

## Supplementary Information

### Surface-Tension-Free Fabrication to Minimize Defects in Cobalt-Silica Membranes via Freeze Drying Technique for H<sub>2</sub> Separation at High Temperatures

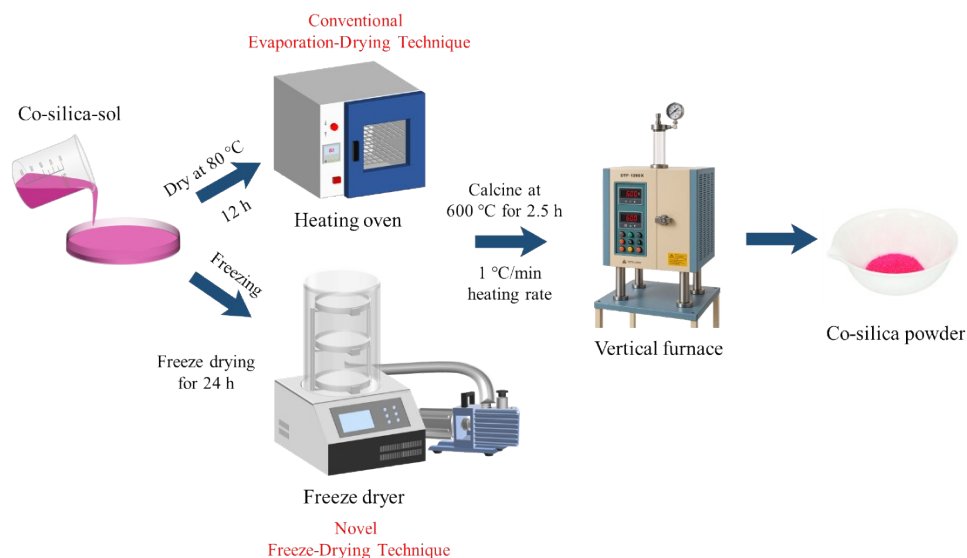
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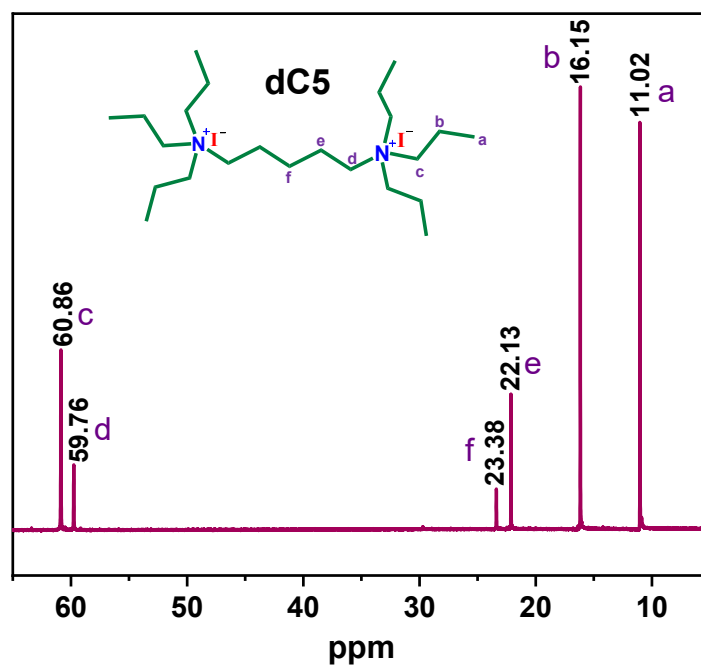
<sup>b</sup> School of Chemical Engineering, Dalian University of Technology, Dalian 116000, P. R. China

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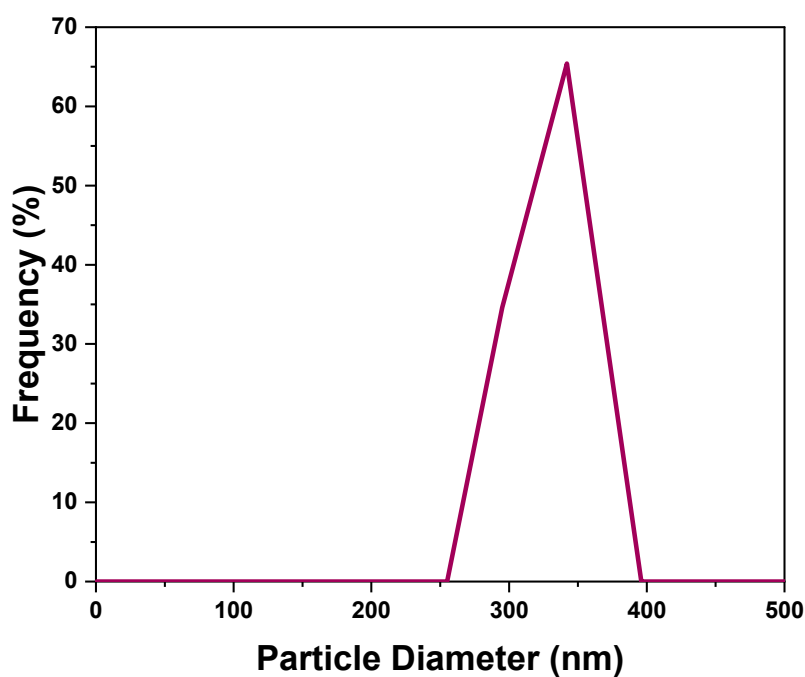
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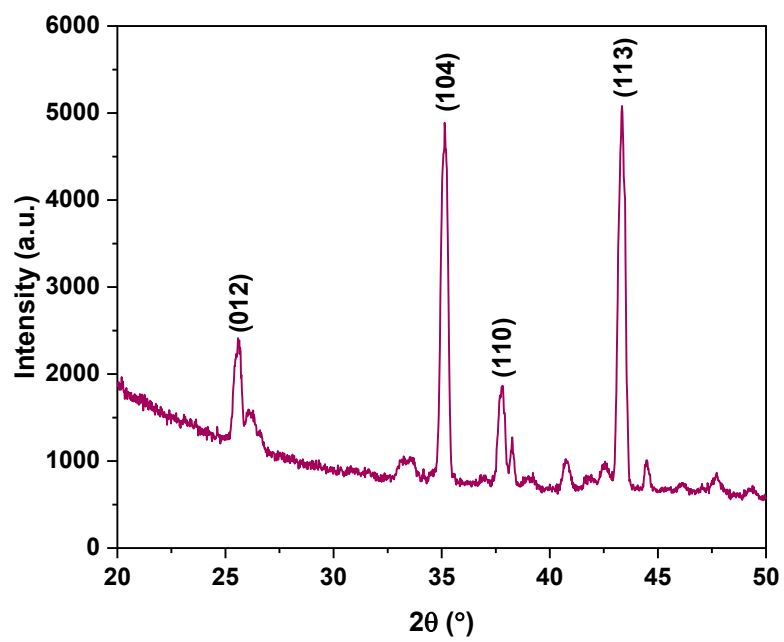
**Figure S1.** Schematic representation of the synthesis method of Co-silica powders via conventional evaporation-drying and novel freeze-drying technique



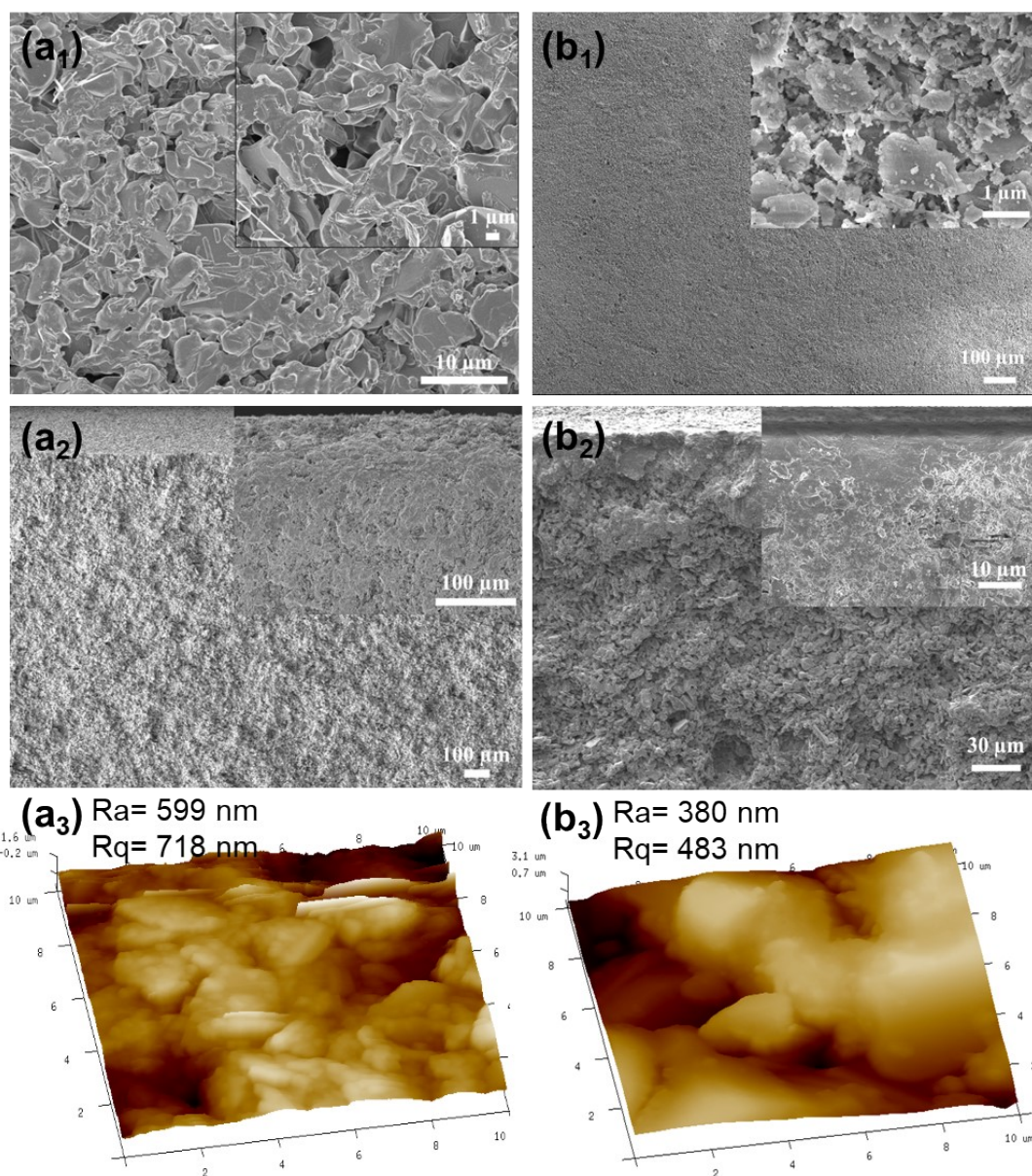
**Figure S2.**  $^{13}\text{C}$  NMR liquid spectrum of an aqueous solution of dC5 in deuterated chloroform ( $\text{CDCl}_3$ ) (The peaks of  $\text{CDCl}_3$  between 77.3 and 76.7 ppm are excluded from the graph)



**Figure S3.** Dynamic Light Scattering (DLS) analysis of cobalt-silica sol



**Figure S4.** X-ray diffraction (XRD) analysis of  $\alpha$ -alumina supports



**Figure S5.** SEM surface and cross-section images with higher magnification in the insets and AFM images showing average roughness (Ra) and root mean square roughness (Rq), respectively (a<sub>1</sub>, a<sub>2</sub>, a<sub>3</sub>)  $\alpha$ -alumina substrate; (b<sub>1</sub>, b<sub>2</sub>, b<sub>3</sub>) MFI nanosheet interlayer