Supplementary Information (SI) for ChemComm.

SUPPORTING INFORMATION

Tuneable Electronic Control of Noble Metal Catalysis by Polymeric Supports

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SECTION 1. EXPERIMENTAL PART

S1.1. Materials

All the AR-grade chemical reagents were purchased from Merck and Sigma-Aldrich and used as received without further purification.

S1.2. Synthetic methods

Emeraldine salt

For the preparation of the emeraldine salt (ES) form of polyaniline (PA), 1.8 g ammonium persulfate were dissolved in 100 mL 1M HCl solution. In another reaction vessel, 100 ml 1M HCl solution were mixed with 3 mL aniline monomer and stirred until a homogeneous solution was obtained. After that, we added dropwise the ammonium persulfate solution into the aniline solution without stirring. The solution will turn slowly to black (dark greenish-black). The synthesis time was 5 hours without stirring at room temperature.

The resulting emeraldine salt was filtered (the colour of the filtrate was purple) and washed with 5 mM HCl solution until the filtrate turned into colourless. Finally, the product was refrigerated with liquid nitrogen and dried under vacuum for 48 hours. The colour of the product is dark greenish-black.

Emerladine base

For the preparation of the emeraldine base (EB), we put 300 mg emeraldine salt into a sodium hydroxide solution (c = 1M) and stirred for 24 hours at room temperature. The resulting emeraldine base was filtered and washed with distilled water 10 times. Finally, the product was refrigerated with liquid nitrogen and dried under vacuum for 48 hours. The colour of the product is dark blue-black.

Leucoemerladine base

For the preparation of leucoemeraldine base, emeraldine base of 150 mg was added into 15 ml hydrazine hydrate solution (60%) and stirred under a nitrogen atmosphere for 48 hours at room temperature. The resulting leucoemeraldine base was filtered and washed with distilled water 10 times. Finally, the product was refrigerated with liquid nitrogen and dried under vacuum for 48 hours. The colour of the product is dark purplish-blue.

Leucoemerladine salt

For the preparation of the leucoemeraldine salt, emeraldine salt of 150 mg were added into 15 ml hydrazine hydrate solution (60%, Sigma Aldrich) and stirred under a nitrogen atmosphere for 48 hours at room temperature. The resulting leucoemeraldine salt was filtered and washed with distilled water a few times. Finally, it was refrigerated with liquid nitrogen and dried under vacuum for 36-48 hours. The colour of the product is light purplish-blue.

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Synthesis of 2 nm Pt nanoparticles

0.1 g of NaOH was dissolved in 5 mL of ethylene glycol and the solution was added to another 5 mL of ethylene glycol solution containing 0.08 g of $H_2PtCl_6 \cdot \times H_2O$ (~0.2 mmol). The mixture was heated to 160 °C in an oil bath and held at that temperature for 2 h under an argon atmosphere. The resulting nanoparticles were precipitated with 2 mL of 2M HCl solution and re-dispersed in ethanol (10 mL) with 0.1 g of polyvinylpyrrolidone (PVP; Mw = 55,000). The nanoparticles were repeatedly washed by precipitating with hexane, centrifuging, and re-dispersing in ethanol before use. The concentration of the Pt nanoparticles in the solution was determined by ICP-MS techniques.

Loading of Pt nanoparticles on the polyaniline (PA) supports

a, Loading protocol: We have reported the protocol elsewhere previously.[1] Briefly, an ethanolic suspension of the Pt nanoparticles and the target support were mixed together in ethanol and sonicated in an ultrasonic bath (40 kHz, 80 W) for 3 h. The amount of the nanoparticle suspension and the support was set to achieve 2 wt% dispersion of the nanoparticles on the support. The supported nanoparticles were collected by centrifugation. The product was then washed with ethanol three times and dried at 80 °C overnight.

b, PVP removal process: The removal of the capping agent (PVP) was necessary to activate the anchored platinum sites on PA surfaces. For this purpose, a post-synthetic plasma treatment was used. The plasma treatment – published previously[2] – was performed in a low-pressure system (40 kHz, Femto B unit, Diener electronic). The ambient-air plasma was produced in a vacuum. For the evaluation of the treatment, the sample was characterized at different time intervals to measure its mass and the Raman intensities.

Synthesis of Pt–PANI composites with sub-nanometer size of platinum nanoparticles

DMF solution (1 mL) was added to the reaction flask containing emeraldine salt of PANI (200 mg) and dispersed uniformly, and recorded it as solution A. $PtCl_2$ (10 mg) were added to a reaction flask containing hydrochloric acid (1 M, 0.5 mL) and DMF solution (0.5 mL), dissolved by ultrasonication, and recorded as solution B. Then solution B was added to solution A, mixed well by ultrasonication for 20 minutes, and allowed to stand for 4 hours to react. The final product (Pt^{US}–LS) was isolated by centrifugation, washed with deionized water and ethanol, vacuum dried at 60 °C, and collected for use.

S1.3. Characterisation methods

For specific surface area study, BET (Brunauer-Emmett-Teller) N_2-sorption experiments were performed, using a NOVA3000 (Quantachrome) instrument. Prior to the measurements, the solids were degassed with N_2 at 100 °C for 5 h under vacuum to clean the surface from the adsorbents. The measurements were carried out at the temperature of liquid N_2. The actual platinum loadings on the supports of the carriers were determined with an Agilent 7900 ICP-MS (inductively coupled plasma–mass spectrometry) (Agilent Technologies) device. For the quantitative analysis, ICP multielement standard solution IV (CertiPUR) was used. Prior to the measurements, an accurately measured amount (a few milligrams) of the solids was dissolved in 5 mL of cc. HCl. After dissolution, the samples were diluted to 100 mL with distilled water and filtered. The morphological features of the prepared catalysts were examined with a FEI TECNAI G2 20 X-Twin high-resolution transmission electron microscope operating at an accelerating voltage of 200 kV. The samples were drop casted on carbon film mesh copper grids from ethanol suspensions.

The Raman spectra were recorded with a Bruker Senterra II Raman microscope. Spectra were collected by using a light source of 532 nm wavelength with 12.5 mW laser power. Final data were obtained by averaging 32 spectra with an exposition time of 4 s. The active surface specimens of the catalyst candidates were identified by X-ray photoelectron spectroscopy (XPS). XP spectra were recorded by a SPECS instrument coupled with a PHOIBOS 150 MCD 9 hemispherical analyser, under a chamber pressure of 10^{-9}–10^{-10} mbar range. The analyser was applied in a fixed analyser transmission (FAT) mode with 20 eV pass energy. The Al Kα radiation (hν = 1486.6 eV) of a dual anode X-ray gun was the excitation source. The gun was run at 210 W power (14 kV, 15 mA). The binding energy scale was adjusted by considering the position of the main C 1s component to 284.5 eV in all cases as can be seen below (Fig. P1). For evaluating the obtained data, commercial (CasaXPS, Origin) software packages were applied.

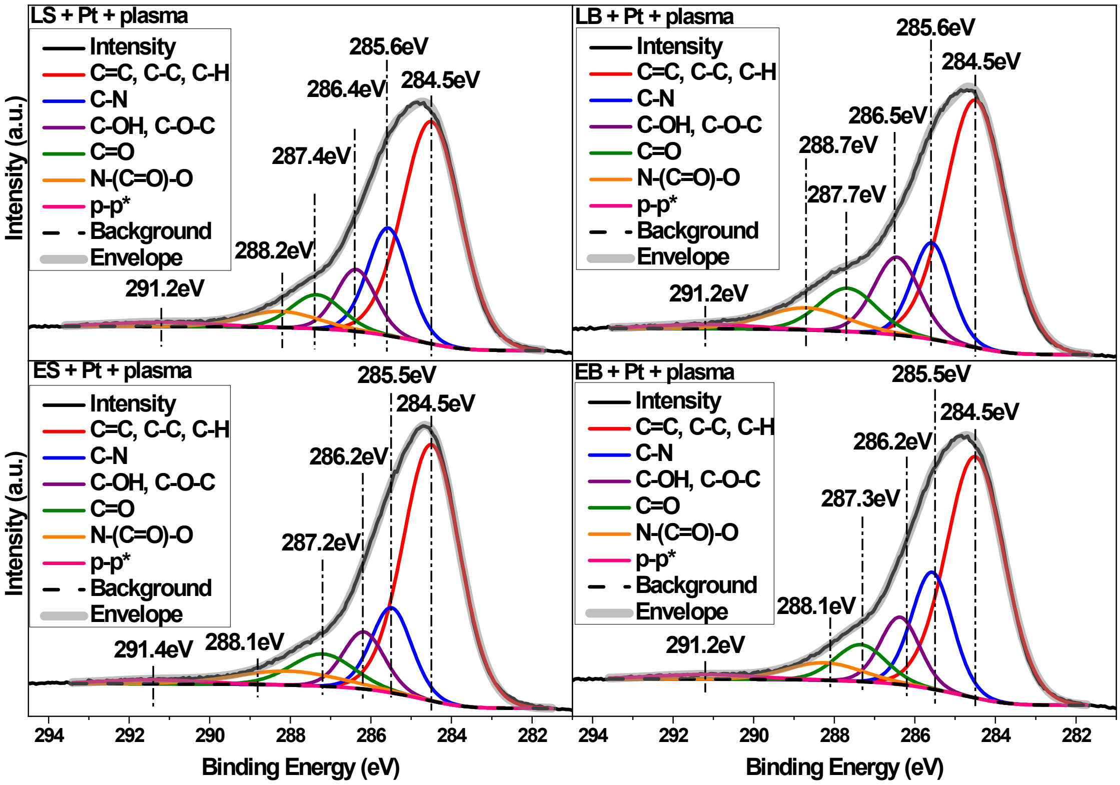

Figure P1. Carbon 1s energy region of the XP spectra of supported platinum catalysts.

Infrared spectroscopy measurements (In-situ Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS)) were carried out with an ‘Agilent Cary-670′ FTIR spectrometer equipped with ‘Harrick Praying Mantis’ diffuse reflectance attachment. The sample holder had two BaF_2 windows in the infrared beam path. The spectrometer was purged with dry nitrogen. The spectrum of the catalysts was used as background. At room temperature, the mixture of CO:N_2 (molar ratio of 1:4) was introduced into the DRIFTS cell and the samples were kept under this conditions for 2 hours. The tubes were externally heated to avoid condensation. All spectra were recorded between 4000 and 800 cm^{-1} at a resolution of 2 cm^{-1}. Typically, 32 scans were registered. Due to the short optical path within the DRIFTS cell, the contribution of the reactant gases was negligibly small, and from gas phase products only the most intense features were observable.

S1.4. Catalytic measurements

a, General procedure for Suzuki-Miyaura cross-coupling reaction

Pt–PA (0.3 mol% Pt), aryl chloride (1.2 mmol), phenylboronic acid (1.2 mmol), Cs_2CO_3 (1.4 mmol) and methyl ethyl ketone (MEK):EtOH = 3:1 mixture (2 ml) were placed in a sealed vial and the mixture was stirred at 75 °C for 3 h under an inert atmosphere. The reaction mixture was diluted with EtOAc, centrifuged (5000 rpm t = 5 min) to remove the catalyst and concentrated under vacuum. The obtained crude product was analysed by a gas chromatograph (GC). The GC was a Hewlett-Packard 5890 Series II instrument equipped with 50-m-long Agilent HP-1 column and a flame ionization detector. The temperature was increased in stages from 50 °C to 250 °C. The products were identified *via* using authentic samples. To determine the catalytic markers (product yield, product selectivity, reactant conversion), quantitative GC analysis of the crude products took place using an internal standard technique. N-decan was used as an internal standard in all cases.

b, General procedure for platinum-catalysed oxidation of 2-allylphenols to 2-methylbenzofurans or p-quinone

Pt–PA (0.3 mol% Pt), 2.0×10^{-3} mol of 2-allyphenol, 2.2×10^{-3} mol of H_2O_2, 3 mL of DMSO/water (1:1) were placed in a one-neck round bottom flask and the mixture was stirred at 50 °C for 2 h under an inert atmosphere. Then, the reaction mixture was diluted with EtOAc, centrifuged (5000 rpm t = 5 min) to remove the catalyst and concentrated under vacuum. The obtained crude product was analysed by a gas chromatograph using the same method as it was described above.

c, Recycling tests

After the oxidation reaction, the Pt–PA specimens were separated from the reaction mixture by centrifugation, washed thoroughly with water and alcohols and then reused as catalysts in subsequent cycles under the optimized reaction conditions. The structural features of the catalysts used were monitored by Raman spectroscopy measurements.

d, Hot filtration tests

The hot filtration experiments were performed as follows. After stirring the reaction mixture of the 2-allylphenol oxidation for 1 hour under the optimized reaction conditions, the catalyst was centrifuged out from the reaction mixture, and the flask was placed back to stir another 1 hour. Kinetic curve was determined for this hot filtrated sample in comparison to the normal one treated parallel with above-described.

SECTION 2. SUPPORTING RESULTS

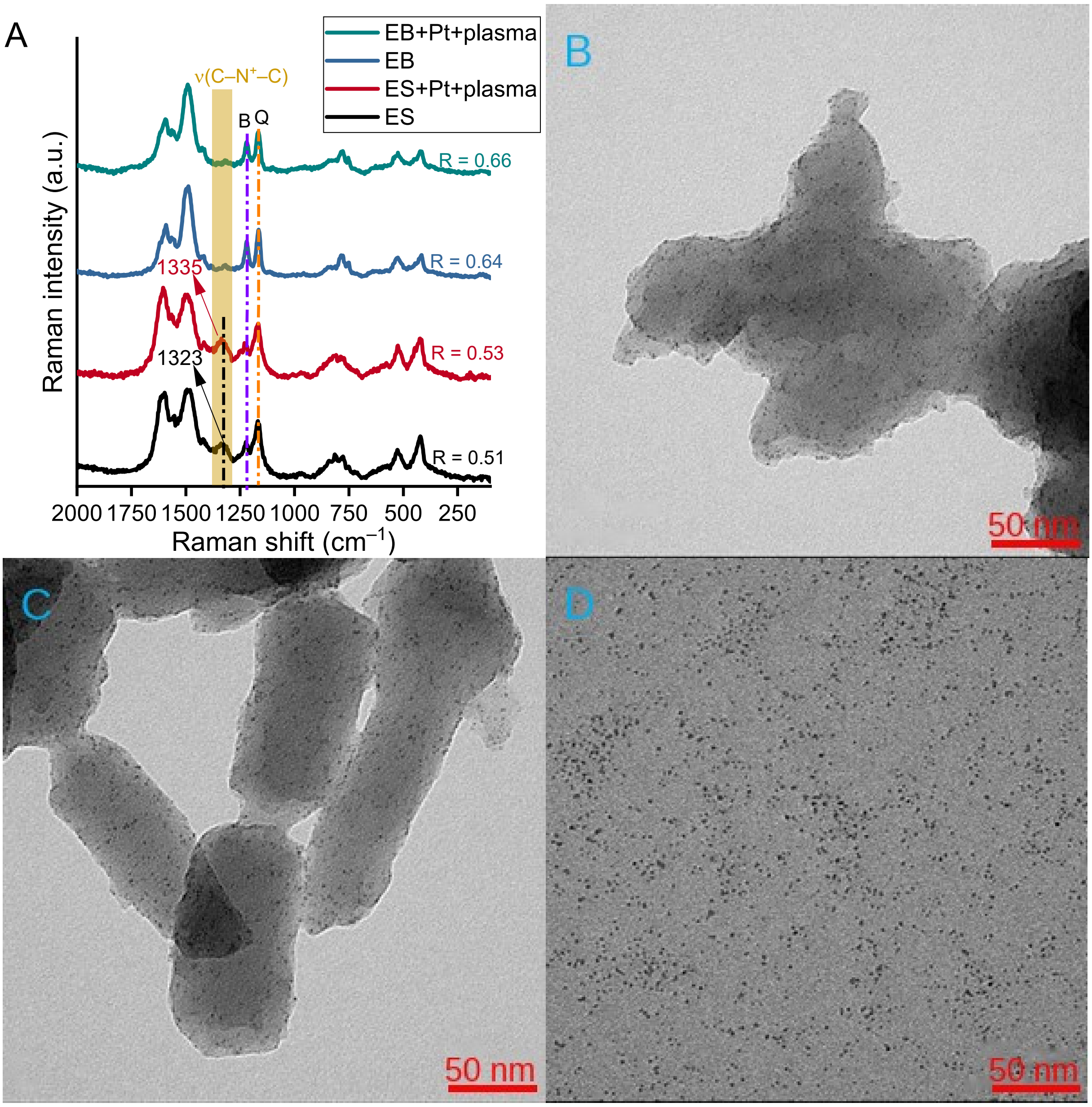

Figure S1. Raman spectra (A) of the pure emeraldine supports and their platinum modified counterparts. TEM images of (B) LS+Pt, (C) LS+Pt after plasma treatment and (D) Pt(CA).

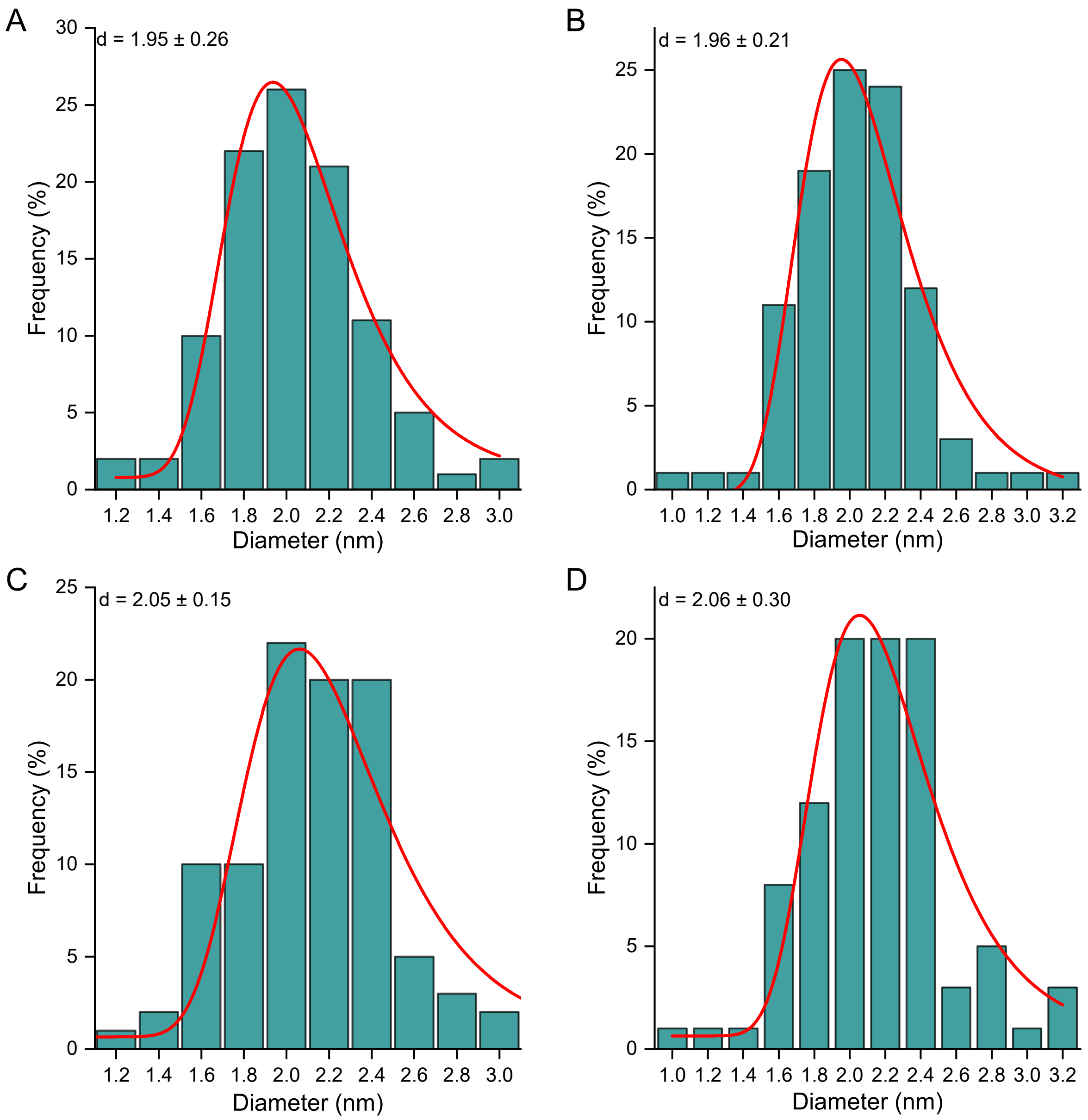

Figure S2. Size distribution of platinum nanoparticles on different forms of PA supports: (A) LS, (B) EB, (C) LB and (D) ES determined by TEM.

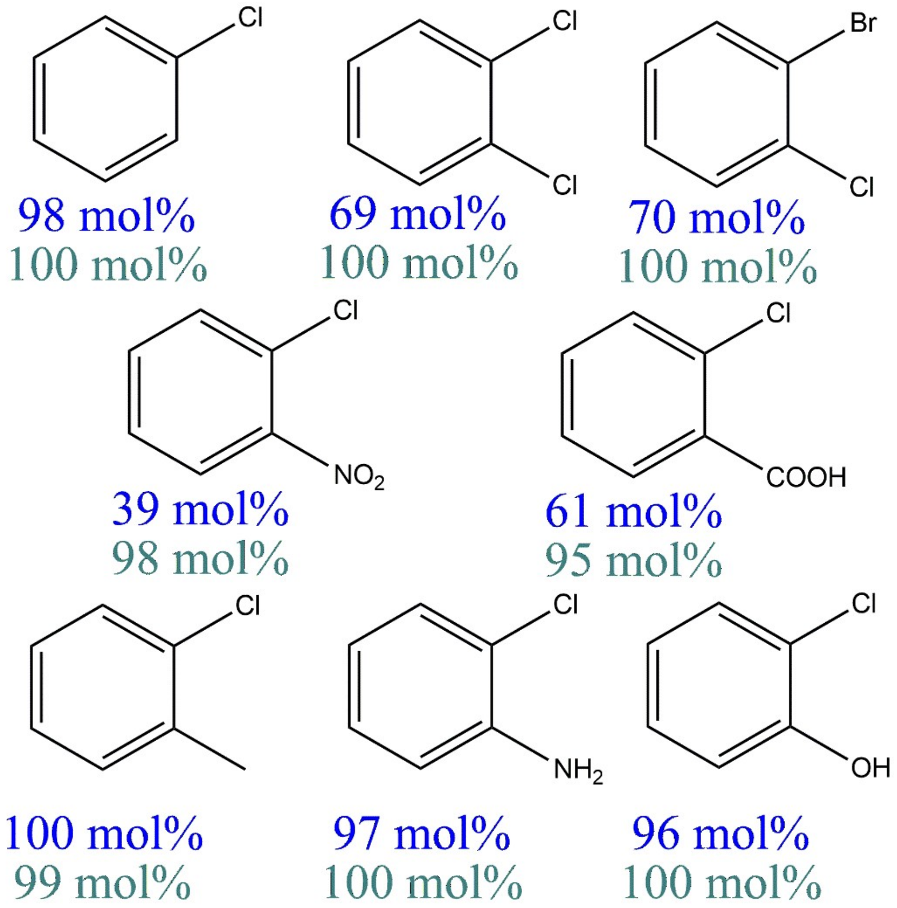

Figure S3. Substrate scope of Pt–PA composites catalysed Suzuki-Miyaura coupling of phenylboronic acid and chloro-benzene. Substrate scope was completed in the presence of Pt–ES catalyst. Blue numbers are for product yields, green numbers are for selectivities Reaction conditions: 1.0×10^{-3} mol of chlorobenzene or its derivative, 1.2×10^{-3} mol of phenylboronic acid, 1.2×10^{-3} mol of Bu_4NCl, 1.4×10^{-3} mol of Cs_2CO_3, 0.3 mol % catalyst, 2.0 mL of MEK:EtOH = 3:1. T = 75 °C, and t = 3 h.

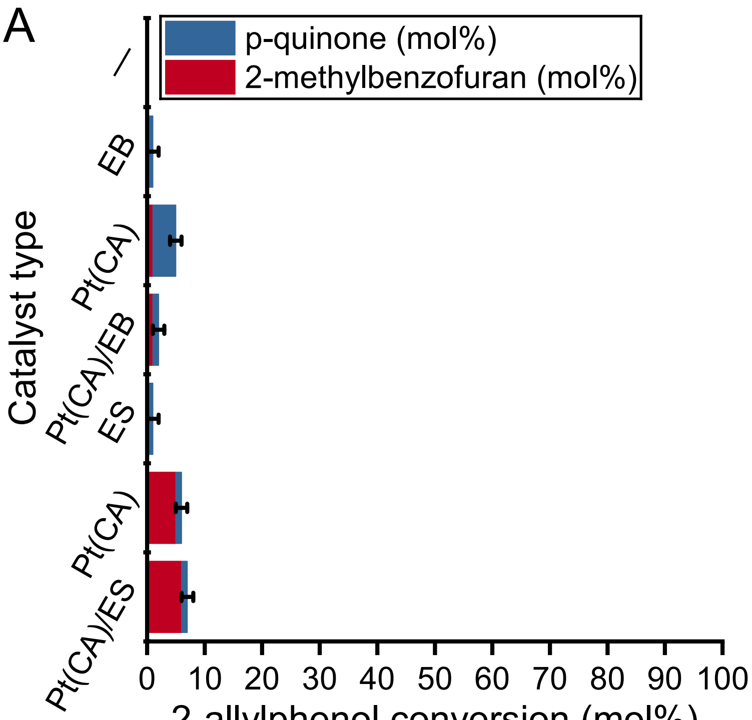

Figure S4. Catalytic performance of different Pt-containing catalyst for oxidation of 2-allylphenol under optimized reaction conditions. Optimized reaction conditions for catalyst with salt-type carriers: 2.0 × 10^{-3} mol of 2-allyphenol, 2.2 × 10^{-3} mol of H_2O_2, 3 mL of DMSO/water (1:1), T = 50°C, t = 120 minutes, under a nitrogen atmosphere.

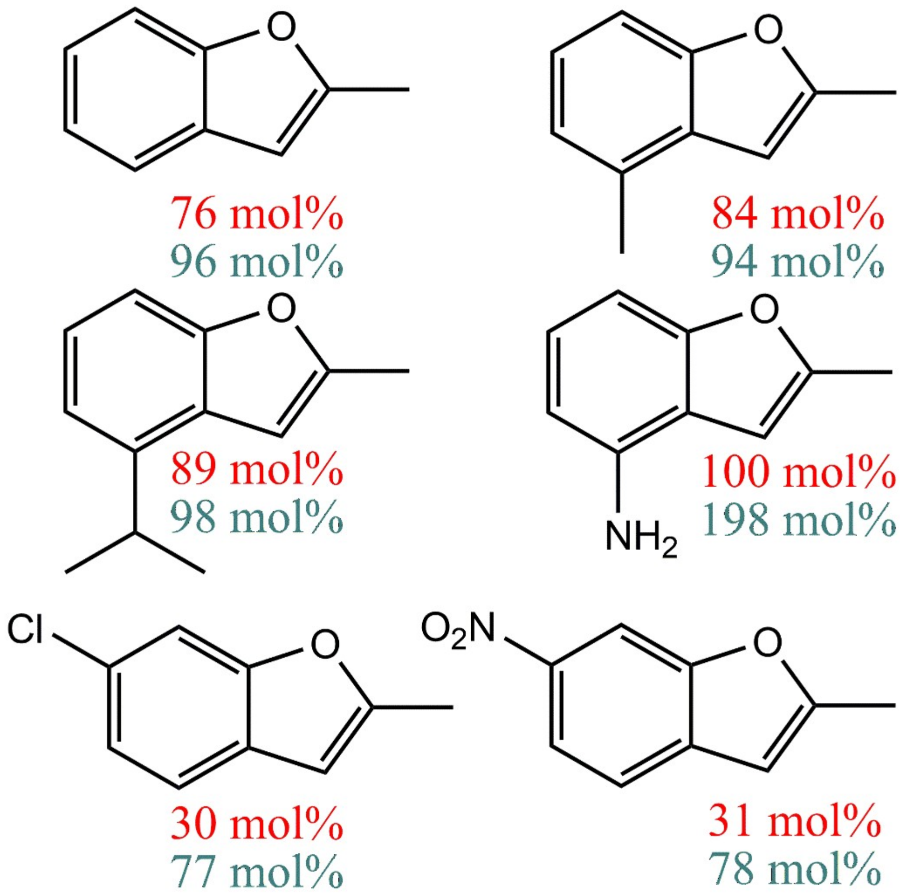

Figure S5. Substrate scope reactions for selective oxidation of 2-allylphenol derivatives over a Pt–ES composite under optimized reaction conditions. Optimized reaction conditions for catalyst with salt-type carriers: 2.0×10^{-3} mol of 2-allyphenol, 2.2×10^{-3} mol of H_2O_2, 3 mL of DMSO/water (1:1), T = 50°C, t = 120 minutes, under a nitrogen atmosphere. (Red numbers: benzofuran yields, green numbers: benzofuran selectivities.)

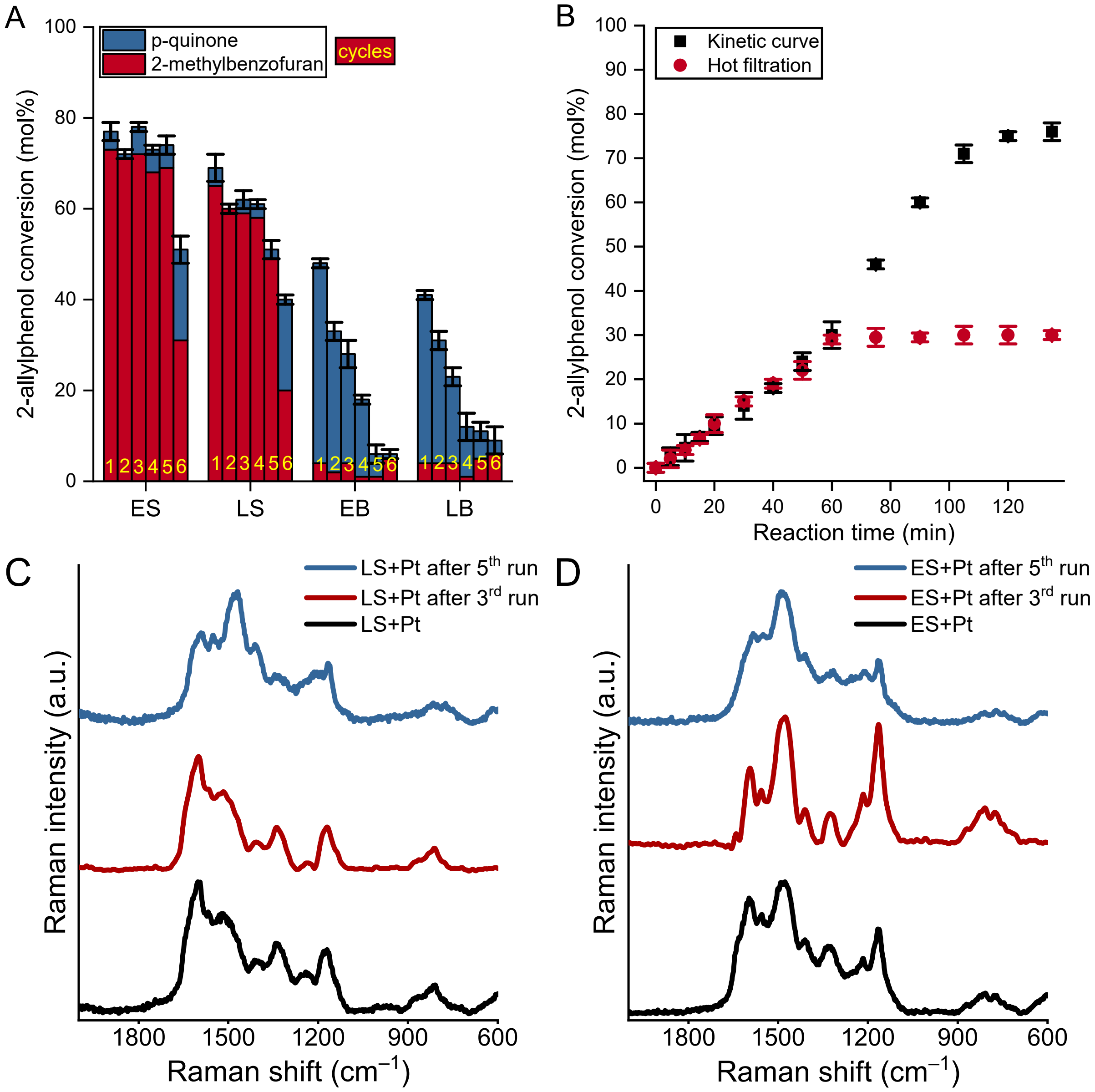

Figure S6. Recycling tests of platinum catalysts supported on different types of Pa in 2-allyphenol oxidative transformations (A). Hot filtration test of Pt–ES catalyst in 2-allyphenol oxidative transformation to 2-methylbenzofuran (B). Raman spectra (C,D) of the supported catalysts before and after their utilization in the third and fifth run of test reaction of oxidation of 2-allylphenol. Optimized reaction conditions for catalyst with salt-type carriers: 2.0×10^{-3} mol of 2-allyphenol, 2.2×10^{-3} mol of H_2O_2, 3 mL of DMSO/water (1:1), T = 50°C, t = 120 minutes, under a nitrogen atmosphere.

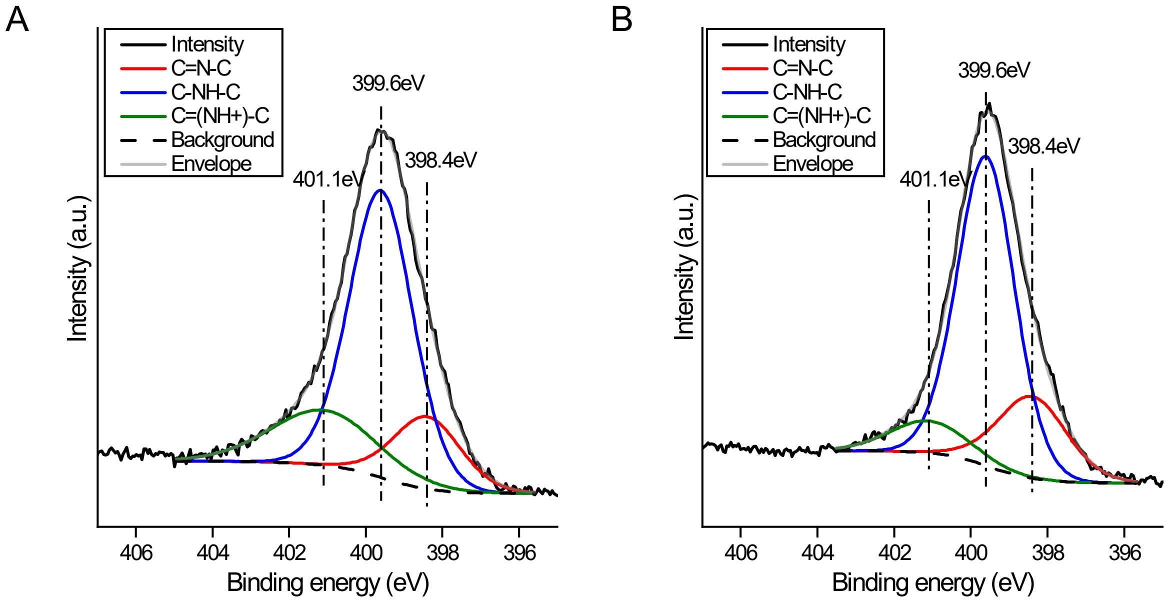

Figure S7. Nitrogen 1s energy region of the XP spectra of LS supported platinum catalyst (A) and LB supported platinum catalyst (B).

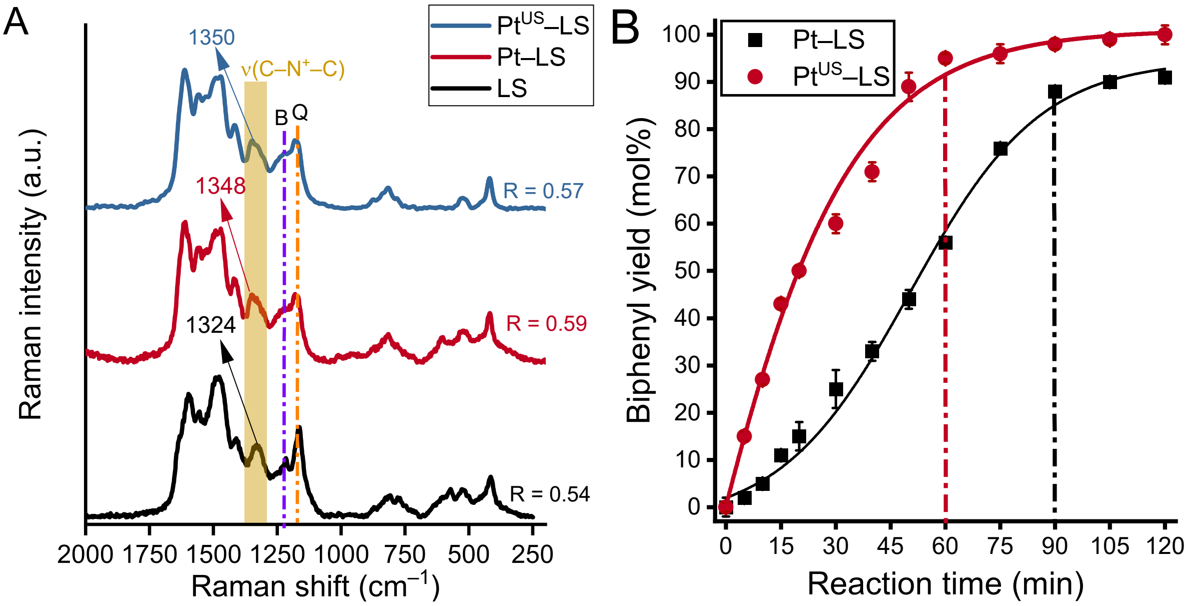

Figure S8. Raman spectrum of LS-based structure (A). Kinetic curves (B) of Pt–LS composites catalysed Suzuki-Miyaura coupling of phenylboronic acid and chloro-benzene. (Blue numbers are for product yields, green numbers are for selectivities Reaction conditions: 1.0×10^{-3} mol of chlorobenzene, 1.2×10^{-3} mol of phenylboronic acid, 1.2×10^{-3} mol of Bu_4NCl, 1.4×10^{-3} mol of Cs_2CO_3, 0.3 mol % catalyst, 2.0 mL of MEK:EtOH = 3:1. T = 75 °C, and t = 2 h.

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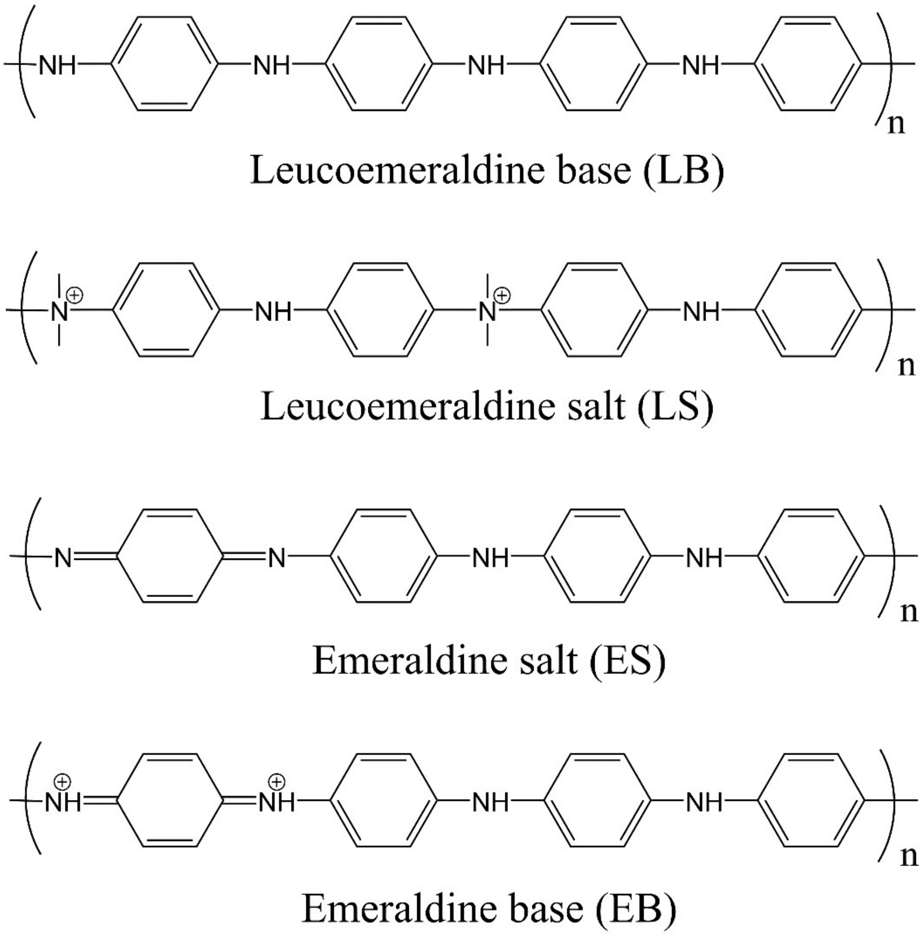

Scheme S1. Different forms (oxidation states) of polyaniline.

SUPPORTING INFORMATION

Table S1. Optimisation of the reaction parameters for benzofuran synthesis from 2-allylphenol over Pt–PA type catalysts. (Pt–ES was used for the optimisation procedure.) Optimum parameters are in the red box.

Entry	**Pt(mol%)**	**H_2O_2 (eq.)**	**solvent**	**T (°C)**	**Reaction time (min)**	**Yield (mol%)***	**Yield (mol%)****
1	5	3.0	DMF	75	180	37	15
2	5	3.0	γ-Valerolactone	75	180	11	7
3	5	3.0	N-Butylpyrrolidinone	75	180	27	10
4	5	3.0	DMSO	75	180	50	12
5	5	3.0	DMSO/EtOAc	75	180	61	10
6	5	3.0	**DMSO/water**	75	180	*65*	*4*
7	5	3.0	water	75	180	4	5
8	5	3.0	DMSO/water	75	**120**	*82*	*3*
9	5	3.0	DMSO/water	75	60	33	1
10	5	3.0	DMSO/water	**50**	120	*79*	*3*
11	5	3.0	DMSO/water	25	120	28	7
12	5	3.0	DMSO/water	100	120	68	20
13	5	2.0	DMSO/water	50	120	80	1
14	5	**1.1**	DMSO/water	50	120	*85*	*1*
15	**2.5**	1.1	DMSO/water	50	120	**73**	**4**
16	1.0	1.1	DMSO/water	50	120	35	5

*2-methylbenzofuran yield, ** p-quinone yield

Table S2. Optimisation of the reaction parameters for quinone synthesis from 2-allylphenol over Pt–PA type catalysts. (Pt–EB was used for the optimisation procedure.) Optimum parameters are in the red box.

Entry	**Pt(mol%)**	**H_2O_2 (eq.)**	**solvent**	**T (°C)**	**Reaction time (min)**	**Yield (mol%)***	**Yield (mol%)****
1	5	3.0	DMF	75	180	1	18
2	5	3.0	γ-Valerolactone	75	180	3	0
3	5	3.0	N-Butylpyrrolidinone	75	180	1	0
4	5	3.0	DMSO	75	180	3	13
5	5	3.0	**DMSO/CH_3CN**	**75**	**180**	*6*	*50*
6	5	3.0	CH_3CN	75	180	9	55
7	5	3.0	DMSO/water	75	180	4	15
8	5	3.0	DMSO/CH_3CN	75	120	8	29
9	5	3.0	DMSO/CH_3CN	75	60	4	10
10	5	3.0	DMSO/CH_3CN	50	120	2	30
11	5	3.0	DMSO/CH_3CN	25	120	0	4
12	5	3.0	DMSO/CH_3CN	100	120	6	58
13	5	2.0	DMSO/CH_3CN	50	120	5	47
14	5	**1.1**	DMSO/CH_3CN	50	120	*5*	*45*
15	**2.5**	1.1	DMSO/CH_3CN	50	120	**4**	**44**
16	1.0	1.1	DMSO/CH_3CN	50	120	7	26

*2-methylbenzofuran yield, ** p-quinone yield

Table S3. Leaching of platinum particles (%) through recycling tests determined by ICP-MS:

Composites	**Number of run**					
	1	2	3	4	5	6
	Leaching of platinum (%)					
Pt–ES	0.1	0.05	0.02	0.1	0.08	0.1
Pt–LS	0.2	0.3	0.05	0.07	0.08	0.07
Pt–EB	29	34	39	44	71	72
Pt–LB	24	30	36	59	61	63

Table S4. Catalytic performances of Pt- and Pd nanoparticle immobilized supports for Suzuki-Miyaura cross coupling reactions with different substrates. (Rate is calculated for total catalyst loading and total reaction time for a comparison:

rate = yield (mol%) / [catalyst loading (mol%) × reaction time (h)]

Metal	Catalyst	Yield (%) (rate, h^{-1})	Model reaction	Reaction conditions	Supporting material (reusability)	Ref
$Pt^{\delta+}$	Pt–ES	100 (167)	chlorobenzene + phenylboronic acid	MEK:EtOH = 3:1; Cs_2CO_3; 75 °C; 0.3 mol% catalyst; 2 h	Emeraldine salt form of PANI (5 times)	This work
$Pt^{\delta+}$	Pt–LS	91 (152)	chlorobenzene + phenylboronic acid	MEK:EtOH = 3:1; Cs_2CO_3; 75 °C; 0.3 mol% catalyst; 2 h	Leucoemeraldine salt form of PANI (5 times)	
Pt(0)	Pt@HPP	91 (101)	chlorobenzene + phenylboronic acid	DMF:H_2O = 4:1; Cs_2CO_3; 75 °C; 0.3 mol% catalyst; 3 h	HPP (reusable 10 times)	2
Pt(0)	Pt@MCF-17	71 (79)	chlorobenzene + phenylboronic acid	DMF:H_2O = 4:1; Cs_2CO_3; 75 °C; 0.3 mol% catalyst; 3 h	MCF-17 (reusable 10 times)	
Pt(0)	Pt@SBA-15	24 (27)	chlorobenzene + phenylboronic acid	DMF:H_2O = 4:1; Cs_2CO_3; 100 °C; 0.3 mol% catalyst; 3 h	SBA-15 (decreasing yields)	
Pd(0)	Pd/HCP	98 (12)	4'-chloroacetophenone + phenylboronic acid	EtOH:H_2O = 1.5:1; Na_2CO_3; 100 °C; 2 mol% catalyst; 4 h	HCP (reusable 2 times)	3
Pd(0)	Pd/linear polystyrene	6 (0.2)	chlorobenzene + phenylboronic acid	H_2O; KOH; 80 °C; 1.5 mol% catalyst; 20 h	Linear polystyrene (no data)	4
Pd(0)	Pd/linear polystyrene	99 (66)c	4'-chloroacetophenone + phenylboronic acid	H2O; KOH; 80 °C; 1.5 mol% catalyst; 1 h	Linear polystyrene (reusable 2 times)	
Pd(II)	Pd/triazine-based mesoporous polymer	65 (4)	chlorobenzene + phenylboronic acid	DMF:H_2O = 1:5; K_2CO_3; 90 °C; 0.8 mol% catalyst; 22 h	Triazine-based mesoporous polymer (6 times)	5
Pd(0)	Pd/PPh_3-functionalized	99 (55)	chlorobenzene +	EtOH:H_2O = 1.5:1; K_3PO_4;	PPh_3-functionalize	6

	HCP		phenylboronic acid	80 °C; 0.6 mol% catalyst; 3 h	d HCP (no data)	
Pd(0)	Pd/dibenzylideneacetone-functionalized macroporous resin	48 (8)	chlorobenzene + phenylboronic acid	DMF:H_2O = 1:1; Na_2CO_3; 100 °C; 0.5 mol% catalyst; 12 h	Dibenzylideneacetone-functionalized macroporous resin (reusable 5 times)	7
Pd(II)	Pd/NH_2-functionalized graphene oxide	15 (0.6)	chlorobenzene + phenylboronic acid	EtOH:H_2O = 2:1; K_2CO_3; 60 °C; 1 mol% catalyst; 24 h	NH_2-functionalized graphene oxide (reusable 10 times)	8
Pd(0)	Pd/tritylaniline-based HCP	48 (0.5)	chlorobenzene + phenylboronic acid	H_2O; NaOH; 100 °C; 3.8 mol% catalyst; 24 h	Tritylaniline-based HCP (reusable 5 times)	9
Pd(0)	Pd/NHC-functionalized HCP	88 (115)	chlorobenzene + phenylboronic acid	H_2O; K_3PO_4; 80 °C; 0.8 mol% catalyst; 1 h	N-heterocarbene-functionalized HCP (reusable 5 times)	10
Pd(II)	Pd/pyridine-dendron-functionalized SBA-15	70 (27)	chlorobenzene + phenylboronic acid	EtOH:H_2O = 2:1; K_2CO_3; 50 °C; 0.3 mol% catalyst; 10 h	Pyridine-dendron-functionalized SBA-15 (reusable 7 times)	11
Pd(II)	Pd/mercaptopropyl-modified SBA-15	67 (1)	chlorobenzene + phenylboronic acid	H_2O; K_2CO_3; 100 °C; 2 mol% catalyst; 24 h	Mercaptopropyl-modified SBA-15 (reusable 5 times)	12
Pd(II)	Pd/mercaptopropyl-modified SBA-15	99 (2)c	4'-chloroacetophenone + phenylboronic acid	H_2O; K_2CO_3; 100 °C; 2 mol% catalyst; 24 h	Mercaptopropyl-modified SBA-15 (reusable 5 times)	
Pd(0)	Pd/PNIPAM/PMAA-modified SBA-15	19 (39)	chlorobenzene + phenylboronic acid	EtOH:H_2O = 1:4; K_2CO_3; RT; 0.03 mol% catalyst; 9 h	PNIPAM/PMAA-modified SBA-15 (reusable 5 times)	13
Pd(0)	MSN-APS-ALD-BZLM-Pd	98 (6682)	aryl bromide (X = Br, R = H) +	K_2CO_3; EtOH; 80 °C; 0.176 mol% Pd; 5 min	Mesoporous silica nanoparticle support with	14

			phenylboronic acid		APS/ALD/BZLM ligand environment (reusable 5 times)	
Pd(0)	NHC-Pd(II)@GO	98 (4900)	aryl bromide (X = Br, R = CN) + phenylboronic acid	K_2CO_3; H_2O:EtOH = 1:1; RT; 0.02 mol% Pd; 1 h	Graphene oxide immobilized NHC-Pd(II) complex (reusable 11 times)	15
Pd(0)	Pd-NHC-CPTes@SiO_2	98 (4900)	aryl iodide (X = I, R = H) + phenylboronic acid	K_2CO_3; iPrOH:H_2O = 1:1; 50 °C; 0.03 mol% Pd; 40 min	Silica-supported Pd-NHC complex (reusable 6 times)	16
Pd(0)	Pd@PHB-CS	99 (4400)	aryl bromide (X = Br, R = 4-CHO) + phenylboronic acid	K_2CO_3; EtOH:H_2O = 1:1; 50 °C; 0.045 mol% Pd; 0.5 h	Chitosan-modified poly(3-hydroxybutyrate) support (reusable 5 times)	17

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