Electronic Supplementary Information (ESI)

The effect of Pt NPs crystallinity and distribution on the photocatalytic activity of Pt-g-C₃N₄

F. Fina, a H. Ménard b and J. T. S. Irvine* a

a School of Chemistry, University of St Andrews, St Andrews, KY16 9ST, Scotland, UK.

b Sasol Technology (UK) Ltd., St Andrews, KY16 9ST Scotland, UK.

![XPS Cl 2p spectra for Pt-g-C₃N₄](image)

**Fig.S1** XPS Cl 2p for Pt-g-C₃N₄ a) impregnated and b) impregnated followed by calcination before photocatalytic test.
Fig.S2 XPS Pt 4f region for the calcined Pt-g-C$_3$N$_4$ after photocatalysis.

Fig.S3 XPS Pt 4f region of the impregnated Pt-g-C$_3$N$_4$ showing the evolution of the platinum peak after photocatalytic test for different lengths of time.
Fig. S4 Steady state H₂ evolution rate for impregnated Pt-g-C₃N₄ reduced under 5 % H₂/Ar for different lengths of time (12 h, 24 h, 36 h and 48 h). Test conditions: 0.1 g of catalyst in 0.1 L of 0.05 M aqueous solution of oxalic acid, visible light (λ ≥ 420 nm).

Table S1 Platinum species relative amounts and Pt:N ratio for Pt-g-C₃N₄ reduced in 5 % H₂/Ar for 24 h and 48 h.

<table>
<thead>
<tr>
<th>Reduction length (h)</th>
<th>PtO %</th>
<th>Pt⁰ %</th>
<th>Pt:N</th>
</tr>
</thead>
<tbody>
<tr>
<td>24</td>
<td>44.2</td>
<td>55.8</td>
<td>0.051</td>
</tr>
<tr>
<td>48</td>
<td>37.7</td>
<td>62.36</td>
<td>0.052</td>
</tr>
</tbody>
</table>

Fig. S5 TEM and HRTEM of Pt-g-C₃N₄ reduced in 5 % H₂/Ar for 24 h. Inset-a) Particle size distribution.
**Fig. S6** TEM and HRTEM of Pt-g-C$_3$N$_4$ reduced with NaBH$_4$. Inset-c) Particle size distribution.

**Fig. S7** TEM and HRTEM of Pt-g-C$_3$N$_4$ double reduced with NaBH$_4$ and 5 % H$_2$/Ar. Inset-c) Particle size distribution.