Supplementary Data – RSC Advances

Determination of the loading and stability of Pd in an arborescent copolymer in ethanol by microplasma-optical emission spectrometry

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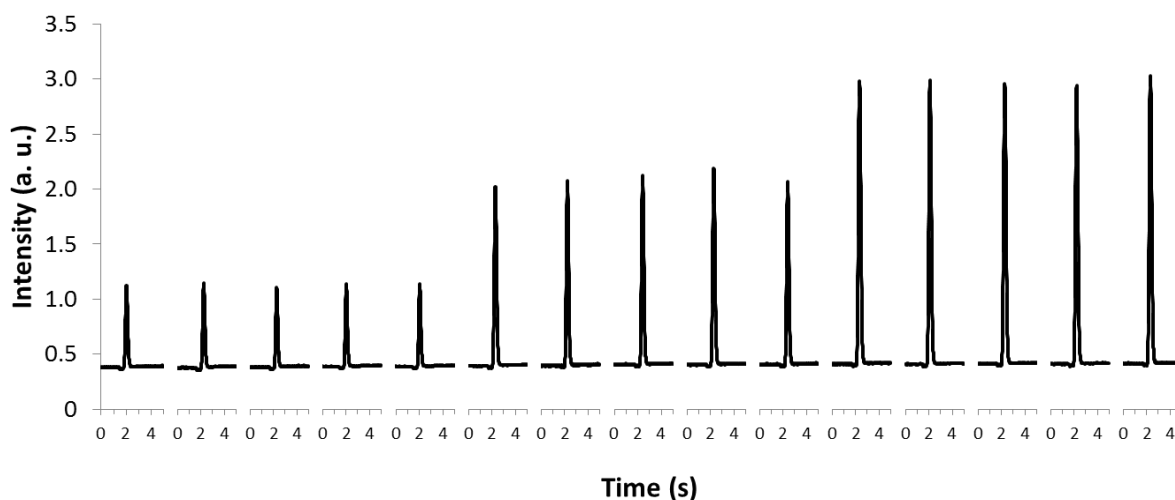E-mail address: gauthier@uwaterloo.ca

Figure S1: Examples of analyte emission signals acquired with the microplasma device over 5 successive runs using a Pd standard solution in water and injection volumes of 3.0, 6.0 and 10.0 µL, respectively. The precision of the signals, and the stability of intra-day microplasma background optical emission are noteworthy. Even though the microplasma was operated continuously for more than 10 hours for these experiments, only a small fraction of the analytical runs performed during this time period has been included above.
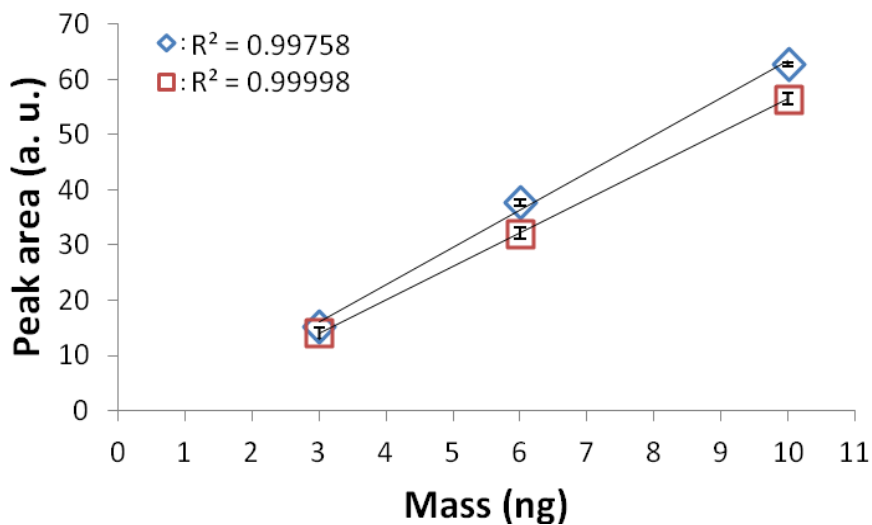
Figure S2: (color online). Calibration curves for Pd standard solutions in a) water (blue diamonds) and b) ethanol (red squares).
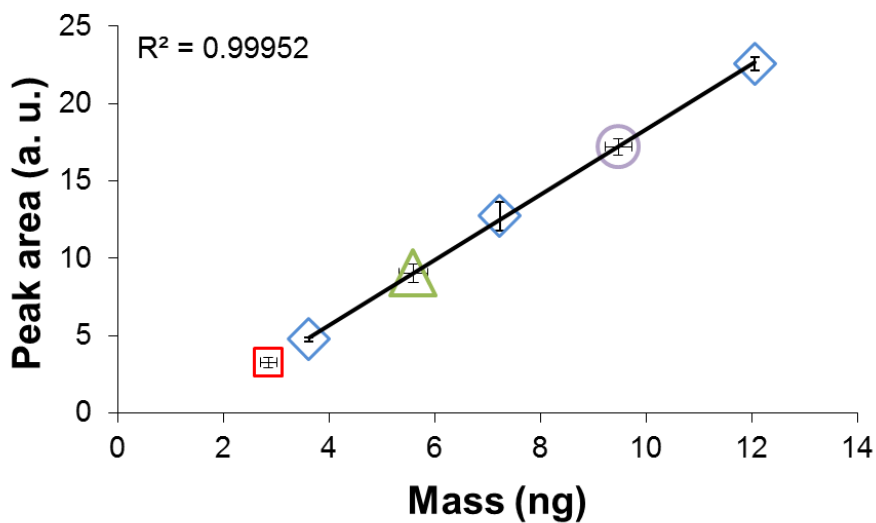

Figure S3: (color online). Calibration curve for Pd in a solution of $G1$-Pd(OAc)$_2$[0.25 equiv] in ethanol (blue diamonds), and analysis of dialyzed sample $G1$-Pd(OAc)$_2$ using injection volumes of 3.0 µL (red square), 6.0 µL (green triangle) and 10.0 µL (purple circle).

Table S1: Precision[a] and detection limit[b] for solutions of Pd-Std in water and ethanol, and *G*1-Pd(OAc)$_2$ in ethanol, determined for a Pd mass ranging from 3-10 ng.

Injection volume (µL)	Pd-Std in Water		Pd-Std in EtOH		*G*1-Pd(OAc)$_2$ in EtOH	
	Precision (%RSD)	Detection limit (pg)	Precision (%RSD)	Detection limit (pg)	Precision (%RSD)	Detection limit (pg)
3.0	0.1	33	7.4	62	3.8	28
6.0	1.7	16	5.7	62	1.3	29
10.0	0.5	44	0.7	65	2.1	26
Average[c]		31 ± 14		63 ± 2		28 ± 2

[a] The precision was determined in terms of the peak area percent relative standard deviation (%RSD), and is the average of at least 3 measurements. [b] The detection limit was estimated using the 3σ criterion, and from a minimum of 3 measurements. [c] Average and standard deviation for all the injection volumes, determined from a minimum of 9 measurements.

Table S2: Pd quantification with 3, 6 and 10 µL injection volumes for solutions prepared with 1.5 molar equivalent of Pd per 2VP units (*G*1-Pd[100 mol %]).

Injection volume (µL)	Calculated Pd Mass (ng)[a]	Pd Attached (mol %)[b]	Calculated Pd/2VP (mol %)	Precision (%RSD)[c]
3.0	2.12	66.1	98.4	2.3
6.0	4.09	64.6	96.3	3.3
10.0	7.42	69.7	103.9	3.0
Average[d]		66.8 ± 2.8	99.5 ± 4.2	

[a] Mass measured with the microplasma-OES instrument, obtained from a minimum of 3 measurements. [b] Mole percent of Pd remaining after dialysis. [c] The precision is expressed in terms of the percent relative standard deviation, in relation to the mol % of Pd/2VP. [d] Average and standard deviation for all the injection volumes, determined from a minimum of 9 measurements. Based on pooled standard deviation and a t-test, it was found statistically valid (at the 95% confidence level) to retain the Pd mass (ng) determined using 3 µL volumes despite the slight extrapolation of the calibration curve to lower concentrations.

Table S3: Pd quantification with 3, 6 and 10 µL injection volumes for solutions prepared with 0.25 molar equivalent of Pd per 2VP units (*G*1-Pd[24 mol %]).

Injection volume (µL)	Calculated Pd Mass (ng)[a]	Pd attached (mol %)[b]	Calculated Pd/2VP (mol %)	Precision (%RSD)[c]
3.0	2.85	93.9	23.8	5.4
6.0	5.59	92.1	23.4	4.9
10.0	9.47	93.6	23.7	2.7
Average[d]		93.3 ± 3.7	23.7 ± 0.9	

[a] Mass measured with the microplasma-OES instrument, obtained from a minimum of 3 measurements. [b] Mole percent of Pd remaining after dialysis. [c] The precision is expressed in terms of the percent relative standard deviation, in relation to the mol % of Pd/2VP. [d] Average and standard deviation for all the injection volumes, determined from at least 9 measurements. The Pd mass (ng) determined from 3 µL was retained despite the slight extrapolation of the calibration curve (based on statistical tests detailed in the caption of Table S2).
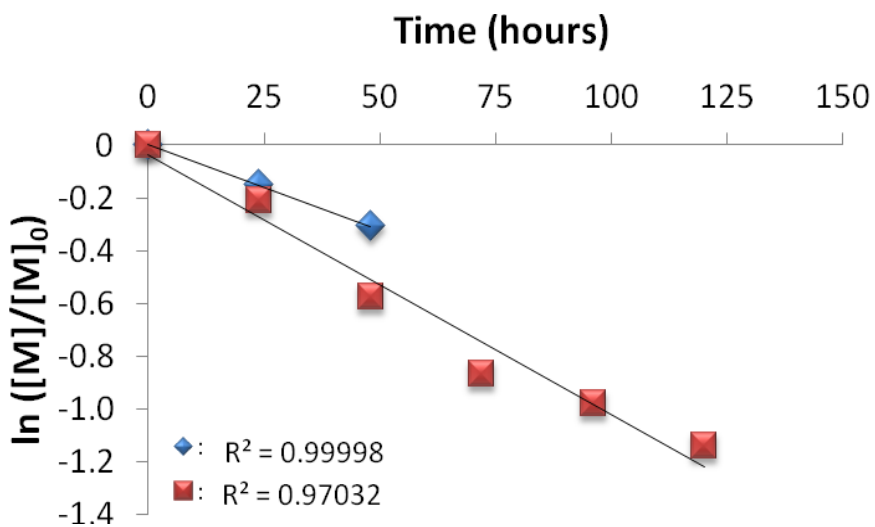
Figure S4: (color online). Graphical analysis of the first-order rate of aggregation of $Pd(OAc)_2$ in ethanol (red squares), and Pd in a standard solution in water (blue diamonds).
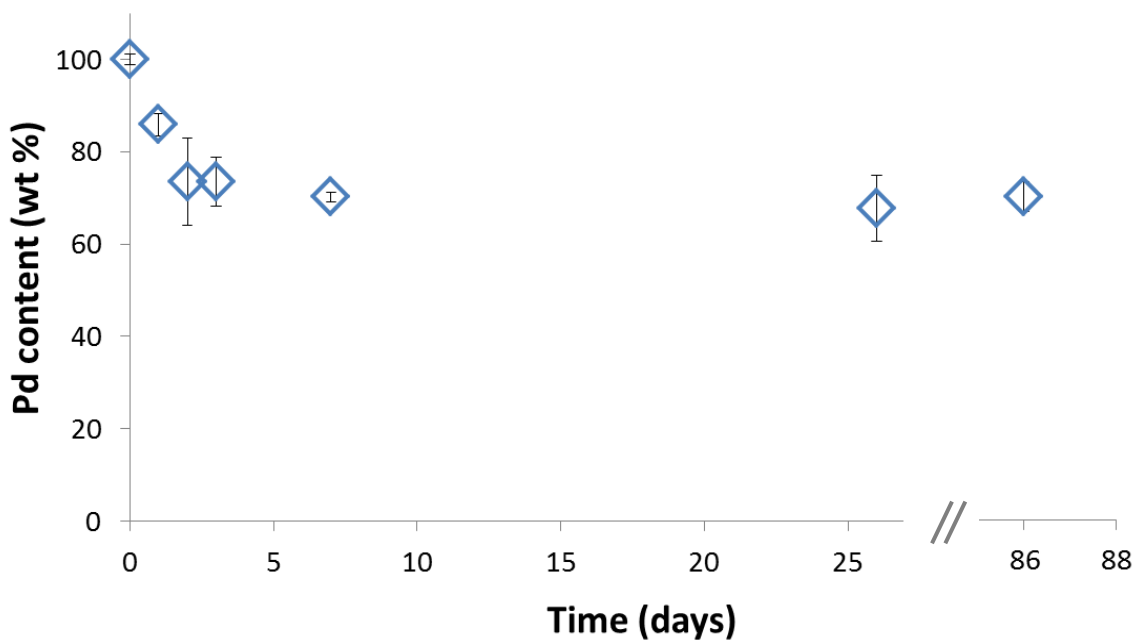

Figure S5: (color online). Change of the mass concentration of Pd in a standard solution in water as measured by microplasma-optical emission spectrometry.