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Electronic Supplementary Information

A new reduction method based on simultaneous Ti₃AlC₂ support etching and metal deposition to prepare Pt catalysts for aqueousphase selective hydrogenation of furfural to furfuryl alcohol

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High resolution XPS spectra of C1s and O1s and detailed information on binding energies and deconvoluted peaks.



Figure s1. High resolution XPS spectra and deconvoluted peaks of C1s (a) and O1s (b) of samples $Ti_3Al_xC_2T_y$, $Pt_{0.5}/Ti_3Al_xC_2T_y$, $Pt_{1.0}/Ti_3Al_xC_2T_y$, $Pt_{2.0}/Ti_3Al_xC_2T_y$, and $Pt_{3.0}/Ti_3Al_xC_2T_y$.

Table s1. Detailed binding energies and fractions of sub-species deconvoluted from high-resolution XPS

Catalysts		C1s			O1s		
						Surface	Adsorbed
		C-C	C-0	COO	Ti-O	O-species	O-species
						e.gOH	e.g. H ₂ O
Ti ₃ Al _x C ₂ T _y	BE / eV	284.8	286.3	288.5	529.6	531.3	533.9
	Fractions / %	69.6	18.9	11.5	68.5	24.7	6.8
Pt _{0.5} /Ti ₃ Al _x C ₂ T _y	BE / eV	284.4	286	288.3	529.6	531.6	534
	Fractions / %	77.2	12.3	10.5	75.5	19	5.5
Pt _{1.0} /Ti ₃ Al _x C ₂ T _y	BE / eV	284.6	286	288.6	529.6	531.7	533.7
	Fractions / %	70.8	18.2	11	71	22.9	6.1
Pt _{2.0} /Ti ₃ Al _x C ₂ T _y	BE / eV	284.7	286.3	288.6	529.7	531.4	534
	Fractions / %	72.8	16.1	11.1	79.7	15.4	4.9
Pt _{3.0} /Ti ₃ Al _x C ₂ T _y	BE / eV	284.6	286.1	288.6	529.5	531.5	533.4
	Fractions / %	73.2	15.7	11.1	68.7	22.9	8.4

spectra of C1s and O1s.

Table s2. Ratio of metallic Pt and oxidized Pt quantified by XPS analysis

Catalyst	Bulk Pt loading	Metallic Pt	Pt-oxidized	
	/ wt% (by ICP)	/ % (by XPS)	/ % (by XPS)	
$Pt_{0.5}/Ti_3Al_xC_2T_y$	0.38	89.5	10.5	
Pt _{1.0} /Ti ₃ Al _x C ₂ T _y	0.76	89.4	10.6	
$Pt_{2.0}/Ti_3Al_xC_2T_y$	1.45	96.7	3.3	
$Pt_{3.0}/Ti_3Al_xC_2T_y$	2.62	95.4	4.6	

Samples	Real loading of Pt	Catalyst mass	Pt mass	Reaction rate	Reaction rate
	/ wt%	/ mg	/ mg _{Pt}	/ mmol _{FUR} ·h ⁻¹	/ mmol _{FUR} ·g _{Pt} ⁻¹ ·h ⁻¹
$Pt_{0.5}/Ti_3Al_xC_2T_y$	0.38	100	0.38	0.223	586.0
Pt _{1.0} / Ti ₃ Al _x C ₂ T _y	0.76	100	0.76	0.514	676.3
$Pt_{2.0}/Ti_3Al_xC_2T_y$	1.45	100	1.45	0.667	459.8
$Pt_{3.0}/Ti_3Al_xC_2T_y$	2.62	100	2.62	0.667	254.5
3.0%Pt/ Ti ₃ Al _x C ₂ T _y	0.63	100	0.63	0.183	291.0
3.0%Pt/TiO ₂	1.84	100	1.84	0.165	89.9
3.0%Pt/AC	0.78	100	0.78	0.3	384.6

Table s3: Detailed information on catalysts and calculation reaction rates.

Section 3

The N₂ adsorption-desorption isotherms and pore size distributions of selected samples of $Ti_3AlC_2(0.6 \text{ m}^2/\text{g})$, $Ti_3Al_xC_2T_y(2.9 \text{ m}^2/\text{g})$ and $Pt_{2.0}/Ti_3Al_xC_2T_y(12.7 \text{ m}^2/\text{g})$.



Figure s2. (a) The N₂ adsorption-desorption curves and (b) the pore size distributions of three selected samples: Ti_3AIC_2 , $Ti_3AI_xC_2T_y$ and $Pt_{2.0}/Ti_3AI_xC_2T_y$.

The SEM images of catalysts showed that most products were fibrous nanomaterials as expected, which were conducive to recycle from aqueous reaction systems.



 $\textbf{Figure s3. SEM images of (a) } Pt_{0.5}/Ti_{3}Al_{x}C_{2}T_{y}, \textbf{(b)} Pt_{1.0}/Ti_{3}Al_{x}C_{2}T_{y}, \textbf{(c)} Pt_{2.0}/Ti_{3}Al_{x}C_{2}T_{y}, \textbf{(d)} Pt_{3.0}/Ti_{3}Al_{x}C_{2}T_{y}, \textbf{(d)} Pt_{3.0}/Ti$

The Pt particle sizes of benchmark catalysts of Pt/TiO_2 and Pt/AC were analyzed on the basis of TEM images using the same method. The histograms of them were shown in **Figure s4** as insets, which demonstrated that the size of Pt NPs was 3-10 nm on TiO₂ and was 2-9 on AC.



Figure s4. The TEM image (a) and the HR TEM image (b) of 3%Pt/TiO₂ (mean size: 6.3 nm); the TEM image (c) and the HR TEM image (d) of 3%Pt/AC (mean size 5.8 nm).

TEM images of four catalysts: $Pt_{0.5}/Ti_3Al_xC_2T_y$, $Pt_{1.0}/Ti_3Al_xC_2T_y$, $Pt_{2.0}/Ti_3Al_xC_2T_y$, and $Pt_{3.0}/Ti_3Al_xC_2T_y$, with different magnifications were provided to give more information on the morphologies and particle sizes of Pt NPs.



Figure s5. TEM images of $Pt_{0.5}/Ti_3AI_xC_2T_y$ catalysts



Figure s6. TEM images of $Pt_{1.0}/Ti_3Al_xC_2T_y$ catalysts



Figure s7. TEM images of $Pt_{2.0}/Ti_3AI_xC_2T_y$ catalysts

Figure s8. TEM images of $Pt_{3.0}/Ti_3AI_xC_2T_y$ catalysts

The TEM images of **spent** $Pt_{2.0}/Ti_3Al_xC_2T_y$ showed that the Pt nanostructures were mostly nanoparticles preapred by the new reduction method.

Figure s9. TEM images of spent $Pt_{2.0}/Ti_3AI_xC_2T_y$

The TEM images of **spent 3%Pt/Ti₃Al_xC₂T**_y showed that the Pt nanostructures were mostly nanoclusters preapred by the chemical reduction (CR) method.

Figure s10. TEM images of spent 3%Pt/Ti₃Al_xC₂T_y