

# Waste for Product—Synthesis and Electrocatalytic Properties of Palladium Nanopyramid Layer Enriched with PtNPs

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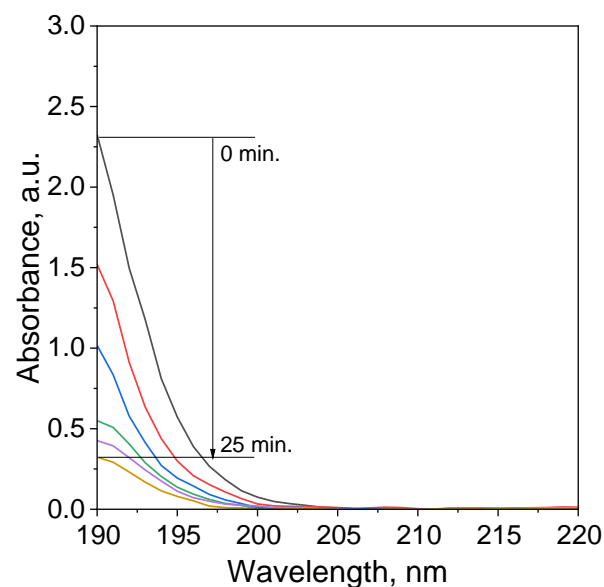
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## 1. UV-Vis spectra for solution containing chloride ions.



**Figure S1.** Spectra for ROY sample containing only chloride ions (ROY\_Cl) at different time of adsorption process. Conditions:  $m_{\text{ROY}} = 0.5 \text{ g}$ ,  $C_{0, \text{Cl}^-} \sim 3 \text{ mmol/dm}^3$ ,  $V = 50 \text{ mL}$ ,  $500 \text{ rpm}$ ,  $T = 20^\circ\text{C}$ .

## 2. Results from Raman analysis

Table S1. Deconvolution parameters.

ROY		Pt@ROY		Pd@ROY		Pd-Pt@ROY	
Position [cm <sup>-1</sup> ]	Intensity [a.u.]	Position [cm <sup>-1</sup> ]	Intensity [a.u.]	Position [cm <sup>-1</sup> ]	Intensity [a.u.]	Position [cm <sup>-1</sup> ]	Intensity [a.u.]
1171	0.23	1169	0.22	1169	0.24	1169	0.23
1300	0.44	1298	0.49	1302	0.40	1297	0.45
1343	1.58	1346	1.57	1345	1.55	1349	1.54
1560	0.43	1558	0.48	1559	0.42	1557	0.54
1604	1.24	1605	1.25	1606	1.26	1606	1.27

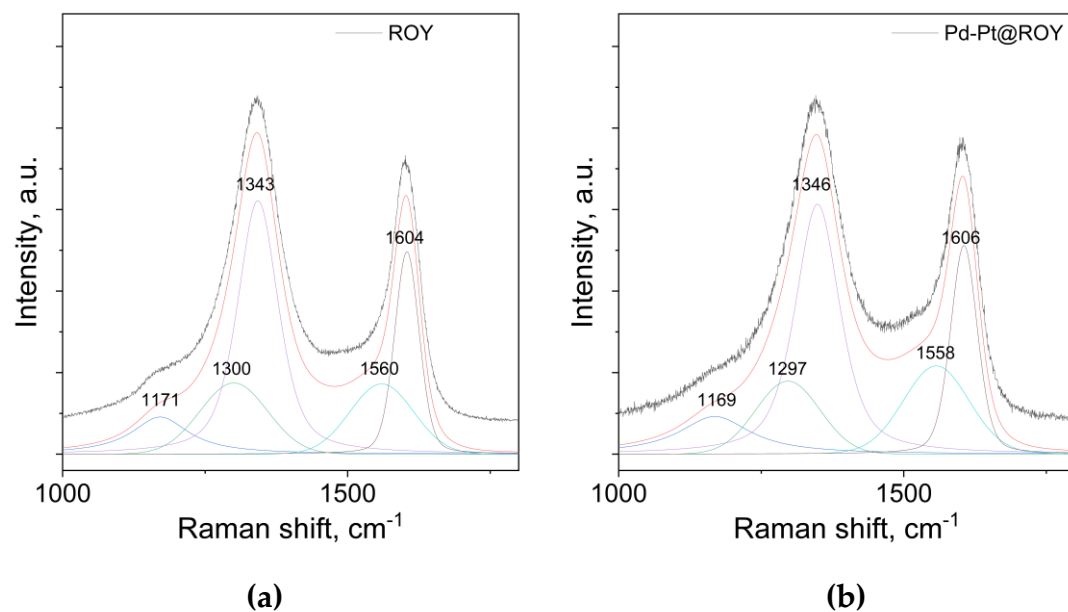


Figure S2. Deconvoluted Raman spectra of ROY (a) and Pd-Pt@ROY (b) samples.

### 3. Detailed results from IR analysis.

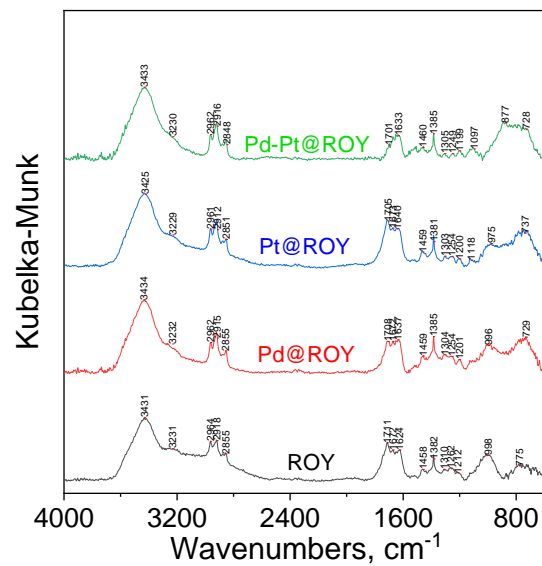
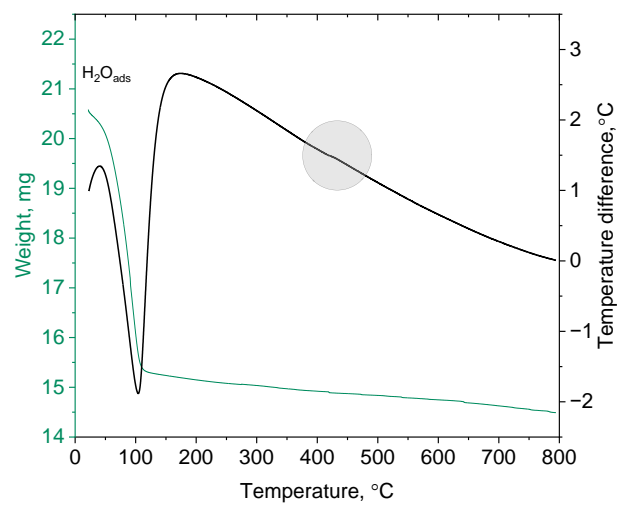
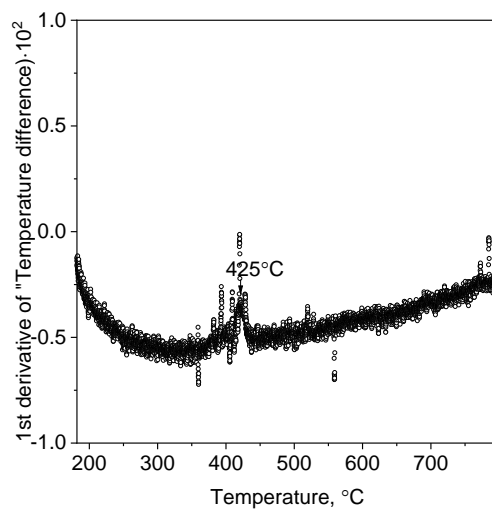


Figure S3. IR analysis performed for ROY, Pd@ROY, Pt@ROY and Pd-Pt@ROY samples with characteristic bands. .

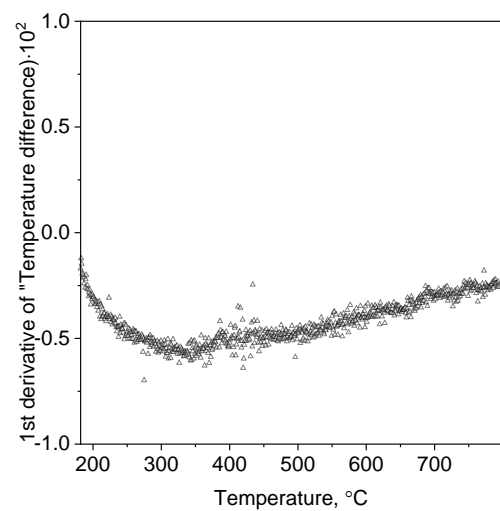
#### 4. TGA analysis



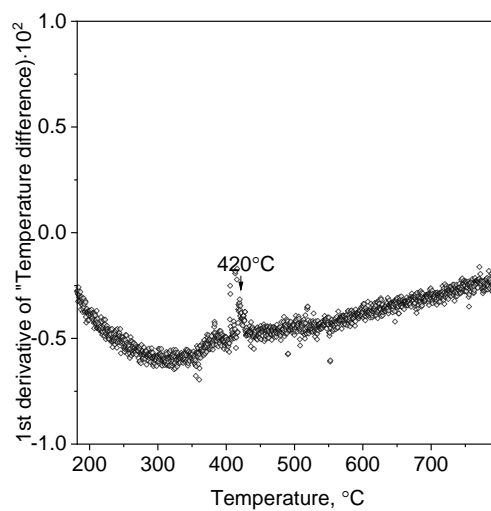
(a)



(b)



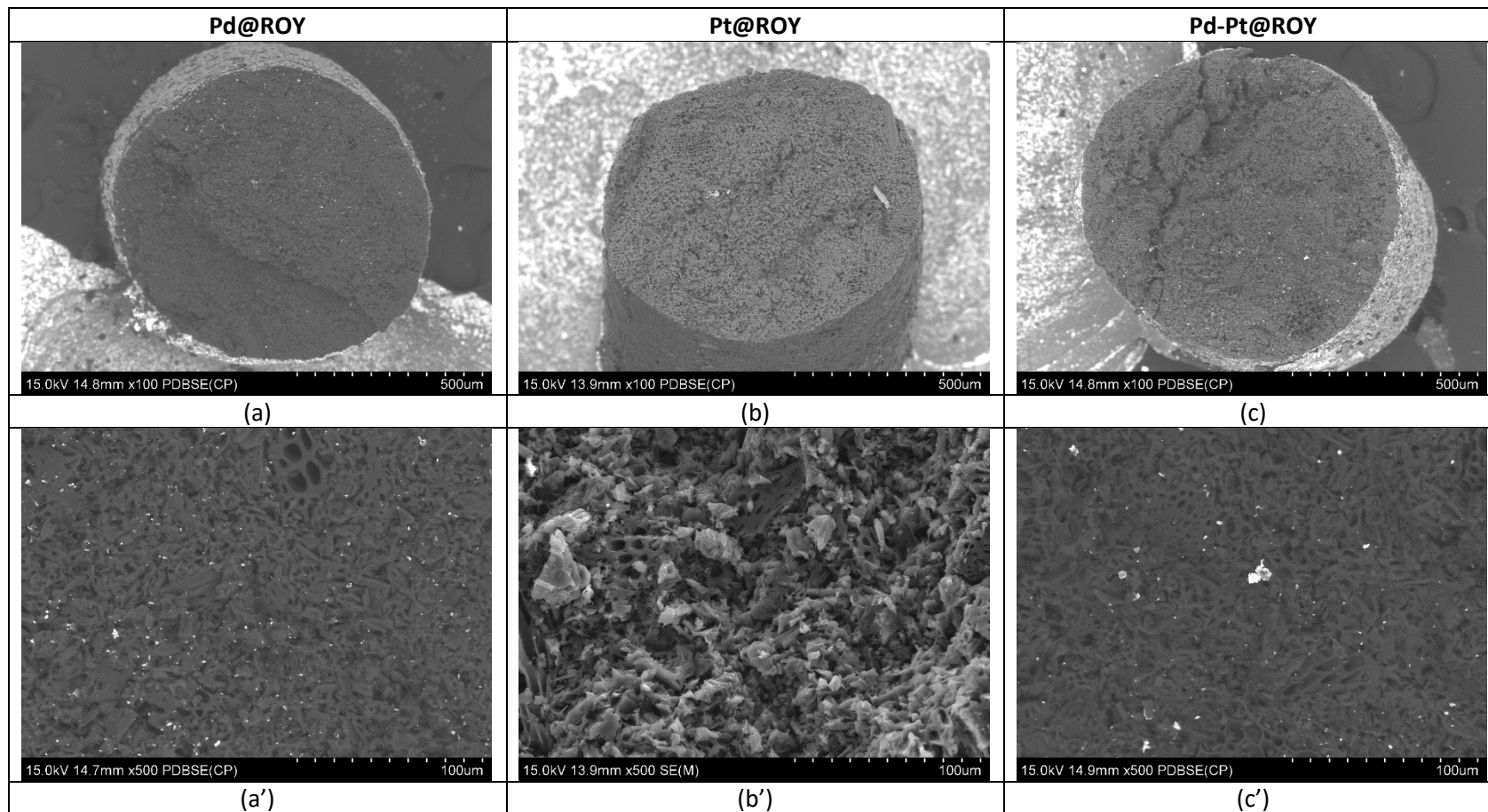
(c)



(d)

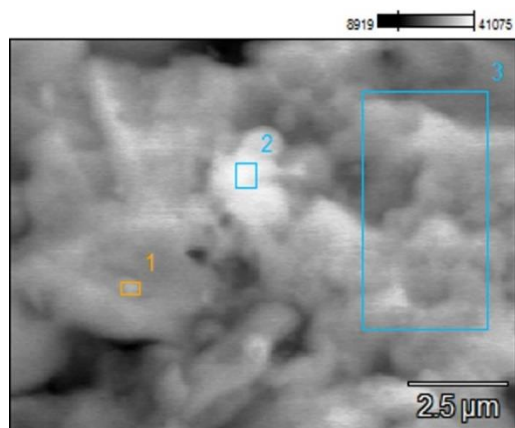
**Figure S4.** TGA analysis of Pd@ROY sample with marked interesting area (grey) (a); 1<sup>st</sup> derivative of temperature difference for Pd@ROY (b), Pt@ROY (c) and Pd-Pt@ROY (d).

## 5. Results from SEM/EDS analysis.

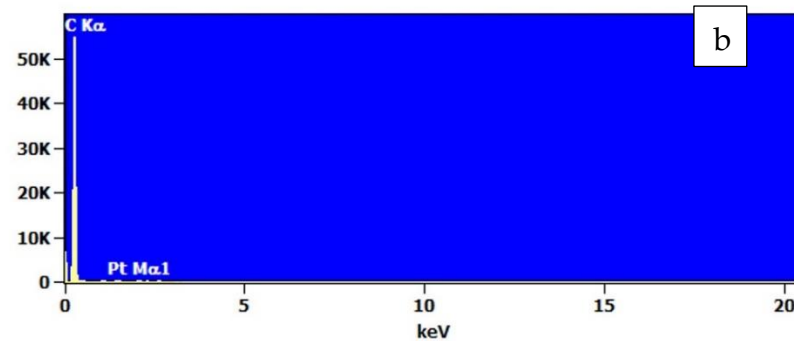


**Figure S5.** Cross section of the obtained materials at different magnifications. Samples: Pd@ROY (a, a'); Pt@ROY (b, b'); Pd-Pt@ROY (c, c').

a

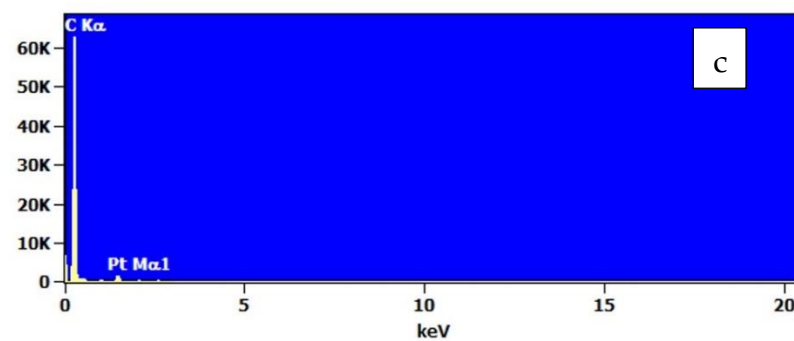


Full scale counts: 55052



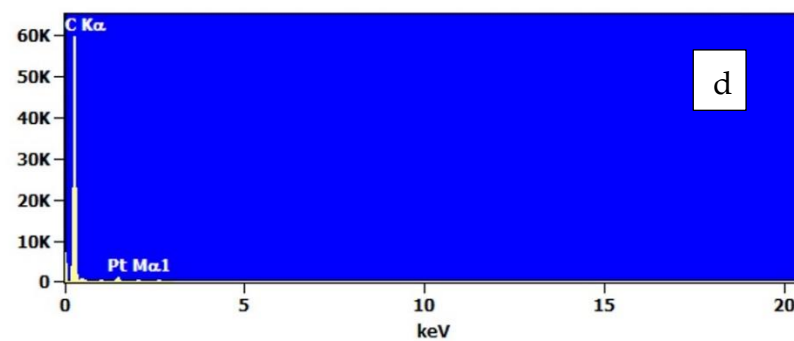
b

Full scale counts: 62978



c

Full scale counts: 59937

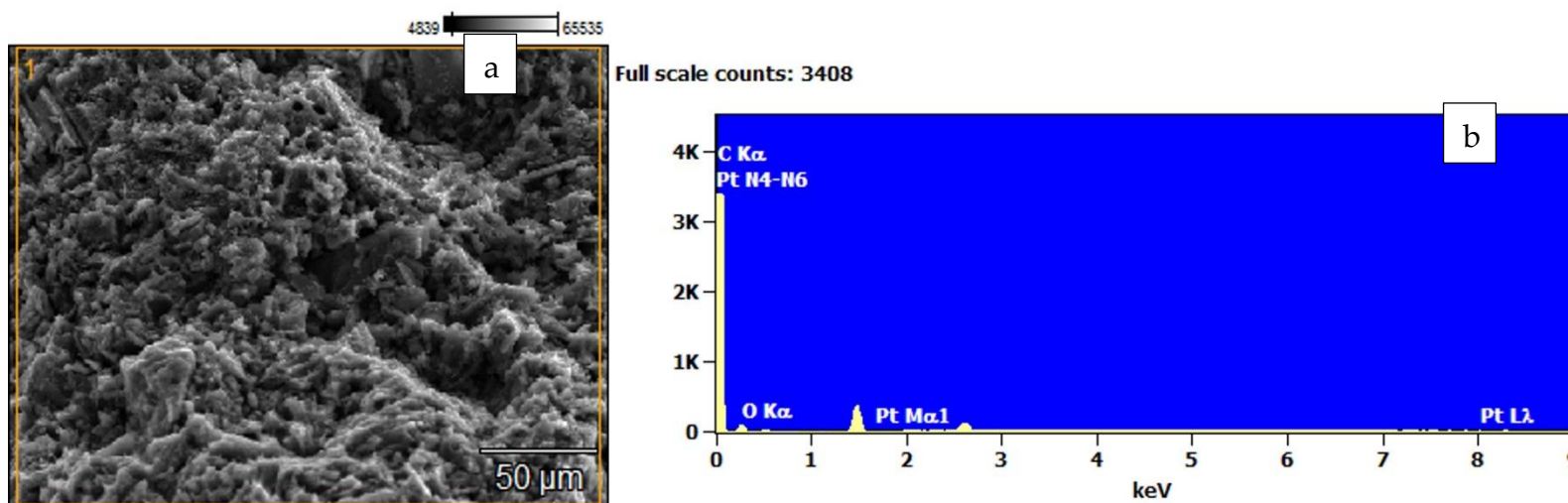


d

**Figure S6.** The SEM micrographs of Pt@ROY surface (a) and EDS analysis at selected area (1, 2, 3) (b, c, d), respectively.

**Table S2.** The EDS analysis of Pt@ROY surface at selected areas 1, 2,3.

Area	Element concentration [weight %]	
	C-K	Pt-M
surface 1	99.43	0.57
surface 2	99.61	0.39
surface 3	99.48	0.52
Area	Element concentration [atom%]	
	C-K	Pt-M
surface 1	99.96	0.04
surface 2	99.98	0.02
surface 3	99.97	0.03

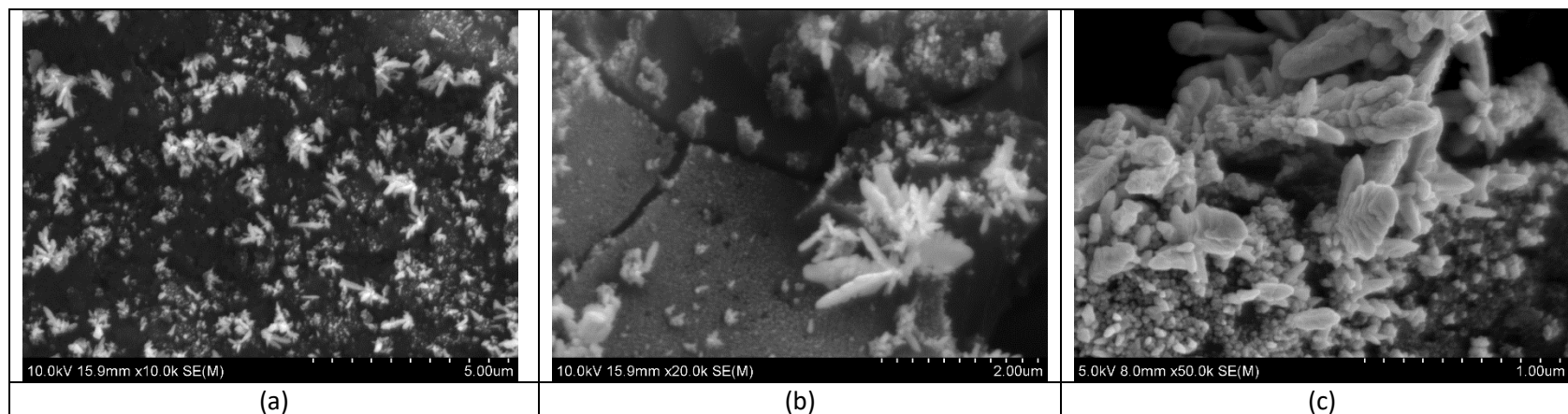


**Figure S7.** The SEM micrographs of Pt@ROY cross section (a) and EDS analysis at selected area (1) (b).

**Table S3.** The EDS analysis of Pt@ROY surface (a) at selected area 1.

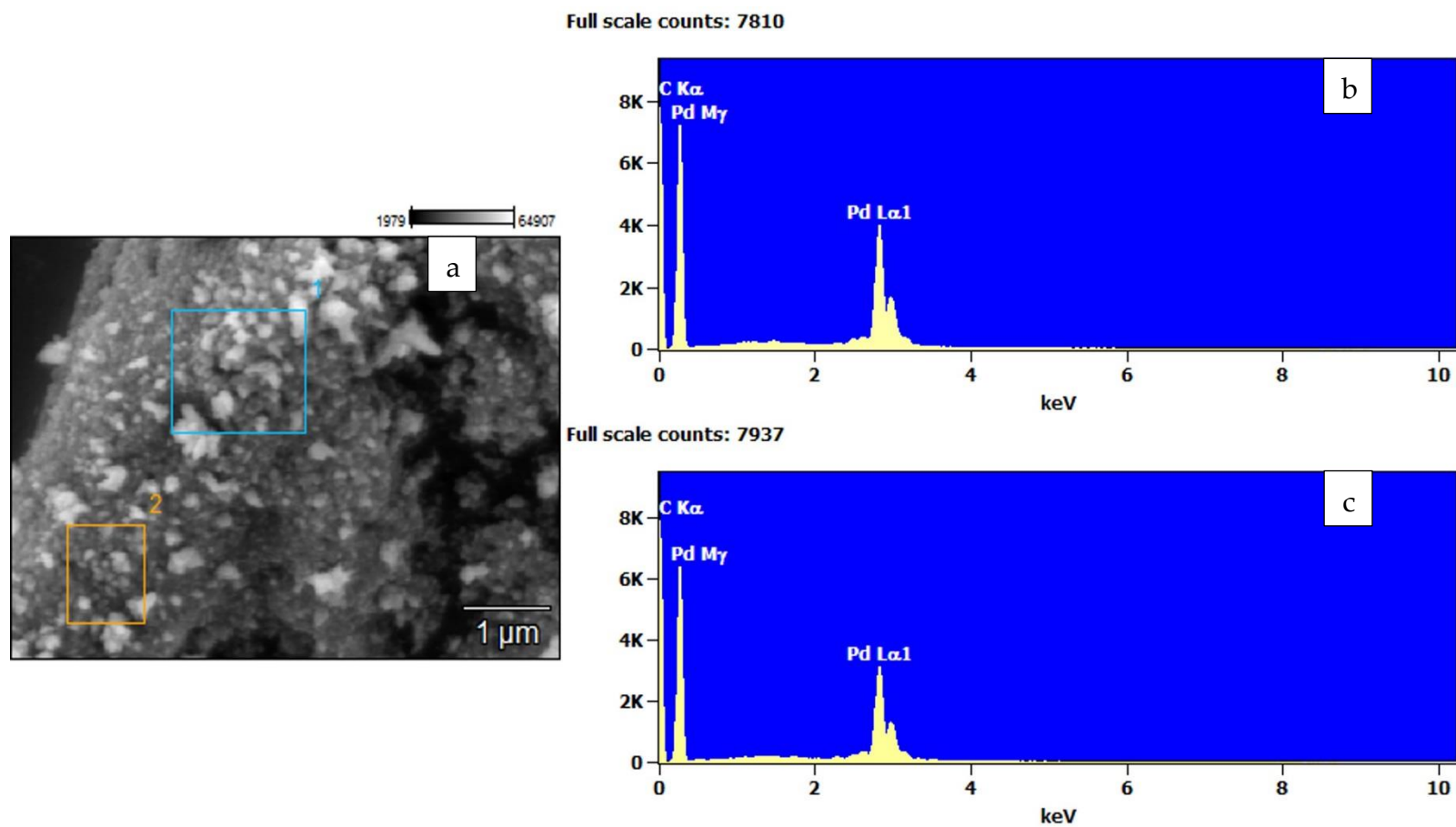
Area	Element concentration [weight %]
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	C-K	O-K	Pt-M
surface	55.65	33.38	10.96
Area	Element concentration [atom %]		
	C-K	O-K	Pt-M
surface	68.38	30.79	0.83



**Figure S8.** SEM analysis of Pd@ROY at different magnifications (a – c).



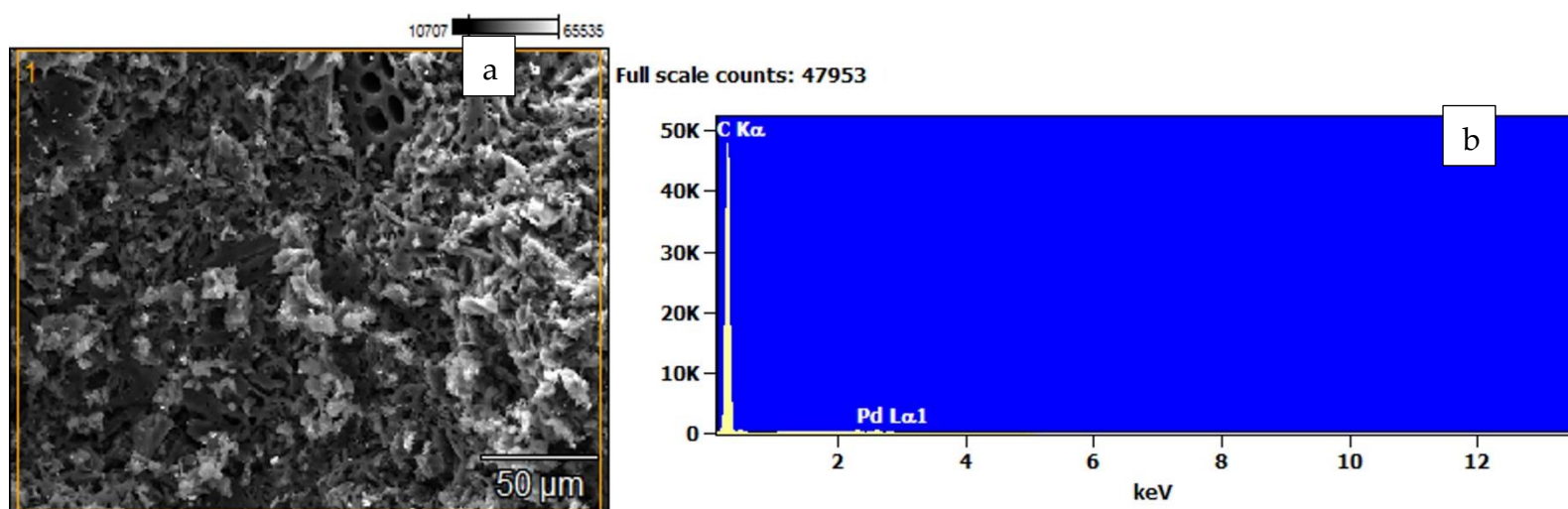


**Figure S9.** The SEM micrographs of Pd@ROY surface (a) and EDS analysis at selected area (1, 2) (b, c), respectively.

**Table S4.** The EDS analysis of Pd@ROY surface at selected areas 1, 2.

Area	Element concentration [weight %]		
	C-K	O-K	Pd-L
surface 1	26.40	0.20	73.40
surface 2	28.53	0.22	71.25
Area	Element concentration [atom %]		

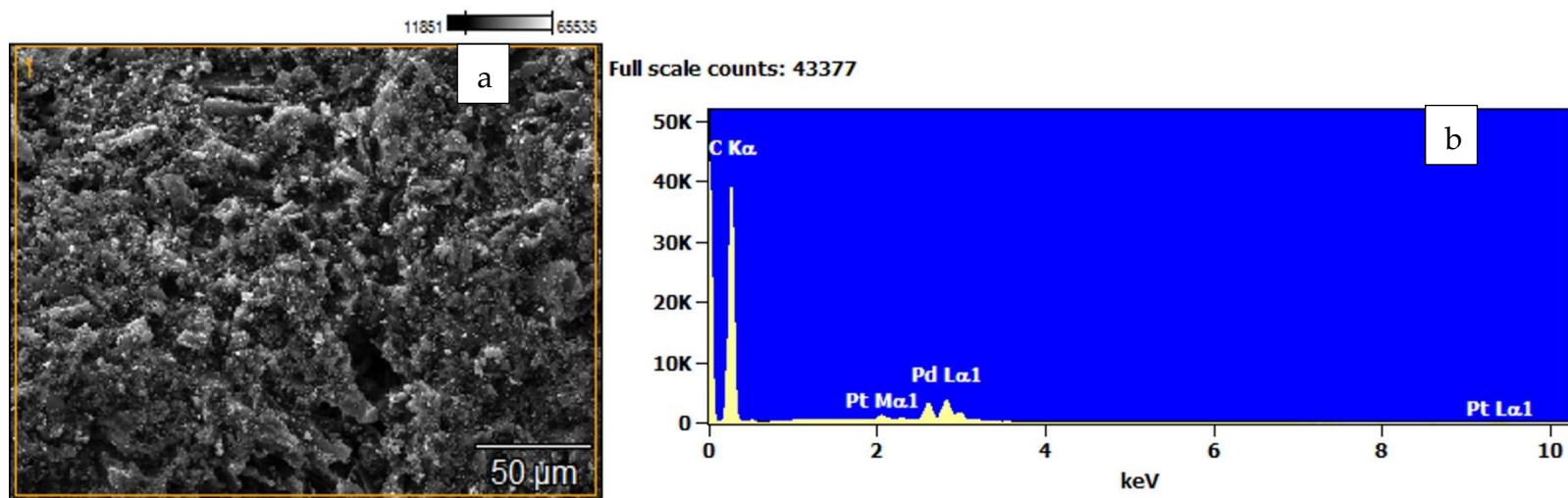
	C-K	O-K	Pd-L
surface 1	75.97	0.43	23.78
surface 2	77.66	0.44	21.89



**Figure S10.** The SEM micrographs of Pd@ROY cross section (a) and EDS analysis at selected area (1) (b).

**Table S5.** The EDS analysis of Pd@ROY cross section at selected area.

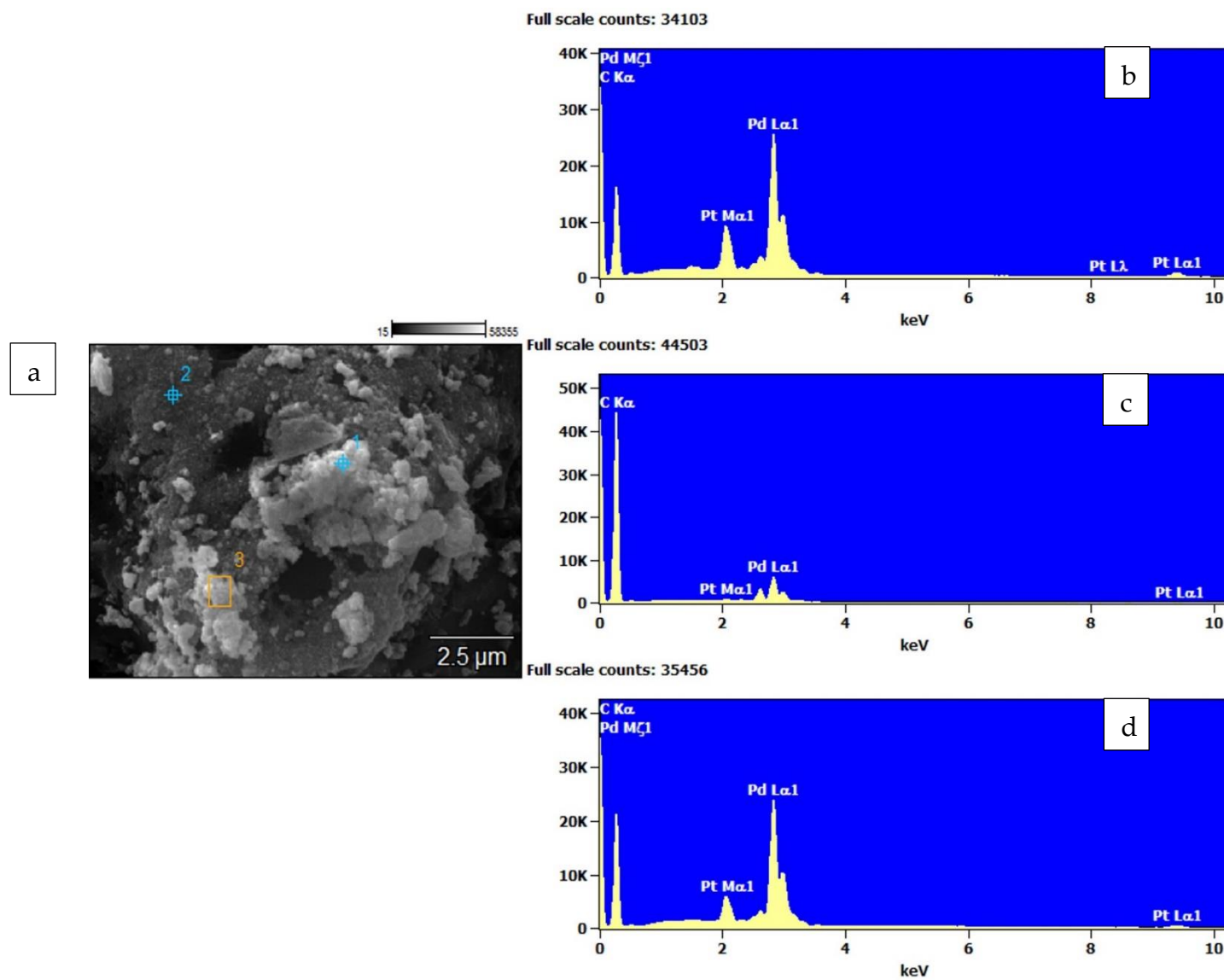
Area	Element concentration [weight %]	
	C-K	Pd-L
cross section	98.91	1.09
Area	Element concentration [atom %]	
	C-K	Pd-L
cross section	99.88	0.12



**Figure S11.** The SEM micrographs of Pd-Pt@ROY surface (a) and EDS analysis at selected area (1) (b).

**Table S6.** The EDS analysis of Pd-Pt@ROY surface (a) at selected area 1.

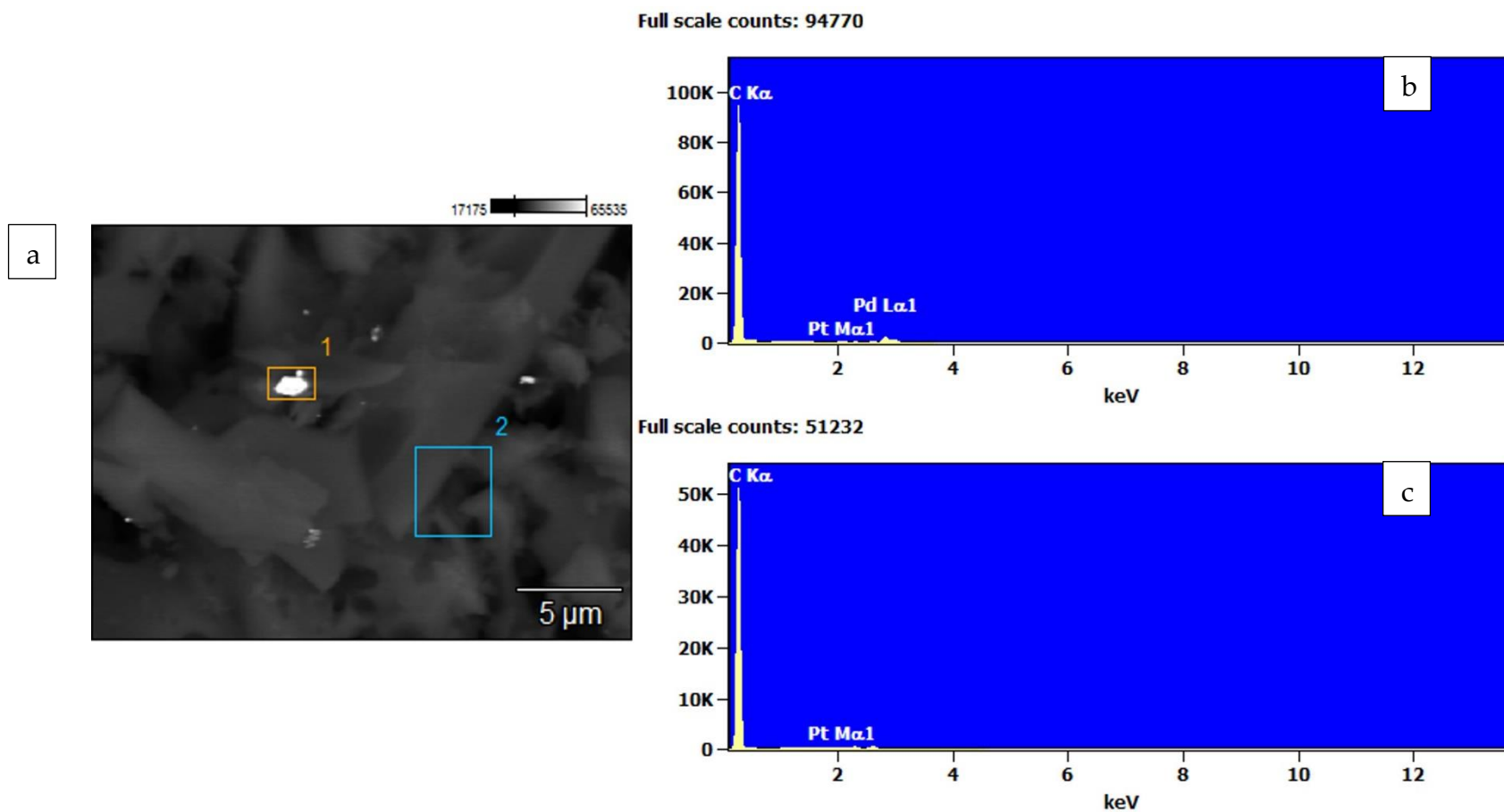
Area	Element concentration [weight %]		
	C-K	Pd-L	Pt-L
surface	89.14	7.28	3.58
Area	Element concentration [weight %]		
	C-K	Pd-L	Pt-L
surface	98.84	0.91	0.25



**Figure S12.** The SEM micrographs of Pd-Pt@ROY surface (a) and EDS analysis at selected point (1, 2) (b, c) and area (3) (d), respectively.

**Table S7.** The EDS analysis of Pd-Pt@ROY surface (a) selected point (1, 2) (b, c) and area (3) (d), respectively.

Area	Element concentration [weight %]		
	C-K	Pd-L	Pt-L
Point 1	34.30	47.93	17.77
Point 2	86.63	11.99	1.38
Surface 1	42.14	45.30	12.56
Area	Element concentration [atom %]		
	C-K	Pd-L	Pt-L
Point 1	84.06	13.26	2.68
Point 2	98.37	1.54	0.10
Surface 1	87.74	10.65	1.61

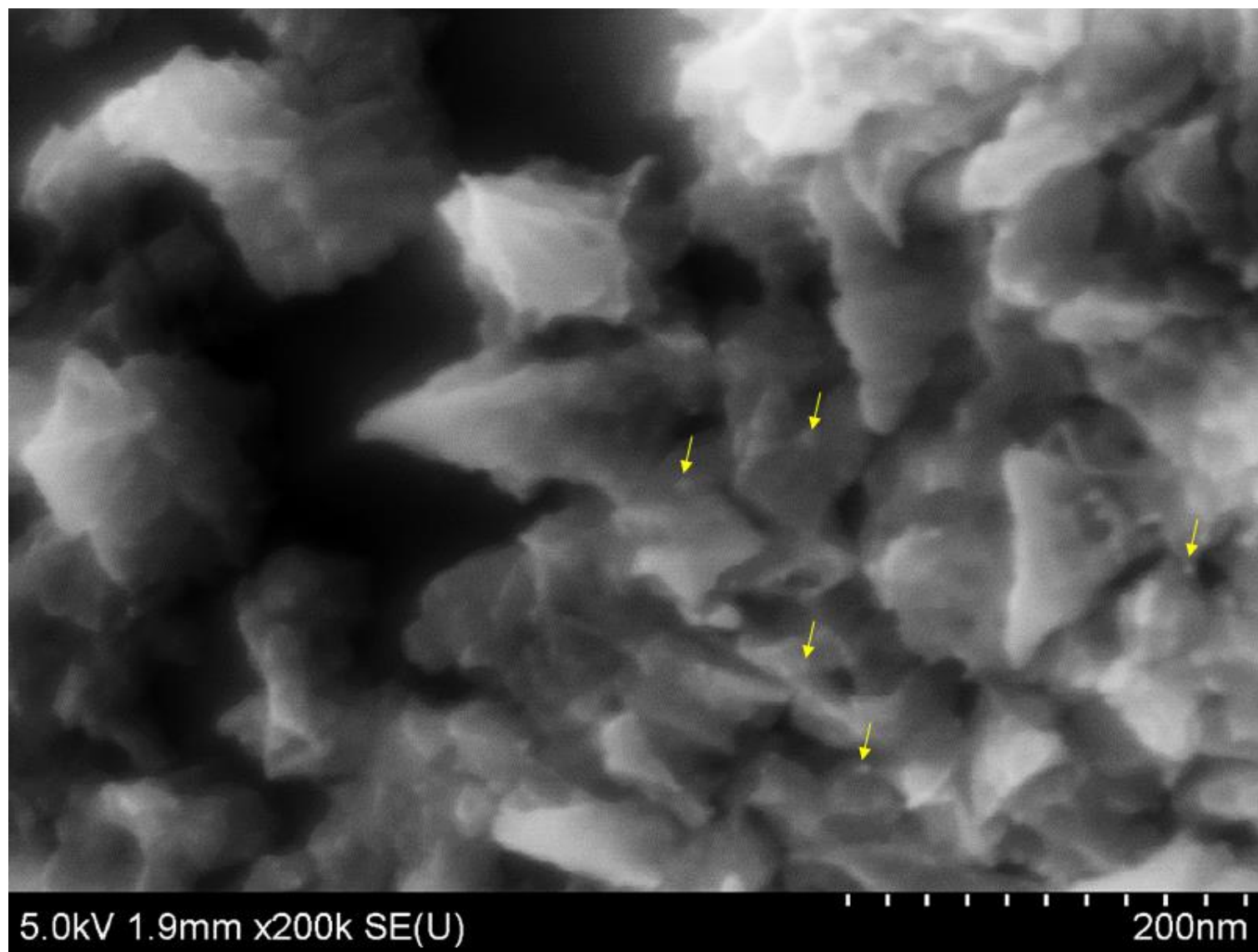


**Figure S13.** The SEM micrographs of Pd-Pt@ROY cross section (a) and EDS analysis at selected area (1, 2) (b, c).

**Table S8.** The EDS analysis of Pd-Pt@ROY cross section (a) at selected area (1, 2) (b, c), respectively.

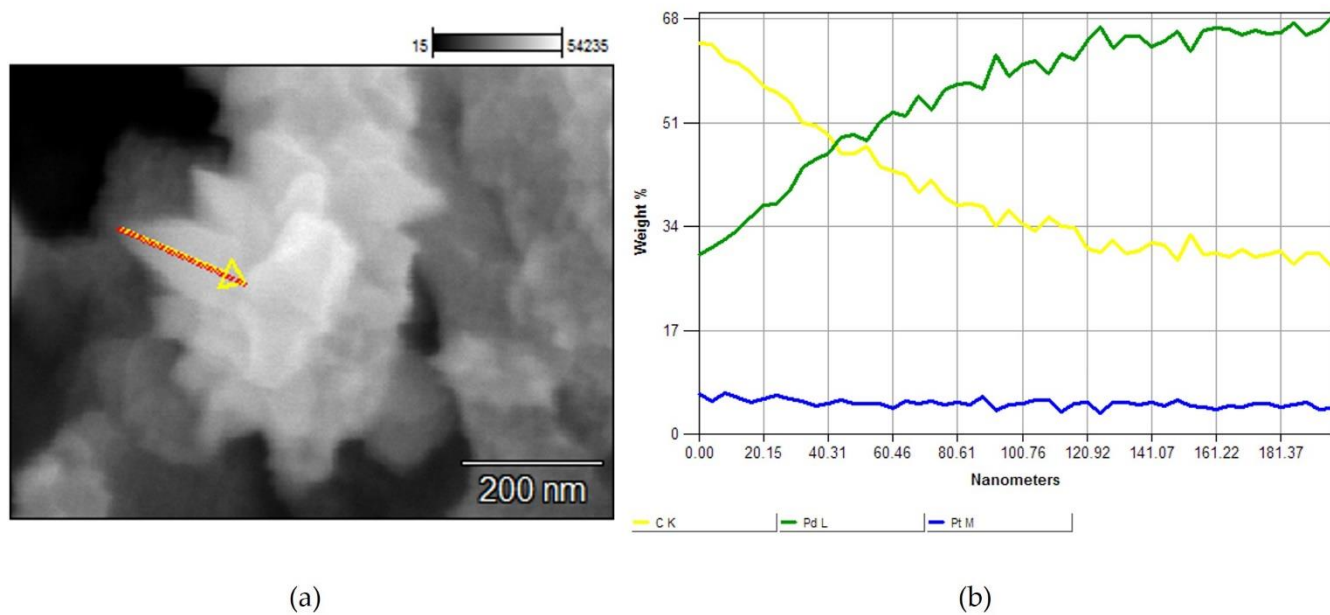
Area	Element concentration [weight %]		
	C-K	Pd-L	Pt-M
Cross section 1	92.33	6.81	0.87
Cross section 2	99.33	-	0.67
	C-K	Pd-L	Pt-M

Cross section 1	99.12	0.82	0.06
Cross section 2	99.96	-	0.04

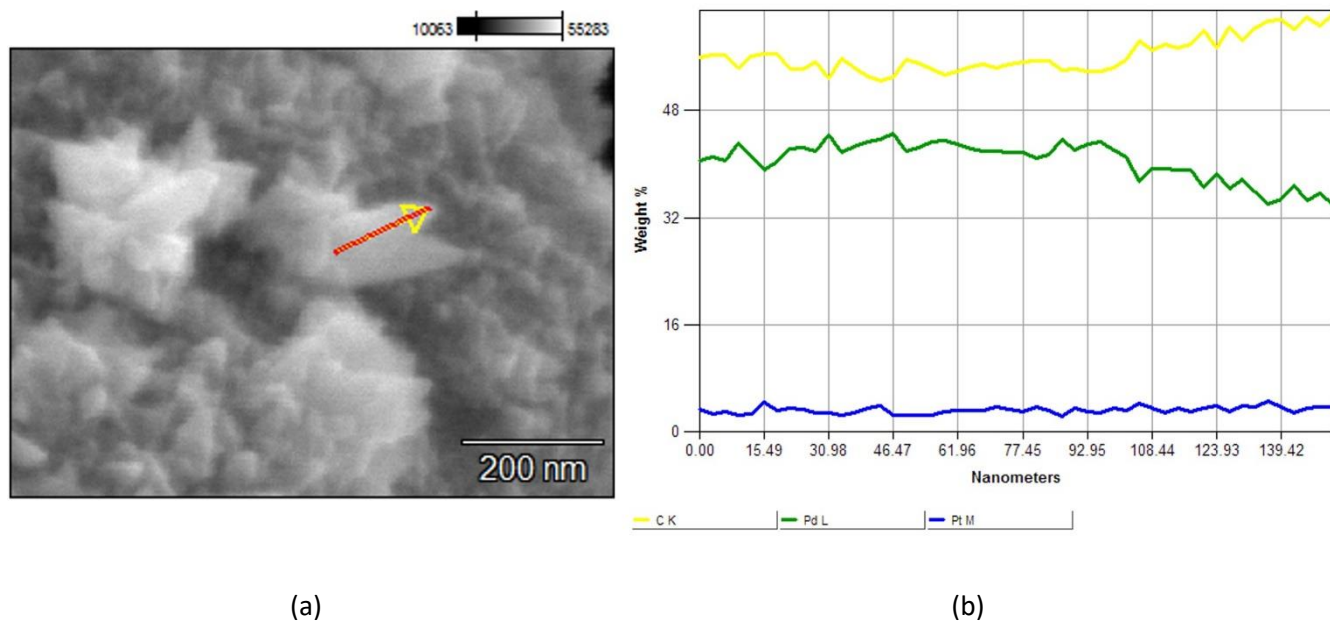


**Figure S14.** SEM analysis of Pd-Pt@ROY sample with selected points (PtNPs) (yellow arrow).





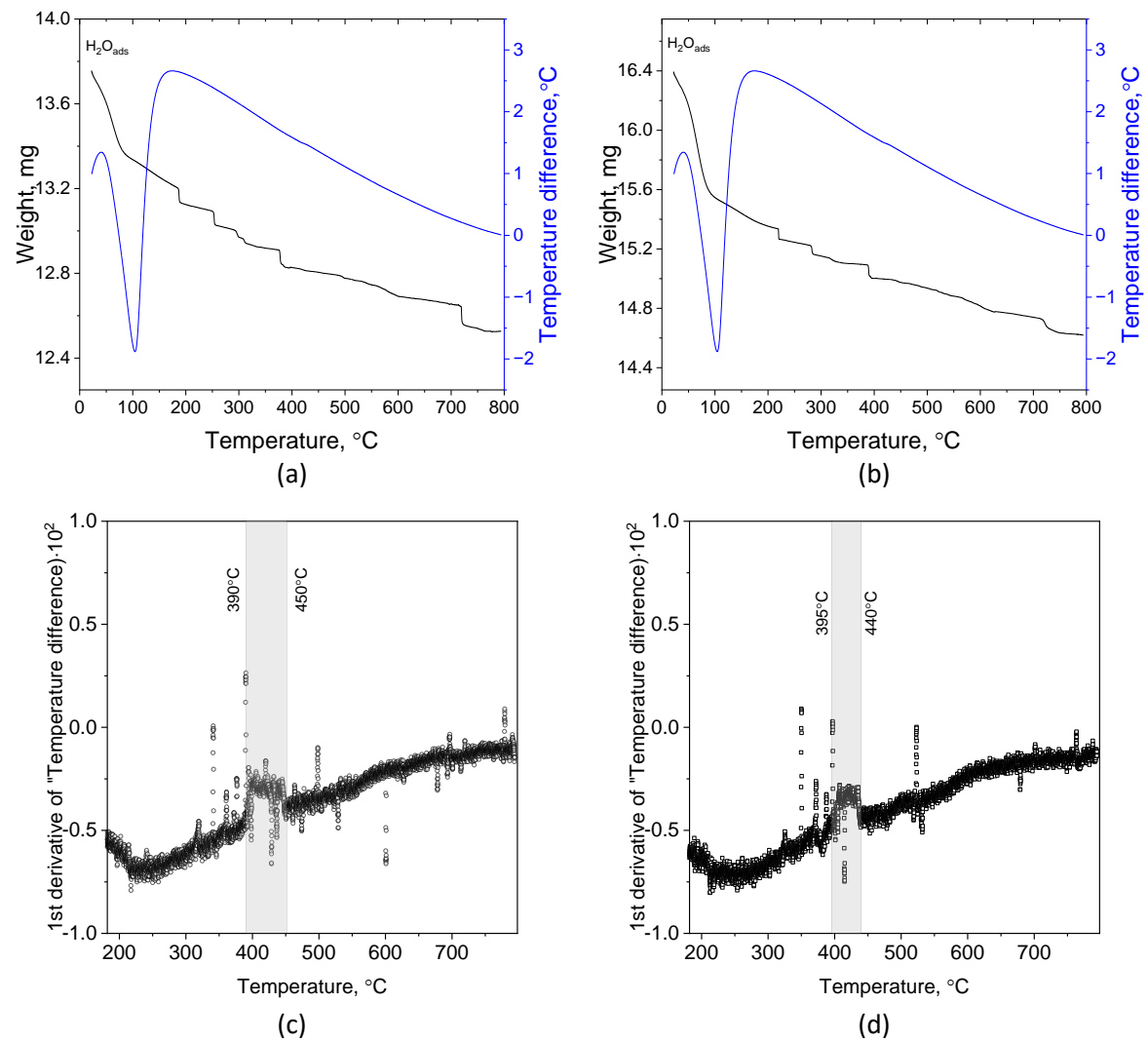
**Figure S15.** The SEM micrographs of Pd-Pt@ROY nanopyramid and marked direction of analysis (arrow) (a) and EDS profile at selected line (b).



**Figure S16.** The SEM micrographs of Pd-Pt@ROY nanopyramid and marked direction of analysis (arrow) (a) and EDS profile at selected line (b).

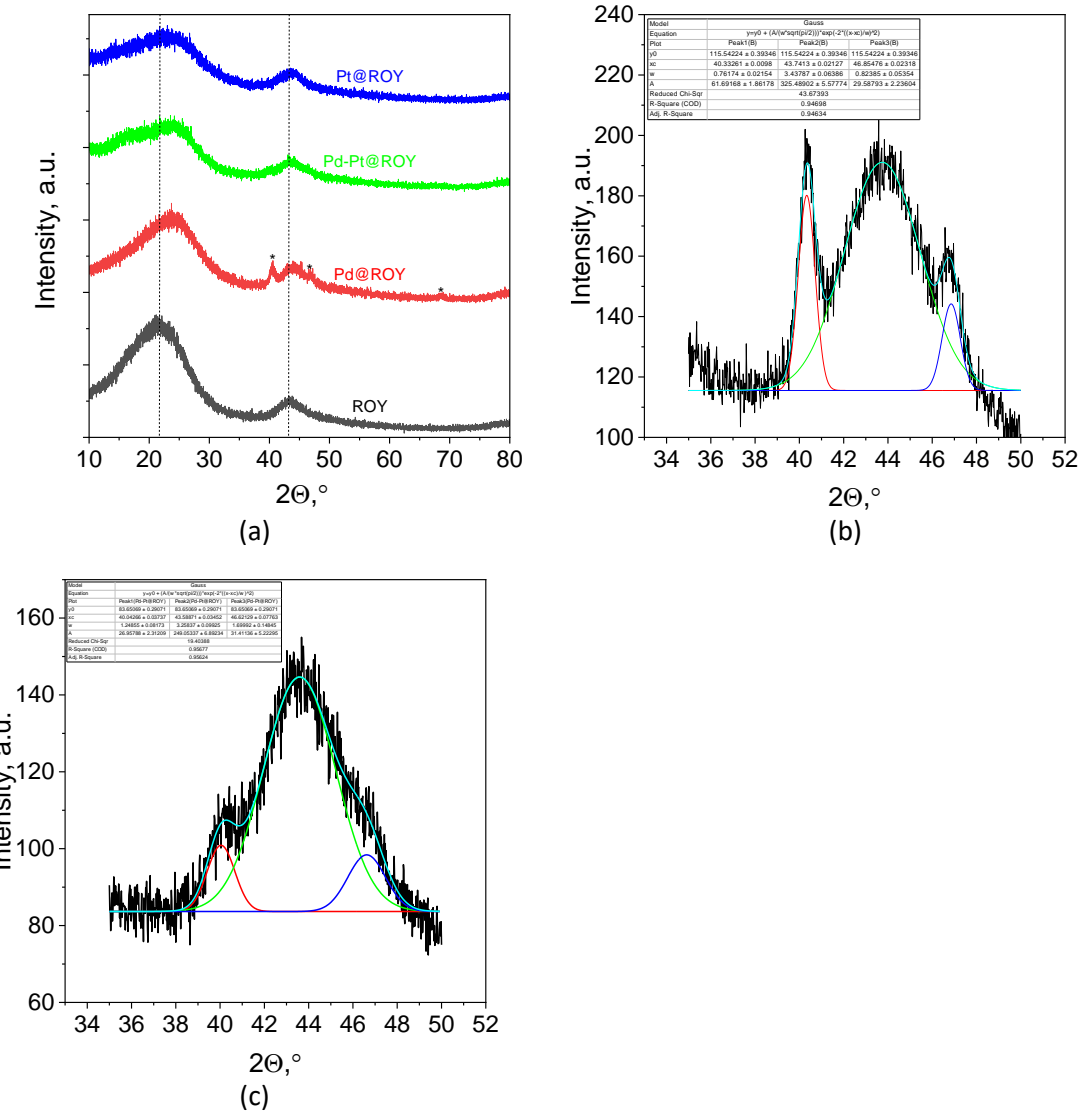
## 6. TGA analysis of Pd@ROY\_Cl and ROY\_Cl samples

Obtained results from TGA analysis for selected samples, i.e. Pd@ROY\_Cl and ROY\_Cl were shown in Fig. S17.



**Figure S17.** TGA of Pd@ROY\_Cl (a); ROY\_Cl samples (b); 1<sup>st</sup> derivative of temperature difference for Pd@ROY\_Cl (c) and ROY\_Cl (d) samples. Notation:  $H_2O_{ads}$  – adsorbed water.

7. XRD analysis



**Figure S18.** XRD patterns for ROY, Pd@ROY, Pd-Pt@ROY and Pt@ROY samples (a) \* denote peaks location characteristic for Pd; Fragment of XRD pattern for Pd@ROY sample and peak deconvolution using Gaussian mode in Origin software, and peaks assignments: 40.042, 43.589 and 46.621° (b); Fragment of XRD pattern for Pd-Pt@ROY sample and peak deconvolution using Gaussian mode in Origin software and peaks assignments: 40.333, 43.741 and 46.855° (c).