Electronic Supplementary Material (ESI) for Environmental Science: Nano.

Supporting Information for

Nanoparticle size and natural organic matter composition determine aggregation behavior of polyvinylpyrrolidone coated platinum nanoparticles

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Collection of water samples for NOM isolation

Water samples for natural organic matter (NOM) extraction were collected from four different environments including marshlands, a seepage lake, a blackwater river, and the ocean (Table S1). Water samples were collected from marchlands at the air-water interface from three different sites in the Florida Everglades representing pristine (LOX 8 site, NOM 6), relatively pristine (2B South site, NOM 2), and eutrophic environments (F1 site, NOM 1). Marine water from the surface (Pacific Ocean; NOM 4) was collected at the Natural Energy Laboratory of Hawaii Authority (NELHA) on the island of Hawaii, USA (near Kailua-Kona). At NELHA, surface (5 m depth) oceanic waters were pumped continuously at a rate of more than 0.5 m^3 s^{-1} through high-density polyethylene (HDPE) distribution systems. Water was transferred from HDPE pipelines to the laboratory through polyvinylchloride (PVC) pipes that were flushed continuously for several days before and throughout the study. The temperatures of surface water at NELHA was 24 to 28.5 °C. Water was collected from Williams Lake (MN; NOM 3), a groundwater fed lake in North-Central Minnesota dominated by NOM of autochthonous sources (e.g. algae, bacteria). Lastly, water was collected from the Suwannee River (GA; NOM5), a blackwater river draining the Okeefenokee Swamp.

Solid phase extraction of natural organic matter (NOM)

The hydrophobic organic acid fraction (HPOA) was isolated from the six water samples according to procedure described in Aiken et al. (1992) under ambient atmosphere [1] and are labeled NOM 1 to 6 as summarized in Table S1. The isolation procedure was consistent with recommendation of the International Humic Substances Society (IHSS) to isolate humic and fulvic acids. Samples were acidified to pH 2 with 12 N HCl, and loaded onto a pre-cleaned XAD-8 column. Chloride was removed from the HPOA fraction retained on the XAD-8 column by rinsing with high purity water until the conductivity of the effluent was < 700 µS cm^{-1}. The sample was back eluted off the resin with 0.1 M NaOH through a proton-saturated cation exchange resin (CER; Bio-Rad Laboratories) to remove sodium ions. Eluting the sample directly through a CER column minimized the contact time between the sample and the 0.1 M NaOH to minimize the possibility for oxidation of reduced organic sulfur species [2]. The pH of de-salted HPOA samples was approximately 3.3. HPOA samples were freeze dried immediately.

UV-vis optical measurement of NOM

Ultraviolet and visible light (UV-vis) absorption spectra were measured from 190 to 800 nm using spectrophotometer (Agilent Technologies, model 8453) and a 1cm quartz cuvette. Sample spectra were measured with respect to a blank spectrum, containing ultra-high purity water (UHPW). The specific ultraviolet absorbance at 254 nm ($SUVA_{254}$) of NOM samples, a proxy aromaticity [3], is defined as the decadic UV absorbance at 254 nm divided by the dissolved organic carbon (DOC) concentration. Decadic absorbance values were converted to absorption coefficient as follows:

$$\alpha_\lambda = \frac{A_\lambda}{l} \tag{S1}$$

where, α_λ is the absorption coefficient (cm^{-1}), A_λ is the absorbance, and l is the path length (cm). The $SUVA_{254}$ values were calculated by dividing the decadic absorption coefficient at 254 nm (α_{254}) by DOC concentration and reported in units of L mg^{-1} m^{-1}. [3]

Table S1. Sampling site descriptions and chemical characteristics of NOM hydrophobic organic acid (HPOA) samples including elemental analysis and specific ultraviolet absorbance at 254 nm ($SUVA_{254}$).

NOM ID	NOM Sample	Sampling site description [a]	Weight % [b]						$SUVA_{254}$ ($L\ mg^{-1}\ m^{-1}$)
			C	H	O	N	S	Ash	
NOM 1	Florida Everglades F1 site HPOA	Eutrophic marshland located in Water conservation Area (WCA) 2A in the northern Everglades. Vegetation dominated by cattails	53.1	4.4	39.3	1.3	1.9	2.8	4.0
NOM 2	Florida Everglades 2B south HPOA	Relatively pristine marshland located in WCA 2B in the northern Everglades. Vegetation dominated by saw grass	52.2	4.8	40.2	1.6	1.2	7.3	3.2
NOM 3	Williams lake HPOA (MN)	Seepage lake in North-Central Minnesota. Organic matter dominated by autochthonous sources (*e.g.* algae, bacteria)	55.2	5.7	36.5	1.8	0.8	2.1	1.9
NOM 4	Pacific Ocean HPOA	Sample collected at Natural Energy Laboratory of Hawaii Authority (NELHA) on the island of Hawaii, near Kailua-Kona, from 5 m depth. Organic matter of marine origin	56.5	6	36	1.1	0.4	1.7	0.8
NOM 5	Suwannee river HPOA (GA)	Blackwater river draining Okeefenokee Swamp. Sampled at Fargo, Georgia. Vegetation dominated by southern floodplain forest (Quercus, Nyassa, Taxodium)	51.8	4.4	42.7	0.8	0.5	4.8	4.8
NOM 6	Florida Everglades Loxahatchee National Wildlife refuge (LOX) HPOA	Pristine marshland in located in LOX 8 wildlife refuge area in the northern Everglades.	51	4.9	n.m.	n.m.	0.6	3.5	3.5

[a] Site description modified from Poulin et al (2017) [4]

[b] Measured by Huffman Hazen Laboratories (Golden, CO); major elemental composition (*i.e.* C, H, O, N, and S) reported as ash-free dry mass

n.m.= not measured

Table S2. Criteria applied for molecular assignment of formulae detected by FT-ICR-MS

	H/C	O/C	# of N
Tannin	0.5-1.25	0.60-0.95	NA
Carbohydrates	1.5-2.0	0.7-1.0	NA
Lipids	1.7-2.25	<0.22	NA
Lignin/CRAM	0.75-1.5	0.2-0.6	NA
Protein	1.5-2.0	0.2-0.5	N≥1
Aminosugar	1.5-1.75	0.55-0.70	N≥1
Condensed hydrocarbons	0.2-0.75	<0.7	NA

CRAM: carboxyl rich alicyclic molecules

Table S3. Relative abundance (percent of total formulas) of molecules grouped based on heteroatom composition as determined by FT-ICR-MS.

NOM ID	Sampling site	CHO (%)	CHON (%)	CHOS (%)	CHOP (%)	CHONS (%)	CHONP (%)	CHONSP (%)	Unassigned (%)
NOM 1	Everglades, Site F1, FL	66.1	16.6	7.5	0.4	5.1	3.2	0.3	0.8
NOM 2	Everglades, Site WCA-2BS, FL	61.8	25.6	5.0	0.8	2.9	3.2	0.2	0.5
NOM 3	Williams Lake, MN	65.0	23.4	4.0	4.1	1.9	0.9	0.5	0.2
NOM 4	Pacific Ocean near Hawaii, surface water (NELHA)	67.4	21.7	1.6	2.4	4.4	0.9	1.1	0.4
NOM 5	Suwannee River, GA	73.6	6.4	1.4	1.3	8.1	6.3	0.7	2.1
NOM 6	Everglades, LOX8, FL	68.5	21.0	2.1	1.8	3.9	2.1	0.2	0.4

Unassigned: Formulas did not meet criteria for compound classifications.

Table S4. Relative abundance (percentage of total formulas) of molecules grouped based on compound class of compounds determined by FT-ICR-MS

NOM ID	Sampling site	Amino sugar (%)	Carb (%)	ConHC (%)	Lignin/ CRAM (%)	Lipid (%)	Protein (%)	Tannin (%)	Unsat HC (%)	Unassigned (%)
NOM 1	Everglades, Site F1, FL	0.8	0.8	24.5	59.0	0.9	2.3	9.8	0.7	1.2
NOM 2	Everglades, Site WCA-2BS, FL	1.1	0.6	19.9	63.4	0.8	3.3	9.4	0.4	1.0
NOM 3	Williams Lake, MN	1.5	0.4	6.6	72.4	1.9	8.0	8.5	0.3	0.4
NOM 4	Pacific Ocean near Hawaii, surface water (NELHA)	1.4	0.3	1.9	79.6	1.5	8.8	5.4	0.6	0.5
NOM 5	Suwannee River, GA	1.0	1.0	25.0	56.3	0.9	2.9	8.9	2.0	1.8
NOM 6	Everglades, LOX8, FL	0.9	0.7	20.3	62.3	0.9	3.3	10.3	0.6	0.5

*Carb: Carbohydrate; ConHC: Condensed hydrocarbon; CRAM: carboxyl rich alicyclic molecules; Unsat HC: Unsaturated hydrocarbon; Unassigned: Formulas did not meet criteria for compound classifications.

Table S5. O/C, H/C and molecular weight (MW) of NOM samples calculated as the number averages of O/C, H/C, and MW of NOM formulas determined by FT-ICR-MS.

NOM ID	Sampling site	Num. Avg. O/C	Num. Avg. H/C	Num. Avg. MW
NOM 1	Everglades, Site F1, FL	0.52	1.05	391
NOM 2	Everglades, Site WCA-2BS, FL	0.52	1.12	369
NOM 3	Williams Lake, MN	0.51	1.26	372
NOM 4	Pacific Ocean near Hawaii, surface water (NELHA)	0.48	1.27	442
NOM 5	Suwannee River, GA	0.50	1.07	375
NOM 6	Everglades, LOX8, FL	0.52	1.12	383

O/C: Oxygen-carbon ratio

H/C: Hydrogen -carbon ratio

Table S6. Chemical composition of moderately hard water (MHW)

Chemical constituent	Concentration (mM)
Sodium bicarbonate ($NaHCO_3$)	1.14
Calcium sulfate ($CaSO_4$)	0.44
Magnesium sulfate ($MgSO_4$)	0.50
Potassium chloride (KCl)	0.05

Table S7. Physicochemical properties of synthesized PVP-PtNPs reported as mean ± standard deviation of three experimental replicates.

PVP-PtNPs suspension	Concentration of PtNPs stock suspension (mg L^{-1})	Core size measured by TEM, $d_{TEM} \pm \sigma_d$ (nm)[a]	Hydrodynamic size measured by DLS, $d_{DLS} \pm \sigma_d$ (nm)[b]	Zeta potential (mV)[b]	% PtNPs detectable by sp-ICP-MS (> 17 nm)	pH	Polydispersity index (PDI)
PtNP$_{20}$	28.4 ± 0.5	9.2 ± 1.2	18.9 ± 0.3	-16.9 ± 3.5	0	7.0	0.36
PtNP$_{30}$	30.2 ± 0.3	10.9 ± 0.8	31.4 ± 0.8	-19.3 ± 1.9	0	6.9	0.19
PtNP$_{50}$	29.7 ± 1.5	18.5 ± 5.0	51.0 ± 0.7	-22.9 ± 1.4	46	7.1	0.20
PtNP$_{75}$	32 ± 0.5	44.5 ± 2.7	74.7 ± 0.2	-25.1 ± 3.0	100	6.9	0.03
PtNP$_{95}$	28.6 ± 0.6	72.5 ± 3.9	93.4 ± 1.0	-27.2 ± 1.7	100	6.8	0.10

σ_d is the standard deviation.

[a] The standard deviation is that of the size distribution.

[b] The standard deviation is that of 3 experimental replicates.

Table S8. Number concentration of 1 μg L^{-1} PVP-PtNPs (PtNP$_{20}$-PtNP$_{95}$) in ultrapure water (UPW) and moderately hard water (MHW) measured by single particle-inductively coupled plasma-mass spectrometer (sp-ICP-MS) and % of measured PtNPs by sp-ICP-MS relative to the theoretical number concentrations calculated from the measured mass concentration and respective NP diameter. All values are reported as the mean ± standard deviation of three experimental replicates.

			in UPW- 0 h			in MHW- 0 h			in MHW- 24 h		
Sample ID	Theoretical number conc. (x10^6 NP mL^{-1})	Theoretical total surface area (x10^9 nm^2 mL^{-1})	Mean size (nm)	Measured number conc. (x10^6 NP mL^{-1})	% measured relative to the theoretical conc.	Mean size (nm)	Measured number conc. (x10^6 NP mL^{-1})	% measured relative to the theoretical conc.	Mean size (nm)	Measured number conc. (x10^6 NP mL^{-1})	% measured relative to the theoretical conc.
PtNP$_{20}$	114.34	30	26.4±11.6	0.31±0.09	0.3±0.1	31±12	1.77±0.19	1.5±0.2	38±19	9.99±0.07	8.7±0.1
PtNP$_{30}$	68.75	26	30.2±12.9	0.83±0.1	1.2±0.1	31±11	1.05±0.05	1.5±0.1	48±15	9.09±0.57	13.2±0.8
PtNP$_{50}$	14.06	15	34.3±10.1	4.65±0.43	33.1±3.1	38±10	2.63±0.26	18.7±1.8	54±13	2.61±0.3	18.5±2.1
PtNP$_{75}$	1.01	6.3	44.1±7.7	1.09±0.04	108.1±2	59±9	0.50±0.05	49.6±2.2	62±9	0.44±0.04	43.2±3.9
PtNP$_{95}$	0.23	3.8	76.7±9.7	0.23±0.03	100.2±6.4	74±9	0.23±0.06	98.0±2.6	74±8	0.18±0.03	75.5±1.6

Table S9. Zeta potential of 1 mg L^{-1} PVP-PtNPs at 0 and 24 hours after mixing with MHW. Zeta potential values are reported as mean ± standard deviation of three experimental replicates.

PVP-PtNPs suspension	pH of PtNPs suspension in MHW	Zeta potential in UPW (mV)	Zeta potential in MHW (mV)	
			0 h	24 h
$PtNP_{20}$	8.1	-16.9 ± 3.5	-13.2 ± 0.8	-7.7 ± 0.8
$PtNP_{30}$	7.9	-19.3 ± 1.9	-14.7 ± 1.0	-9.4 ± 2.2
$PtNP_{50}$	8.0	-22.9 ± 1.4	-16.4 ± 1.1	-13.9 ± 0.6
$PtNP_{75}$	8.0	-25.1 ± 3.0	-19.3 ± 0.9	-17.9 ± 4.2
$PtNP_{95}$	8.1	-27.2 ± 1.7	-23.3 ± 0.8	-22.9 ± 1.4

Table S10. Mass based concentrations of 1 µg L^{-1} PtNPs ($PtNP_{20}$-$PtNP_{95}$) in in ultrapure water (UPW) and moderately hard water (MHW) measured by sp-ICP-MS and % of detected PtNP mass by sp-ICP-MS relative to the nominal mass concentration (1 µg L^{-1}).

	in UPW- 0 h		in MHW- 0 h		in MHW- 24 h	
Sample ID	Pt conc. (µg L^{-1})	% measured relative to the nominal conc.	Pt conc. (µg L^{-1})	% measured relative to the nominal conc.	Pt conc. (µg L^{-1})	% measured relative to the nominal conc.
$PtNP_{20}$	0.017 ± 0.007	1.7 ± 0.7	0.209 ± 0.004	20.9 ± 0.4	0.583 ± 0.016	58.3 ± 1.6
$PtNP_{30}$	0.082 ± 0.007	8.2 ± 0.7	0.426 ± 0.057	42.6 ± 5.7	0.583 ± 0.015	58.3 ± 1.5
$PtNP_{50}$	0.313 ± 0.036	31.3 ± 3.6	0.290 ± 0.048	29.0 ± 4.8	0.046 ± 0.012	4.60 ± 1.2
$PtNP_{75}$	0.851 ± 0.029	85.1 ± 2.9	0.813 ± 0.048	81.3 ± 4.8	0.685 ± 0.049	68.5 ± 4.9
$PtNP_{95}$	0.824 ± 0.028	82.4 ± 2.8	0.706 ± 0.015	70.6 ± 1.5	0.373 ± 0.003	37.3 ± 0.3

Table S11. Number concentration of 1 µg L^{-1} $PtNP_{20}$ suspended in UPW and in MHW in presence of 1 mg L^{-1} NOM at 0 and 24 hours post mixing and % of number of detected PtNPs to the theoretically calculated number concentrations. All analyses were performed using sp-ICP-MS. All values are reported as the mean ± standard deviation of three experimental replicates.

Sample ID	Theoretical number conc. ($x10^6$ NP mL^{-1})[a]	at 0 h			after 24 h		
		Mean size (nm)	Measured number conc. ($x10^6$ NP mL^{-1})	% measured relative to the theoretical conc.	Mean size (nm)	Measured number conc. ($x10^6$ NP mL^{-1})	% measured relative to the theoretical conc.
in UPW (without NOM)	114.34	32 ± 10	0.44 ± 0.03	0.4 ± 0.03	34 ± 14	0.80 ± 0.05	0.7 ± 0.04
in MHW (without NOM)		31 ± 16	0.52 ± 0.03	0.5 ± 0.03	42 ± 10	15.89 ± 5.2	13.9 ± 4.37
NOM 1		54 ± 24	0.32 ± 0.02	0.3 ± 0.02	53 ± 14	12.73 ± 3.12	11.1 ± 2.62
NOM 2		42 ± 16	0.48 ± 0.03	0.4 ± 0.03	66 ± 21	22.46 ± 5.41	19.6 ± 4.37
NOM 3		53 ± 20	0.34 ± 0.02	0.3 ± 0.02	56 ± 14	17.92 ± 4.12	15.7 ± 3.5
NOM 4		47 ± 21	0.66 ± 0.04	0.6 ± 0.03	74 ± 34	58.89 ± 6.39	51.5 ± 6.12
NOM 5		46 ± 20	0.55 ± 0.06	0.5 ± 0.05	53 ± 13	19.79 ± 1.65	17.3 ± 1.75
NOM 6		52 ± 18	0.50 ± 0.05	0.4 ± 0.04	56 ± 14	14.89 ± 0.90	13.0 ± 0.79

[a] Theoretically calculated from the mass concentration and NP diameter

Table S12. Mass concentration of 1 μg L^{-1} $PtNP_{20}$ suspended in UPW and in MHW in presence of 1 mg L^{-1} NOM at 0 and 24 hours post mixing and % measured PtNP mass relative to the actual concentration (= 1 μg L^{-1}). All analyses were performed using sp-ICP-MS.

Sample ID	at 0 h		after 24 h	
	Pt conc. measured by ICP-MS (μg L^{-1})	% measured to the actual conc.	Pt conc. measured by ICP-MS (μg L^{-1})	% measured to the actual conc.
in UPW (without NOM)	0.017 ± 0.004	1.7 ± 0.4	0.02 ± 0.007	2.0 ± 0.70
in MHW (without NOM)	0.033 ± 0.003	3.3 ± 0.3	0.308 ± 0.120	30.8 ± 12.0
NOM 1	0.016 ± 0.002	1.6 ± 0.2	0.188 ± 0.090	18.8 ± 9.0
NOM 2	0.016 ± 0.002	1.6 ± 0.2	0.593 ± 0.176	59.3 ± 17.6
NOM 3	0.019 ± 0.003	1.9 ± 0.3	0.489 ± 0.209	48.9 ± 20.9
NOM 4	0.034 ± 0.007	3.4 ± 0.7	0.978 ± 0.078	97.8 ± 7.8
NOM 5	0.024 ± 0.006	2.4 ± 0.6	0.456 ± 0.142	45.6 ± 14.2
NOM 6	0.020 ± 0.005	2.0 ± 0.5	0.375 ± 0.100	37.5 ± 10.0

Table S13. Number based concentrations of 1 $\mu g\ L^{-1}$ $PtNP_{95}$ suspended in UPW and in MHW in presence of 1 mg L^{-1} NOM and % achieved to the theoretically calculated number concentrations, immediately after mixing and after 24 h. All values are reported as the mean ± standard deviation of three experimental replicates.

Sample ID	Expected number conc. (x10^6 NP mL^{-1})[a]	at 0 h			after 24 h		
		Mean size (nm)	Actual number conc. (x10^6 NP mL^{-1})[b]	% measured to the expected conc.	Mean size (nm)	Actual number conc. (x10^6 NP mL^{-1})[b]	% measured to the expected conc.
in UPW (without NOM)	0.23	76 ± 10	0.24 ± 0.02	102.2 ± 0.03	72 ± 10	0.19 ± 0.03	82.2 ± 0.06
in MHW (without NOM)		72 ± 16	0.25 ± 0.03	105.6 ± 0.05	71 ± 6	0.18 ± 0.02	76.0 ± 0.04
NOM 1		74 ± 14	0.22 ± 0.02	96.3 ± 0.03	74 ± 14	0.23 ± 0.03	98.1 ± 0.05
NOM 2		72 ± 8	0.19 ± 0.03	82.2 ± 0.04	72 ± 10	0.21 ± 0.02	89.8 ± 0.03
NOM 3		73 ± 10	0.17 ± 0.01	75.0 ± 0.03	73 ± 10	0.19 ± 0.02	80.4 ± 0.04
NOM 4		77 ± 11	0.20 ± 0.02	86.4 ± 0.03	77 ± 11	0.21 ± 0.03	89.0 ± 0.04
NOM 5		76 ± 10	0.24 ± 0.03	100.9 ± 0.04	76 ± 10	0.23 ± 0.05	97.8 ± 0.06
NOM 6		72 ± 8	0.18 ± 0.04	78.4 ± 0.06	72 ± 8	0.19 ± 0.03	83.3 ± 0.04

[a] Theoretically calculated from the mass concentration and NP diameter

[b] measured by sp-ICP-MS

Table S14. Mass based concentrations of 1 μg L^{-1} $PtNP_{95}$ suspended in UPW and in MHW in presence of 1 mg L^{-1} NOM and % measured to the actual concentration (1 μg L^{-1}), immediately after mixing and after 24 h. All values are reported as the mean ± standard deviation of three experimental replicates.

Sample ID	at 0 h		after 24 h	
	Pt conc. measured by ICP-MS (μg L^{-1})	% measured to the actual conc.	Pt conc. measured by ICP-MS (μg L^{-1})	% measured to the actual conc.
in UHPW (without NOM)	1.02 ± 0.03	101.6 ± 3.2	0.88 ± 0.05	88.2 ± 5.1
in MHW (without NOM)	1.03 ± 0.05	103.1 ± 4.8	0.84 ± 0.05	84.4 ± 5.2
NOM 1	0.88 ± 0.06	88.2 ± 5.7	0.90 ± 0.07	90.3 ± 6.7
NOM 2	0.91 ± 0.04	91.4 ± 4.1	0.96 ± 0.06	95.7 ± 6.1
NOM 3	0.71 ± 0.04	71.5 ± 4.0	0.84 ± 0.05	83.5 ± 5.0
NOM 4	0.83 ± 0.08	83.2 ± 7.6	0.92 ± 0.07	92.1 ± 7.1
NOM 5	1.00 ± 0.06	100.3 ± 5.7	1.01 ± 0.04	100.7 ± 3.9
NOM 6	0.77 ± 0.05	76.8 ± 5.0	0.86 ± 0.04	85.5 ± 3.8

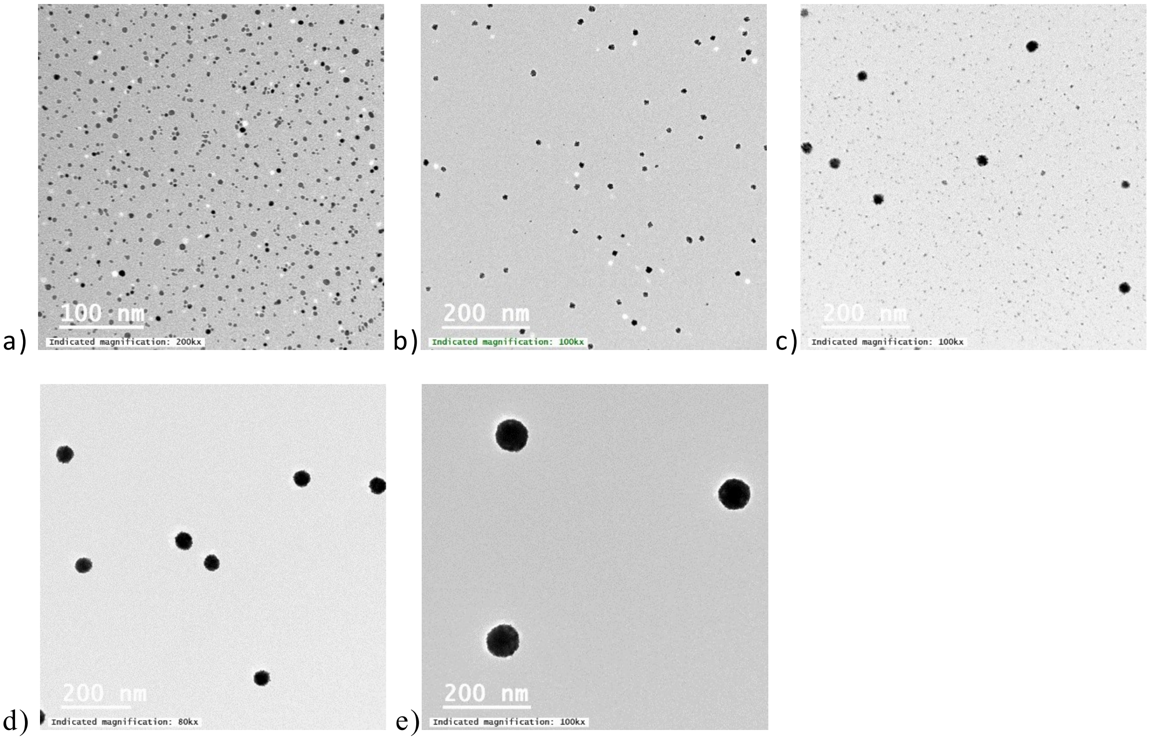

Figure S1. (a-e) Representative transmission electron microscopy (TEM) micrographs of synthesized PVP-PtNPs (PtNP$_{20}$, PtNP$_{30}$, PtNP$_{50}$, PtNP$_{75}$, and PtNP$_{95}$ respectively) prepared by drop deposition method. The scale bar in the TEM micrograph in panel a is 20 nm and in all the other TEM micrographs is 200 nm (b - e).

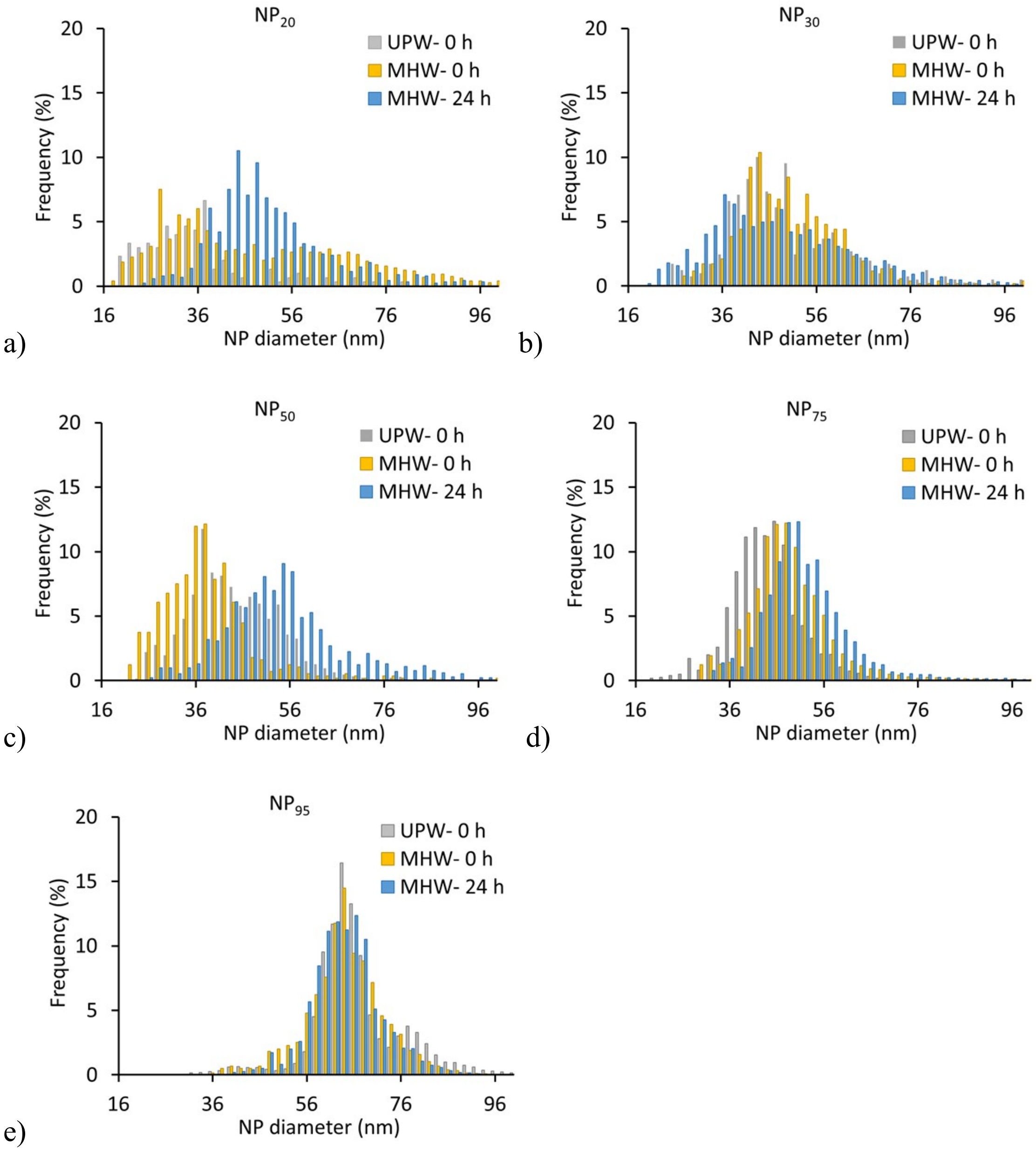

Figure S2. Aggregation behavior of 1 µg L^{-1} (a) NP$_{20}$, (b) NP$_{30}$, (c) NP$_{50}$, (d) NP$_{75}$, and (e) NP$_{95}$ in synthetic moderately hard water (MHW) after 24 h.

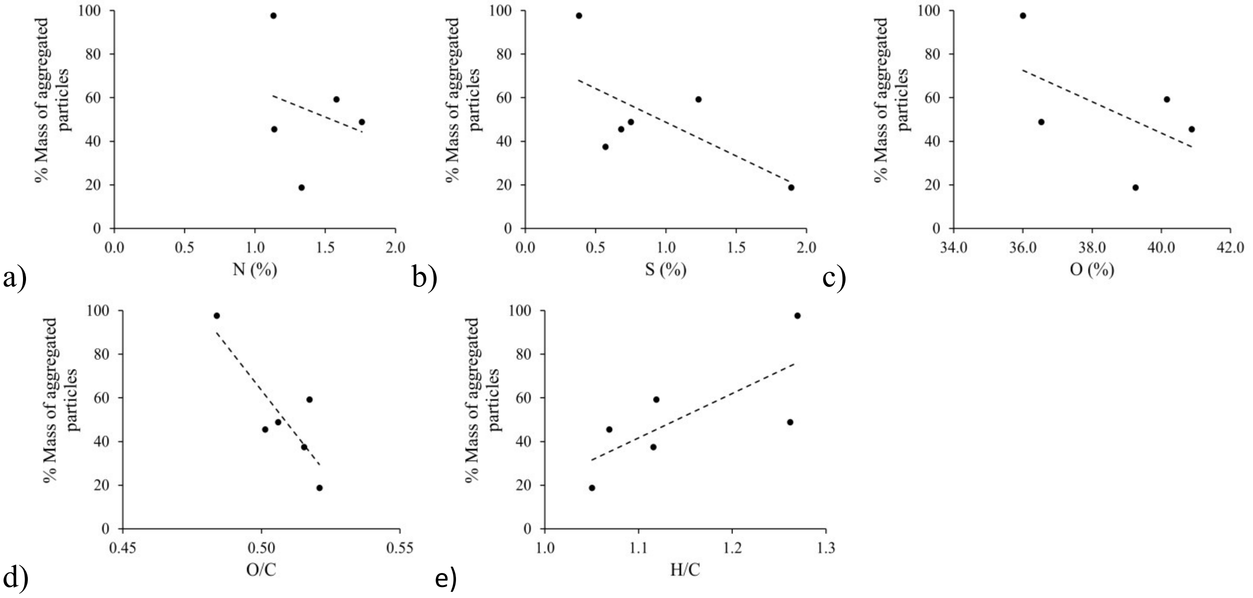

Figure S3. Correlation between % mass of $PtNP_{20}$ undergoing aggregation and NOM elemental composition: (a) N, (b) S, (c) O, (d) O/C, and (e) H/C ratio

Reference

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