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Electronic Supplementary Information (ESI)

for

Continuous liquid-phase synthesis of nickel phosphide nanoparticles in a slug-flow microreactor

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1. Calculations on continuous preparation of nanoparticles:

(1)

The concentrations of each component are calculated by following Equation:

$$C_i = \frac{n_i}{V_{\text{total}}} = \frac{n_i}{\frac{n_{\text{TOP}} M_{\text{TOP}}}{\rho_{\text{TOP}}} + \frac{n_{\text{1-oct}} M_{\text{1-oct}}}{\rho_{\text{1-oct}}}}, \quad i = \text{Ni, TOP, 1-oct} \tag{S1-1}$$

(2)

Taking the thermal expansion of the flow into account, the residence time τ can be calculated as following Equation:

$$\tau = \frac{V_{\text{tube}}}{F} = \frac{\pi d^2 l/4}{L[1+\alpha(T-T_0)]} \text{(for single-phase process)} \tag{S2-1}$$

$$\text{or } \tau = \frac{V_{\text{tube}}}{F} = \frac{\pi d^2 l/4}{G[(T+273)/(T_0+273)] + L[1+\alpha(T-T_0)]} \text{(for two-phase process)} \tag{S2-2}$$

where F is actual flow rate at reaction temperature, l is the tube length, G and L are gas and liquid flow rates at room temperature respectively, T and T_0 are the reaction temperature and room temperature respectively, α is the thermal expansion coefficient of the liquid, typically $\alpha = 0.0007\ \text{K}^{-1}$ for long chain paraffins (taking 1-octadecene to be a paraffin).

(3)

The product yield was calculated by the following Equation:

$$Y = \frac{n_{\text{Ni}_2\text{P}}}{n_{\text{T}}} \times 100\% = \frac{m/M_{\text{Ni}_2\text{P}}}{n_{\text{Ni}}/2} \times 100\% \tag{S2-3}$$

where Y is the yield of Ni_2P, $n_{\text{Ni}_2\text{P}}$ and n_{T} are actual and theoretical mole product of Ni_2P respectively, m is the collected amount of Ni_2P, $M_{\text{Ni}_2\text{P}}$ is the molecular weight of Ni_2P, and n_{Ni} is the mole of $Ni(acac)_2$ feed.

2. Particle size distribution and SEM images:

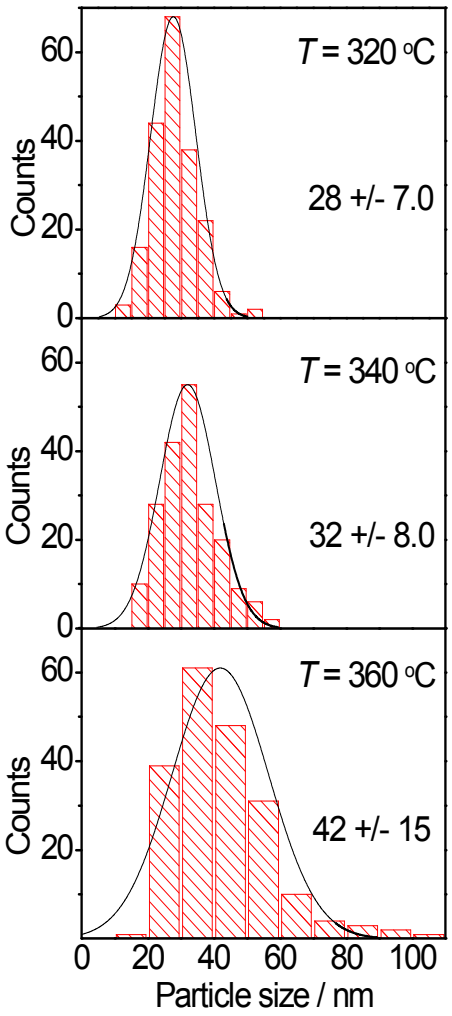

Figure S3. Particle size distributions of Ni_2P nanoparticles synthesized at 320, 340, and 360 °C; τ = 170 s; C_{Ni} = 0.05 M; C_{TOP} = 0.80 M; $C_{1\text{-oct}}$ = 1.70 M; n = 100.

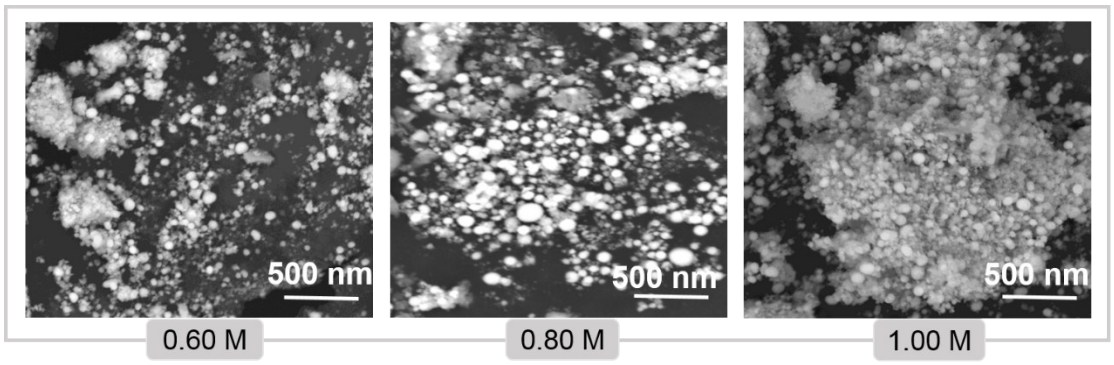

Figure S4. SEM micrographs of Ni_2P nanoparticles synthesized with TOP concentrations of 0.60, 0.80 and 1.00 M; T = 360 °C; L = 1.0 mL/min; τ = 170 s; P/Ni = 16.0; C_{Ni}= 0.05 M.

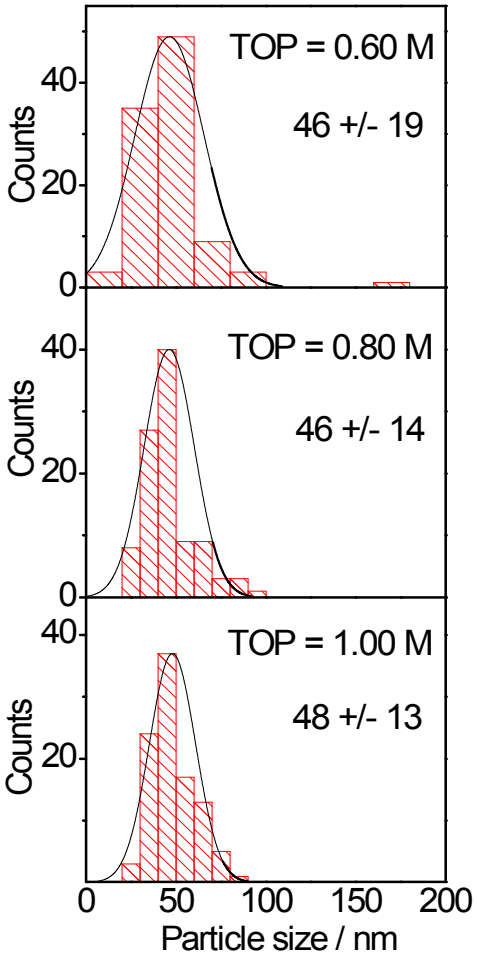

Figure S5. Particle size distributions of Ni_2P nanoparticles synthesized with TOP concentrations of 0.60, 0.80 and 1.00 M; T = 360 °C; L = 1.0 mL/min; τ = 170 s; P/Ni = 16.0; C_{Ni}= 0.05 M; n = 100.

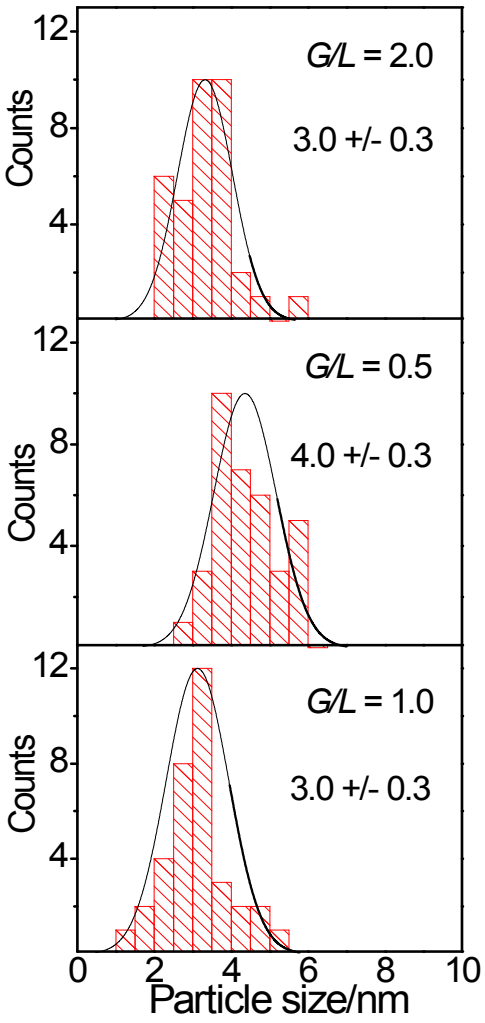

Figure S6. Particle size distributions of Ni_2P nanoparticles synthesized in different gas and liquid flow rates; T = 360 °C; C_{Ni}=0.05 M; C_{TOP} = 0.80 M; $C_{1\text{-oct}}$ = 1.70 M; n = 50.

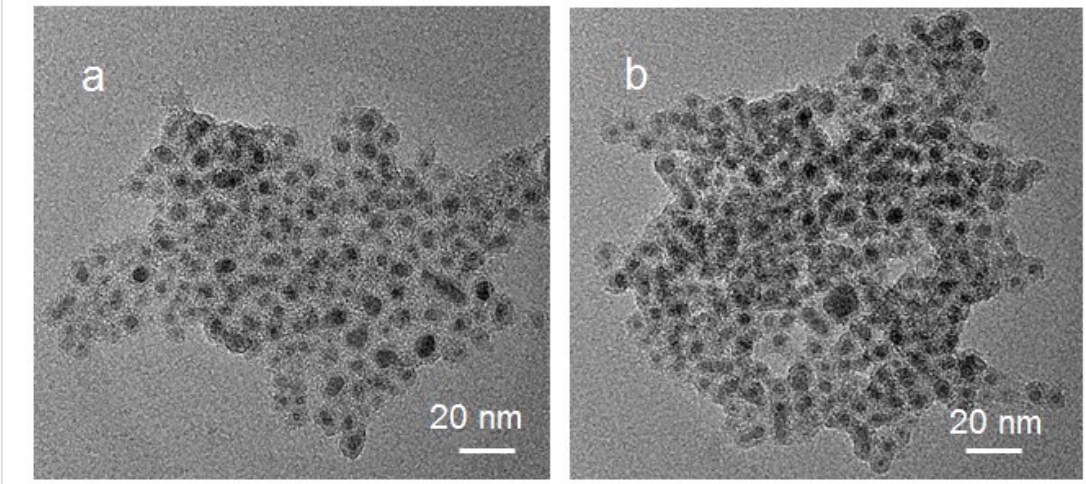

Figure S7. TEM micrographs of Ni_2P nanoparticles synthesized in G/L=0.5; T = 360 °C; C_{Ni}=0.05 M; C_{TOP} = 0.80 M; $C_{1\text{-}oct}$ = 1.70M.

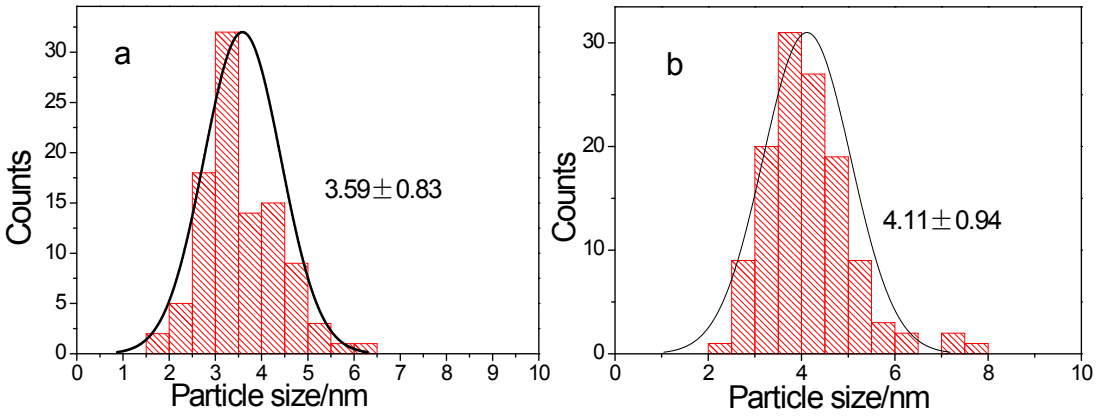

Figure S8. Particle size distributions of Ni_2P nanoparticles synthesized in G/L=0.5; T = 360 °C; C_{Ni}=0.05 M; C_{TOP} = 0.80 M; $C_{1\text{-oct}}$ = 1.70 M; n = 50.