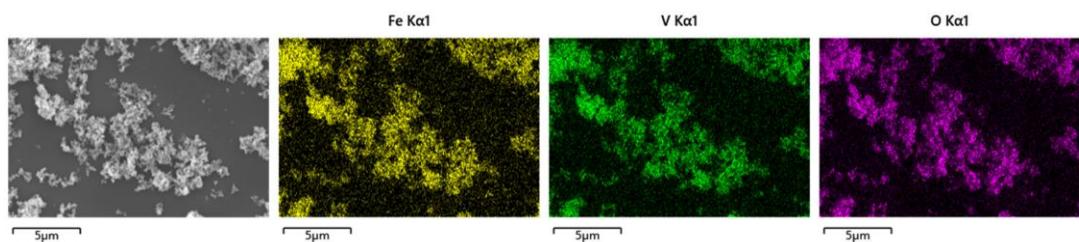


Fig. S8 Energy dispersive spectrometer (EDS) analysis of FVO-air NPs.

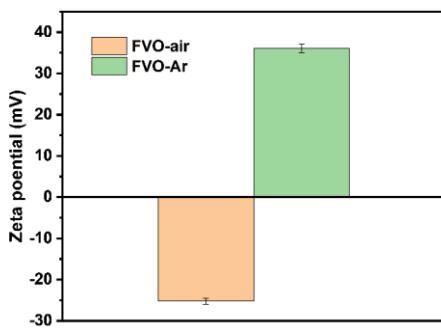


Fig. S9 Zeta potential of FVO-air NPs and FVO-Ar NPs. (n=3)

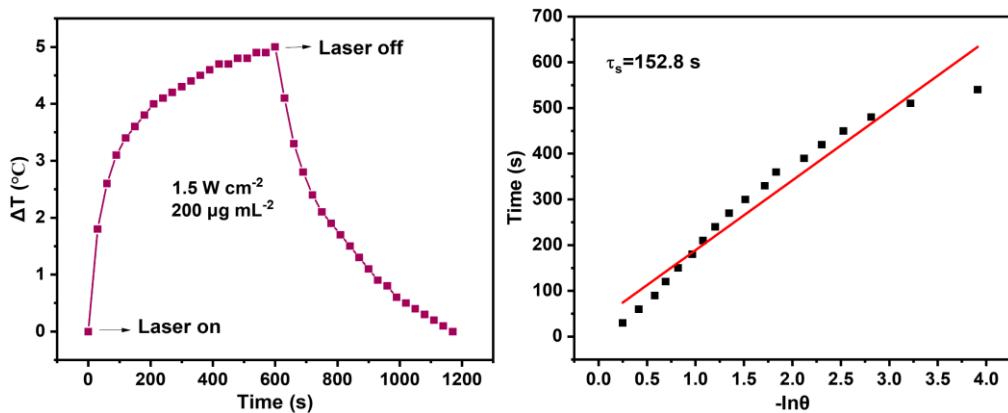


Fig. S10 Photothermal conversion test of FVO-air NPs. (1.5 W cm⁻²)

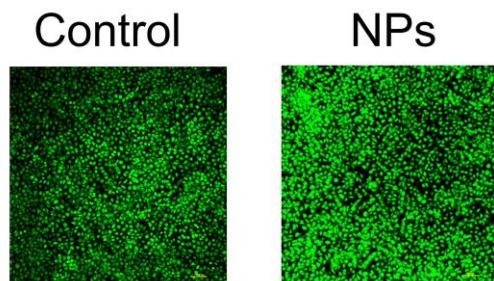


Fig. S11 With or without the addition of FVO-Ar NPs, L929 cells both showed green fluorescence. Scale bar: 100 µm.

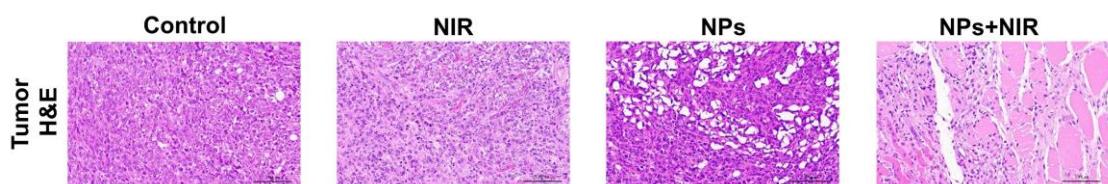


Fig. S12 H&E stained tumor slices on the ¹⁶th day of various treatments (Scale bar: 100 µm).

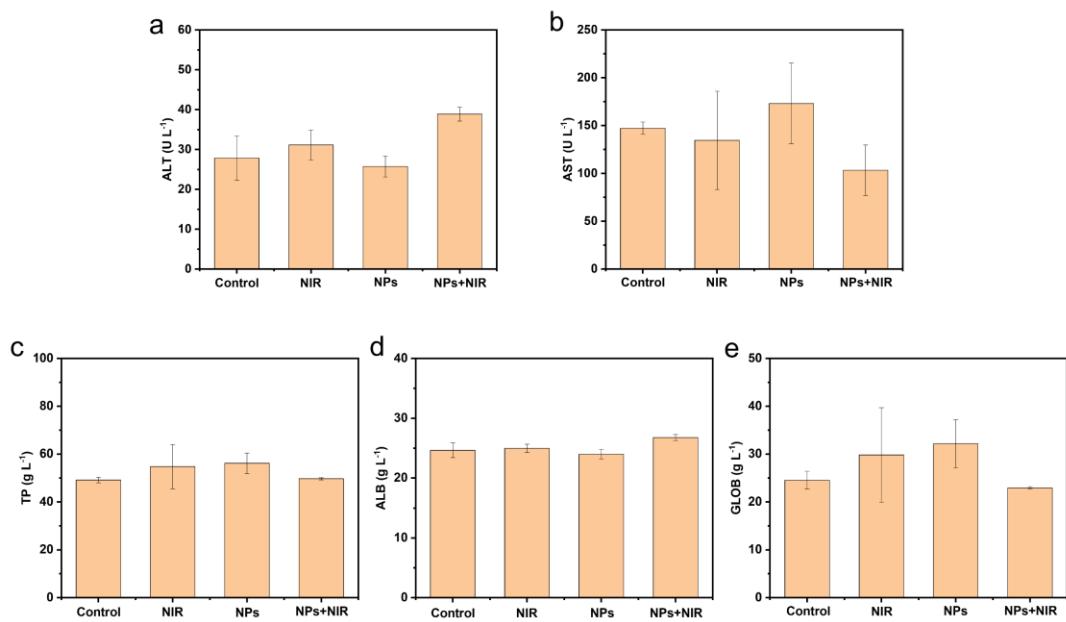


Fig. S13 The liver function indexes of mice after various treatments. (a) Alanine aminotransferase (ALT), (b) Aspartate aminotransferase (AST), (c) Total protein (TP), (d) Albumin (ALB), (e) Globulin (GLOB).