

Heterogeneous Photocatalysed Heck reaction over PdCl₂/TiO₂

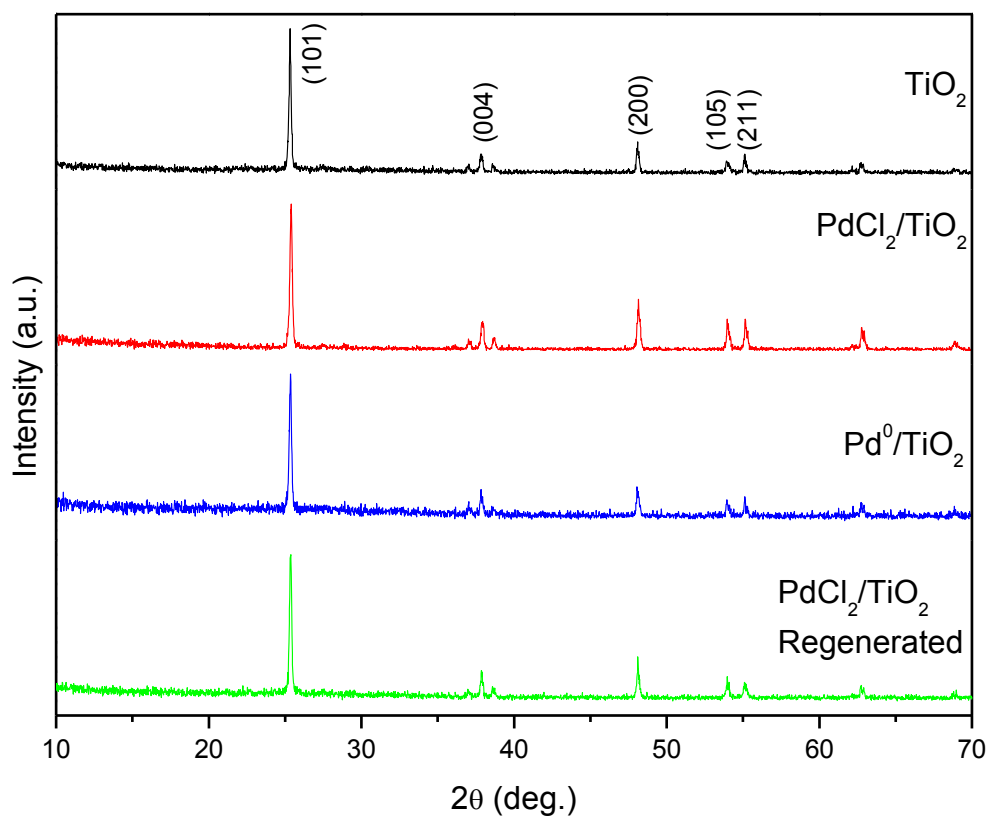
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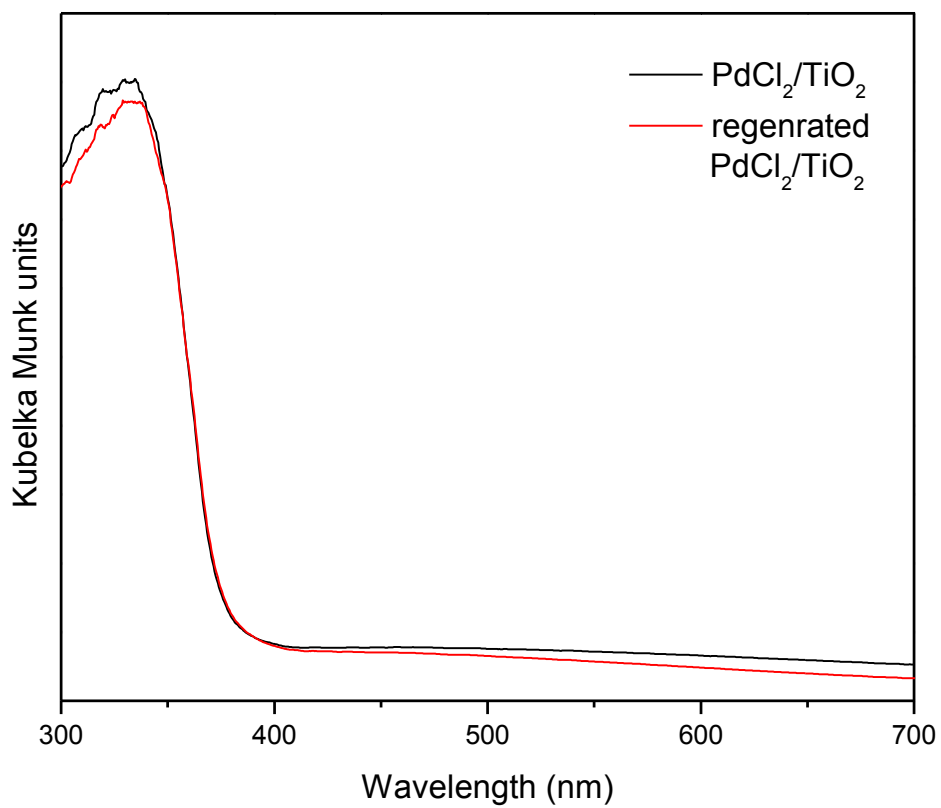
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FigureS1. XRD pattern of the a) TiO_2 , b) $\text{PdCl}_2/\text{TiO}_2$, c) used $\text{PdCl}_2/\text{TiO}_2$ and d) regenerated $\text{PdCl}_2/\text{TiO}_2$.



FigureS2. Diffuse reflectance UV-visible absorption profile of PdCl₂/TiO₂.

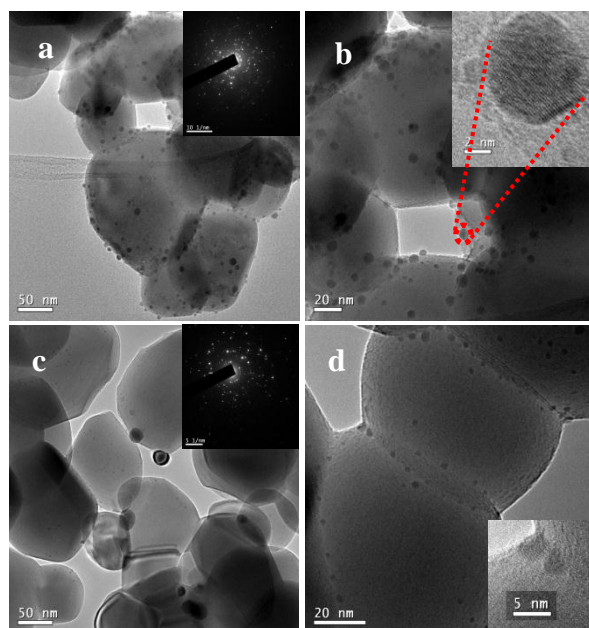


Fig.S3. TEM images of a, b) $\text{PdCl}_2/\text{TiO}_2$ before reaction and c, d) regenerated $\text{PdCl}_2/\text{TiO}_2$. Inset of Fig. a and c is the SAED pattern of $\text{PdCl}_2/\text{TiO}_2$

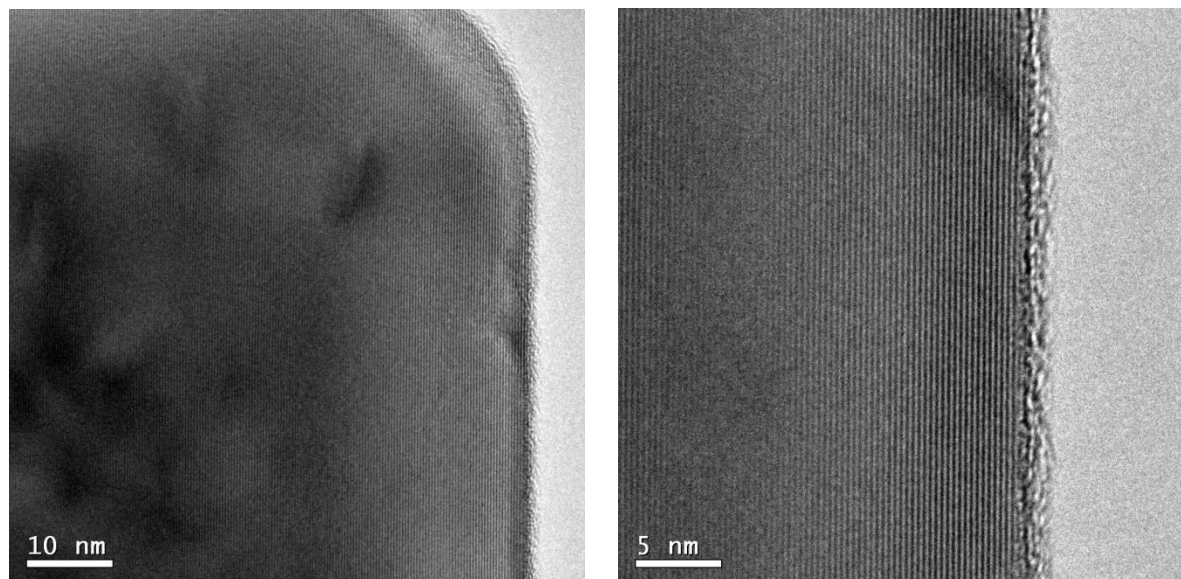


Figure S4. TEM of TiO₂ particles

Measurement of Mercury Lamp Intensity:

The intensity of lamp measured by irradiating ferrioxalate solution by following procedure¹⁻²

Preparation of solution

1. Potassium ferrioxalate:-

Weight 3.2 g of ferric chloride and dissolved in a beaker containing 8 mL of distilled water. 11.6 g of potassium oxalate was dissolved in 35 mL water in another beaker and warmed it. To the hot solution of potassium oxalate, ferric chloride solution was slowly added with constant stirring. The clear solution was then filtered (in hot condition), cooled to room temperature and then in ice bath. The crystals obtained were recrystallized with water and stored in dark.

2. Ferrioxalate solution:-

0.738 g of potassium ferrioxalate was dissolved in 200 mL of water to that 25 mL of 1 N H₂SO₄ was added and diluted to 250 mL.

3. Sodium acetate trihydrate buffer:-

13.6% solution of sodium acetate trihydrate was prepared with dilution of water. 36 mL of 1N H₂SO₄ was added to 60 mL of sodium acetate trihydrate solution (13.6%) and diluted to 100 mL by water.

4. 1,10 Phenanthroline:-

0.1% solution of *o*-phenanthroline was prepared with dilution of water.

Procedure

- i) Pipette out 5 mL (V_1) of ferrioxalate solution into the cuvette and irradiated under the source whose intensity is to be measured in the irradiation chamber.
- ii) 2 mL (V_2) of irradiated solutions were mixed with 2 mL of *o*-phenanthroline solution and 1 mL of buffer solution in 25 mL (V_3) of volumetric flask and then diluted up to mark. 2 mL (V_2) of non-irradiated solution of ferrioxalate was treated exactly the same way and serves as reference solution.
- iii) The irradiation time was varied as 0, 20, 40, 50, 60 and 70 min.
- iv) All solutions were kept in dark for 1 hour (for completion of reaction).
- v) Then absorbance was measured for all the solutions at wavelength 510 nm.

Note: All processing steps have been performed in dark.

Photon flux was calculated using following formula

$$\text{Photon flux} = \frac{D \times V_1 \times V_3 \times 10^{-3} \times N_A}{t \times \Phi \times \epsilon \times d \times V_2}$$

Table 1. Measurement of photon flux

Sr. No.	Time/min	Absorbance	Photon flux/s ⁻¹	Mean/s ⁻¹
1	0	0.189		2.1914 x 10 ¹²
2	20	1.268	4.164 x 10 ¹²	
3	40	1.246	2.047 x 10 ¹²	
4	50	1.370	1.800 x 10 ¹²	
5	60	1.402	1.535 x 10 ¹²	
6	70	1.503	1.411 x 10 ¹²	

References:

- 1) C. A. Parker, *Proc. R. Soc. Lond. A* 1953, **220**, 104-116; doi: 10.1098/rspa.1953.0175.
- 2) C. G. Hatchard, C. A. Parker, *Proc. R. Soc. Lond. A* 1956, **235**, 518-536; doi: 10.1098/rspa.1956.0102.