**Supporting Information**
**for New Journal of Chemistry**

# Supercritical $CO_2$-assisted deposition of NiO on (101)-anatase-$TiO_2$ for efficient heterostructured photocatalysts

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*Powder X-ray diffraction*

XRD pattern of pure NiO prepared by the supercritical fluid chemical deposition (SFCD) method is shown in Fig. S1.

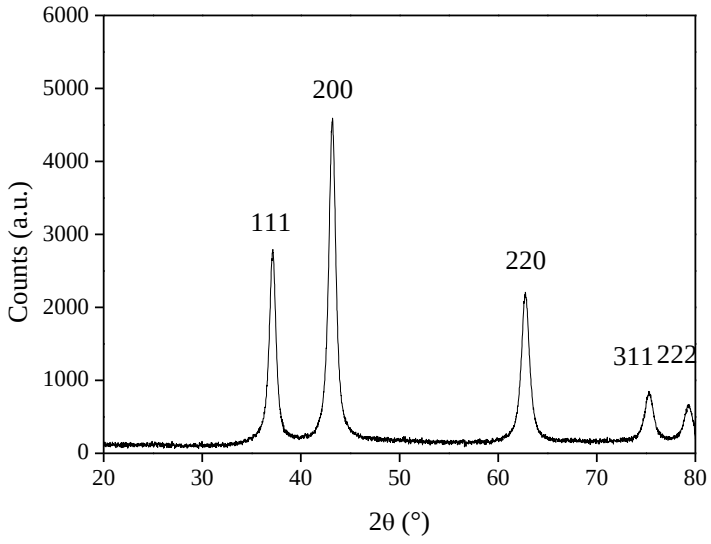


**Fig. S1**: XRD pattern of pure NiO synthesized by the SFCD route.

*Transmission electron microscopy analyses*

Bright field TEM images of 0.25wt%, 1wt% and 10 wt% NiO-$TiO_2$ nanocomposites are shown in Fig. S2.

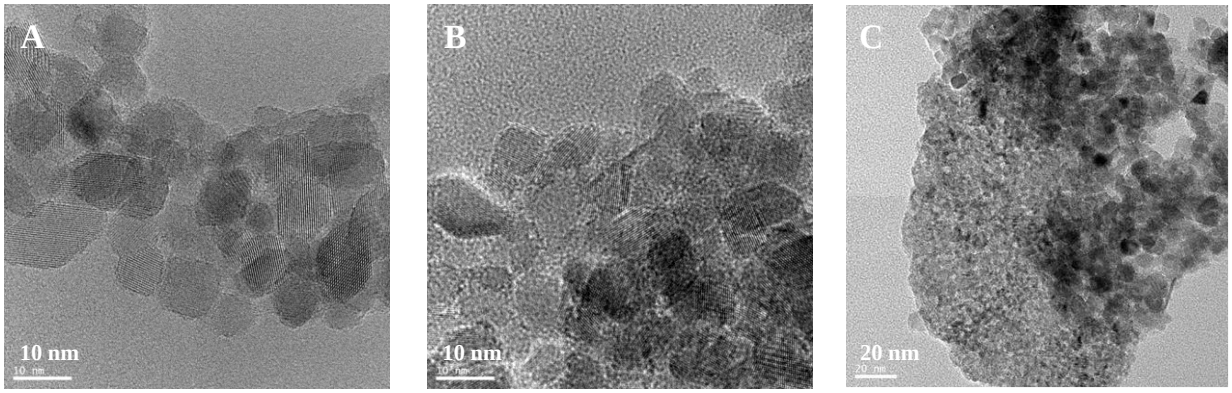


**Fig. S2**: Bright field TEM images of 0.25wt% NiO-$TiO_2$ (**A**), 2wt% NiO-$TiO_2$ (**B**) and 10 wt% NiO-$TiO_2$ (**C**) nanomaterials.

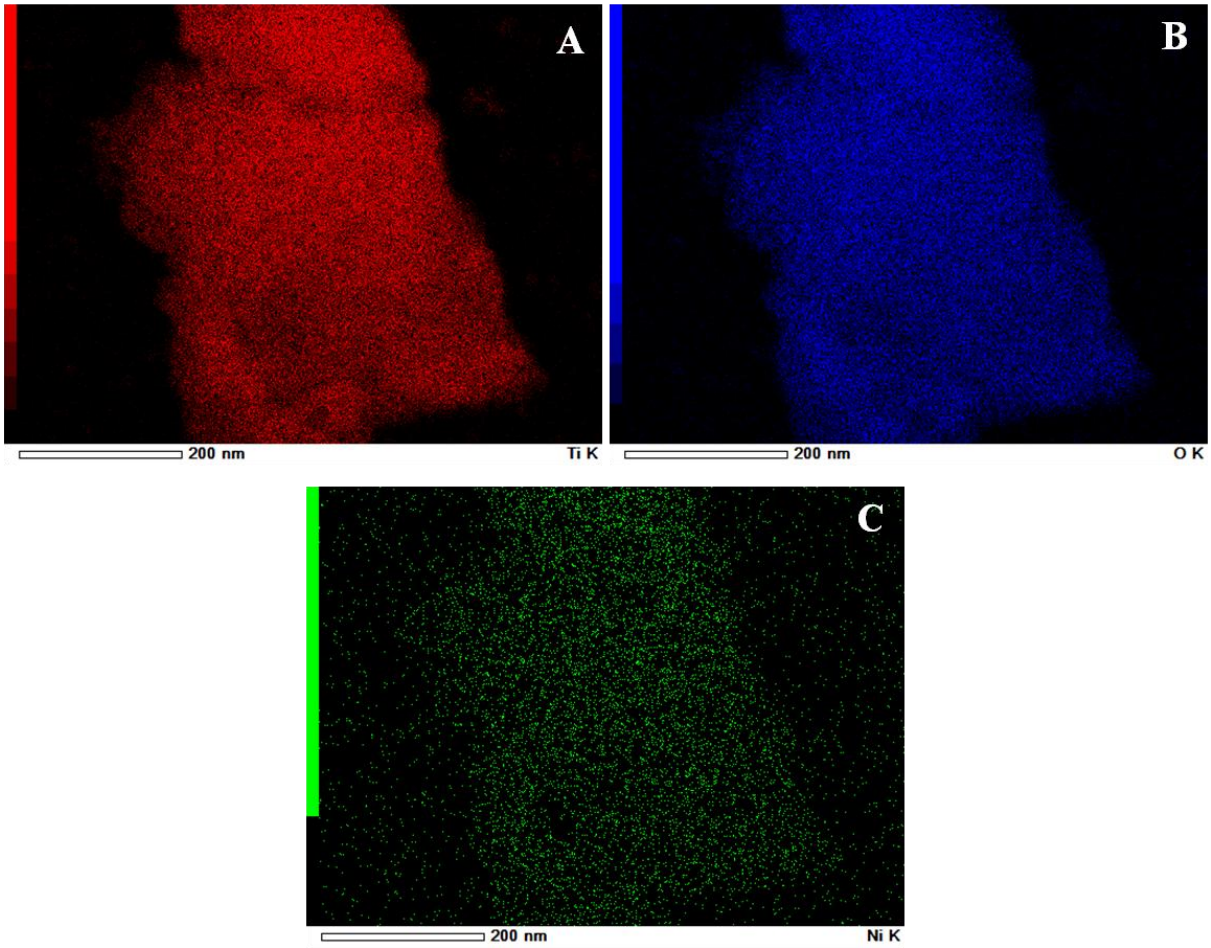


**Fig. S3**: Expand of STEM-EDX elemental mapping of (**A**) Ti, (**B**) O, and (**C**) Ni for 2wt% NiO-$TiO_2$ nanomaterials.

Bright field STEM image of 10 wt% NiO-$TiO_2$ nanocomposite and corresponding elemental mapping for O, Ni and Ti are shown in Fig. S4. Semiquantitative analysis indicates that this sample contains 9wt% NiO which is in accordance with the nominal NiO amount.

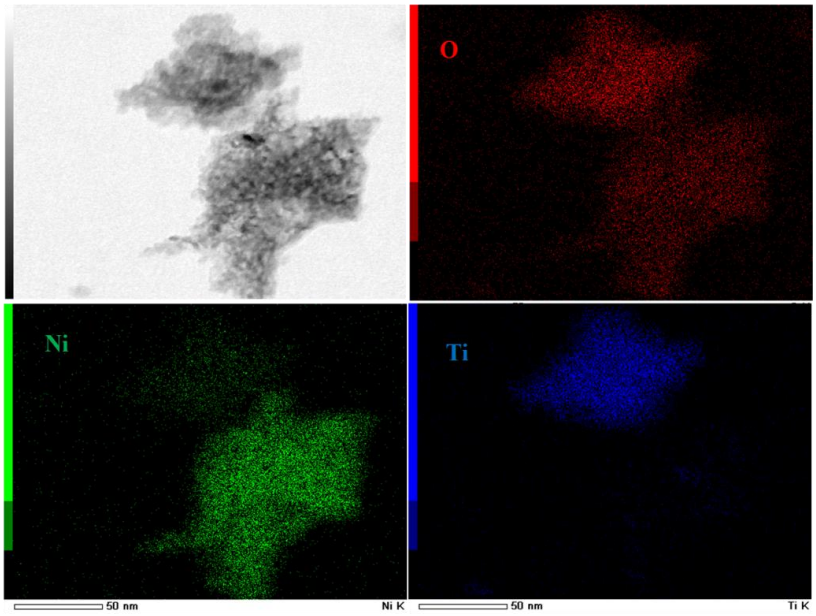


**Fig. S4** (**A**) Bright field STEM image of the 10 wt% NiO-$TiO_2$ nanomaterials and EDX elemental mapping of (**B**) O, (**C**) Ni and (**D**) Ti.

*$N_2$ sorption analysis*

$N_2$ adsorption-desorption isotherms and pore size distribution (BJH model applied to the adsorption branch) are shown in Fig. S5.

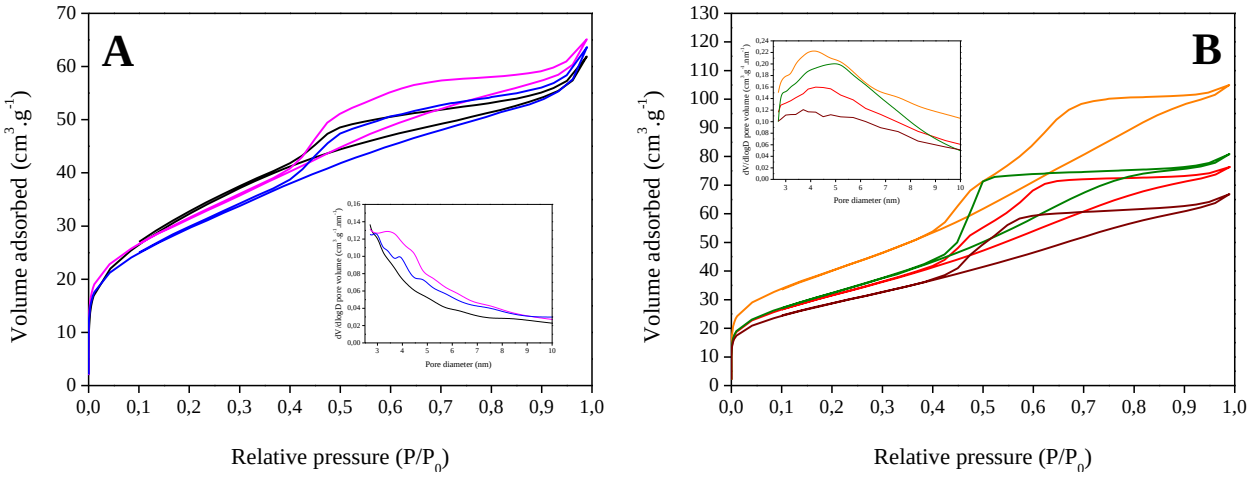


**Fig. S5**: Nitrogen gas adsorption-desorption isotherms and pore-size distribution (inset) of (**A**) pure $TiO_2$ (black), 0.25wt% $NiO/TiO_2$ (magenta) and 0,5wt% $NiO/TiO_2$ (blue) nanomaterials; (**B**) 0.1wt% $NiO/TiO_2$ (orange), 1wt% $NiO/TiO_2$ (red), 2wt% $NiO/TiO_2$ (olive) and 10 wt% $NiO/TiO_2$ (brown) nanomaterials.

Before the $N_2$ sorption study of $TiO_2$-NiO nanocomposites, the accuracy of the Micromeritics ASAP2010 equipment was checked by recording the adsorption-desorption isotherm of a silica-alumina reference material from Micromeritics (Ref: 004-16821-02), the characteristics of which are: $S_{BET}$ = 214 ± 6 $m^2.g^{-1}$; Total pore volume = 0.63 ± 0.08 $cm^3.g^{-1}$; BJH mean pore size = 11.5 ± 1.5 nm. As a consequence, the relative uncertainty concerning $S_{BET}$, total pore volume and BJH mean pore size, determine by this method, are therefore about ± 3%, ± 13% and ± 13%, respectively. These relative uncertainties have been used to report the values given in Table 1.

*X-ray Photoelectron Spectroscopy*

XPS survey spectra of pure $TiO_2$ and NiO-$TiO_2$ nanocomposites are depicted in Fig. S6.

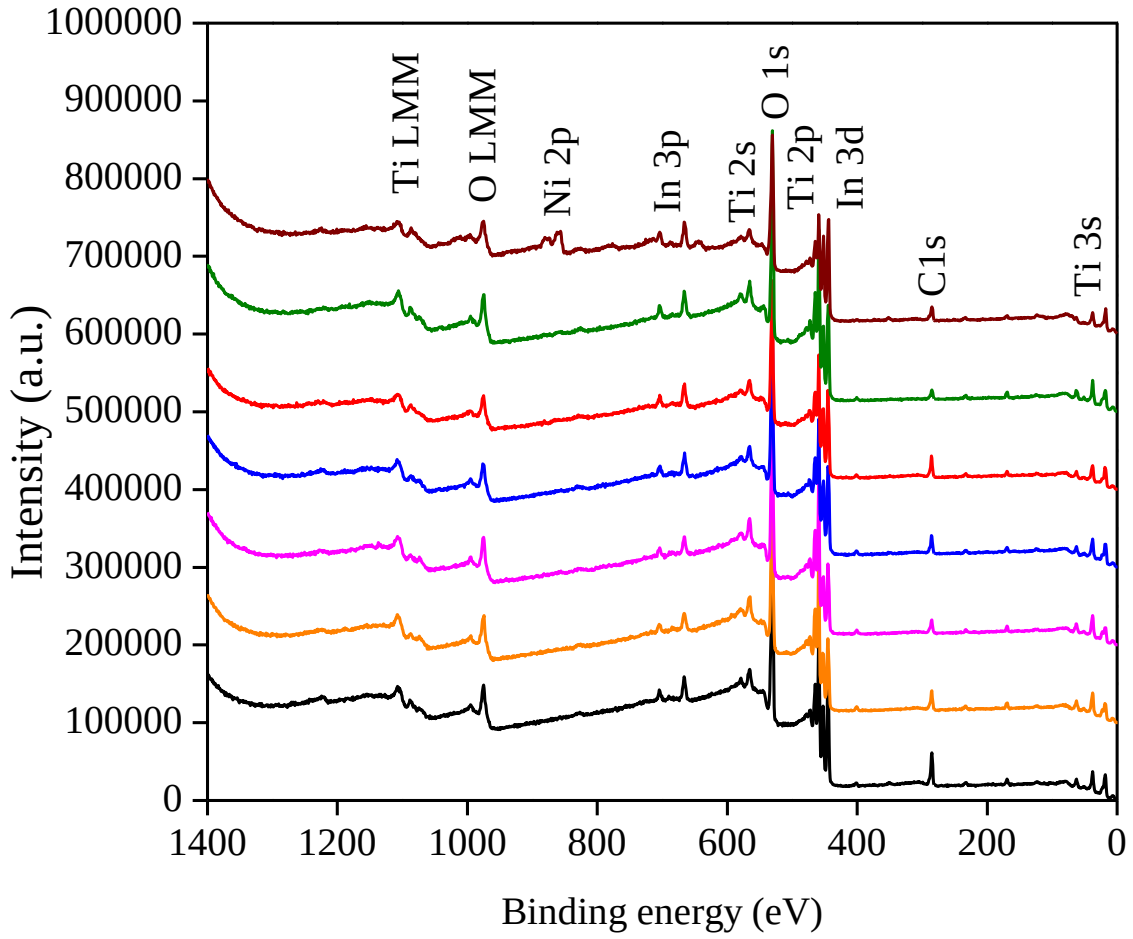


**Fig. S6**: XPS survey spectra of pure $TiO_2$ (black), 0.1wt% NiO-$TiO_2$ (orange), 0.25wt% NiO-$TiO_2$ (magenta), 0,5wt% NiO-$TiO_2$ (blue), 1wt% NiO-$TiO_2$ (red), 2wt% NiO-$TiO_2$ (olive) and 10 wt% NiO-$TiO_2$ (brown) nanomaterials.

*Photocatalysis studies*

The optical density (OD) increased by up to 10 % and changed randomly by up to ±10 % after stirring with the photocatalysts for MB and MO solution, respectively (Table S1). Also, the position of the main peak in MO showed a red shift while that of MB remains at the same position. This modification of absorption spectra arises from change of pH. Absorption spectra of MB and MO solutions with various pH values were investigated by adjusting pH with ammonia and changes of the absorption intensity are summarized in Table S2. For MB, the OD increases by 5.0-12.1 % with decrease of pH from 7.0 to 3.0-5.0 which corresponds to the value of pH measured after stirring the solution with photocatalysts for 30 min and before the irradiation of UV light. For MO, the OD decreases slightly with decrease of pH from 7.0 to 5.0 and changes drastically and randomly with pH between 3.0 and 4.0. The random change of the OD with the photocatalysts can be explained by the pH effect as the pH of the solution with the photocatalysts are between 3.25 and 4.05 almost corresponding to the above mentioned pH region which causes drastic changes of OD. Thus, it could be assumed that the changes of the OD after stirring with the photocatalysts are mainly attributed to pH effect and adsorption of dye onto the samples would be negligible for both MB and MO. The position of the main peak of MO shifts towards red region with decrease of pH, confirming that the pH effect is responsible for the red shift of the peak position in MO with the photocatalysts.

**Table S1.** Change of OD of the most intense peak in MB and MO solution with the pure $TiO_2$ and the NiO-$TiO_2$ nanocomposites with respect to that without the photocatalysts.

| | MB | | MO | |
|---|---|---|---|---|
| Sample | pH | OD change (%) | pH | OD change (%) |
| $TiO_2$ | 3.15 | 8.1 | 3.25 | - 6.2 |
| 0.1wt% NiO-$TiO_2$ | 4.13 | 7.4 | 4.03 | - 3.1 |
| 0.25wt% NiO-$TiO_2$ | 3.43 | 3.7 | 3.43 | 9.9 |
| 0.5wt% NiO-$TiO_2$ | 3.85 | 3.7 | 3.75 | 6.7 |
| 1wt% NiO-$TiO_2$ | 3.71 | 10.3 | 3.70 | 0.1 |
| 2wt% NiO-$TiO_2$ | 4.35 | 7.4 | 4.05 | - 3.4 |
| 10wt% NiO-$TiO_2$ | 6.44 | 0.6 | 5.87 | 0.2 |

**Table S2.** Change of OD of the most intense peak in MB and MO solution with various pH values between 2.0 and 6.0 with respect to that with pH = 7.

| | OD Change | |
|---|---|---|
| pH | MB | MO |
| 2.0 | 27.4 | 53.4 |
| 3.0 | 6.7 | 39.8 |
| 4.0 | 7.1 | - 7.2 |
| 5.0 | 12.1 | - 3.6 |
| 6.0 | 5.0 | - 2.1 |

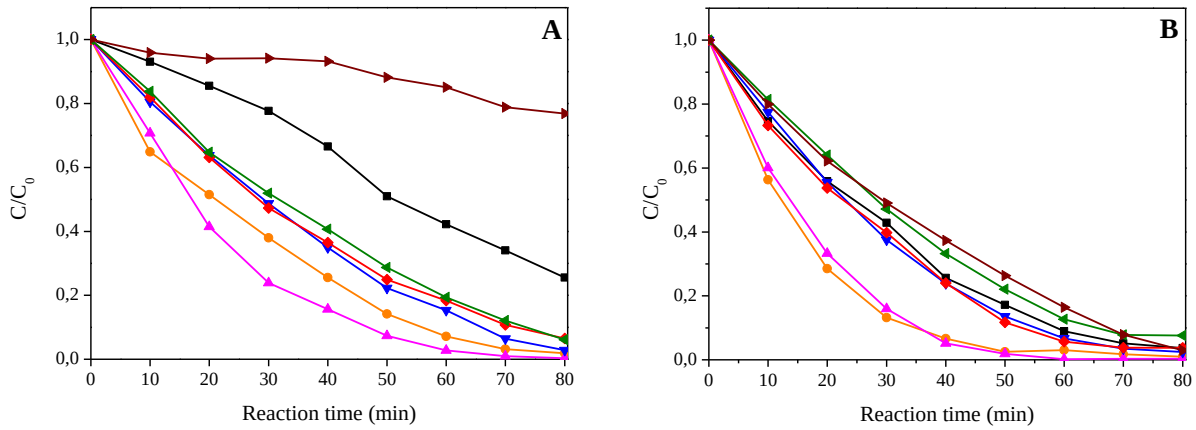


**Fig. S7**: Kinetic of the degradation of MB (**A**, left) and MO (**B**, right) of pure $TiO_2$ (black), 0.1wt% NiO-$TiO_2$ (orange), 0.25wt% NiO-$TiO_2$ (magenta), 0,5wt% NiO-$TiO_2$ (blue), 1wt% NiO-$TiO_2$ (red), 2wt% NiO-$TiO_2$ (olive) and 10 wt% NiO-$TiO_2$ (brown) nanomaterials.