## **Electronic Supplementary Information**

Design, controlled synthesis, and property of 2D CeO<sub>2</sub>/NiO heterostructure assemblies

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Figure S1. FT-IR spectra of (a) Ni(OH)<sub>2</sub>, (b) CeO<sub>2</sub>-Ni(OH)<sub>2</sub>, and (c) CeO<sub>2</sub>/NiO

samples.

Figure S1 presents the FT-IR spectra of the different samples. Spectrum a is typical of  $\alpha$ -Ni(OH)<sub>2</sub> product prepared by the urea method.18,20 The intense and broad band around 3440 cm<sup>-1</sup> is due to the vO-H vibration of H-bonded water molecules located in the interlamellar space of  $\alpha$ -Ni(OH)<sub>2</sub>. The shoulder band at 3640 cm<sup>-1</sup> corresponds to the vO-H stretching of the free OH groups of the brucite-like structure. The very strong absorption band around 2200 cm<sup>-1</sup> is the typical vibration of C=N triple bonds in the OCN<sup>-</sup> anions, the byproducts of urea hydrolysis.<sup>1</sup> The absorption at around 1470 cm<sup>-1</sup> is assigned to the vC=O in the carbonate ions.<sup>2</sup> The bands at around 1385 cm<sup>-1</sup>, 1291 cm<sup>-1</sup>, and 830cm<sup>-1</sup> are ascribed to the vibration of NO<sub>3</sub><sup>-</sup> ions,<sup>2</sup> which comes from the nickel source. The two bands around 649–669 and 475 cm<sup>-1</sup> are ascribed to the  $\delta$ OH and vNi–OH vibrations, respectively.<sup>3</sup> The bands at around 1600 and 1430 cm<sup>-1</sup> are assigned to the vC=O of carbonate ions.<sup>4</sup> Therefore, it can be concluded that sample consists of intercalated water and anions within the interlamellar space of  $\alpha$ -Ni(OH)<sub>2</sub>. After the growth of CeO<sub>2</sub>, spectrum b shows similar band characteristics with weakened C=N and NO<sub>3</sub><sup>-</sup> bands. After calcination, the band characteristic of  $\alpha$ -Ni(OH)<sub>2</sub> disappears (spectrum c), the hydroxides transform into oxides. The bands at 3440 cm<sup>-1</sup> and 1630 cm<sup>-1</sup> can be attributed to the O-H vibration of adsorbed water on the sample surface.<sup>5</sup>. The broad band of 800-400 cm<sup>-1</sup> can be attributed to metal oxides.



Figure S2. XRD patterns and SEM image of product obtained with lower quantity of urea (2 mmol)



Figure S3. XRD patterns and SEM image of product obtained with higher quantity of urea (12 mmol)

## References

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