Atomic Spectroscopy

Article

On-site Rapid Analysis of Metal Ions and Nanoparticles Based on the Preconcentration and Separation with Stereoscopic Amphiphiles

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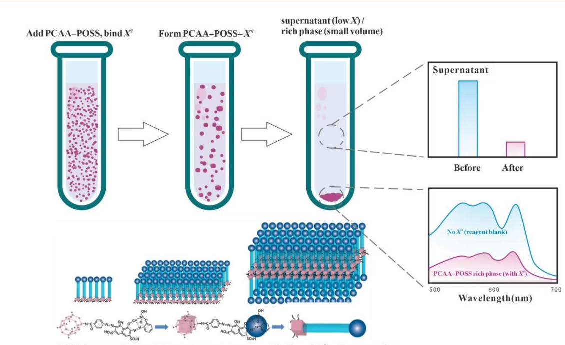
ABSTRACT: A novel on-site analytical approach for the determination of trace metal ions and nanoparticles in environmental water is presented, utilizing a self-developed amphiphilic material that enables rapid enrichment and precipitation of analytes. This material, consisting of a hydrophobic polyhedral oligomeric silsesquioxane (POSS) core covalently linked to functional hydrophilic ligands, spontaneously self-assembles in aqueous solutions to form micron-scale aggregates, thereby facilitating efficient extraction and phase separation without heating, pH adjustment, or auxiliary agents. After enrichment, the precipitate was readily redispersed and subjected to rapid spectrophotometric analysis directly in the field. The method achieved outstanding enrichment factors (up to 495 for metal ions and 475 for nanoparticles), high extraction efficiencies (95–99%), and excellent selectivity for analytes such as Cu^{2+}, Pb^{2+}, Ag nanoparticles, and ZnO nanoparticles. Notably, the detection limits and analytical performance of the on-site spectrophotometric method after preconcentration were significantly improved and comparable to those of laboratory-based atomic absorption spectrometry (AAS). For example, the limit of detection for Cu^{2+} was reduced to 0.63 μg/L (24-fold improvement), for Pb^{2+} to 11.25 μg/L (10-fold improvement), for Ag nanoparticles to 0.056 μg/mL, and for ZnO nanoparticles to 0.065 μg/mL, all with substantial increases in sensitivity after enrichment. The method also demonstrated high precision and accuracy with recoveries ranging from 91% to 108% and relative standard deviations below 5% for real water samples. The results obtained using this approach were in excellent agreement with those obtained using AAS, confirming its reliability. This study established a simple, rapid, and environmentally friendly platform for on-site spectrometric analysis of both metal ions and nanoparticles, providing a practical alternative to conventional laboratory instrumentation for environmental monitoring and analytical chemistry.



INTRODUCTION

Human activities such as mining and industrial production have led to increasingly severe water pollution caused by hazardous chemical compounds. These substances accumulate in plants and animals and enter human tissues via the diet, producing adverse health effects that can be life-threatening. For instance, prolonged heavy metal intake may result in critical dysfunction of human organs[1-3]. Ultrafine nanoparticles, which have recently been the focus of intense study due to the emerging interest in nanotechnology, exhibit intracellular toxicity arising from the production of excess reactive oxygen species[4,5]. A common feature of these substances is that they are highly detrimental, even at trace levels, because they are difficult to remove from human tissues. Therefore, it is extremely important to monitor and remove these substances from water sources that may be consumed by human beings.[6,7]

Among currently available analytical tools, modern instrumentation provides high sensitivity for the determination of trace substances. However, its effectiveness is often obstructed by the matrix effect, meaning that separating a target analyte at an extremely low concentration from a complex matrix is difficult[8-11]. To address this issue, diverse separation and concentration techniques have been proposed, including electrochemical deposition[12], co-precipitation[13], sorption- and membrane-based extractions[14-17], and electrically assisted extractions[18, 19], among others[20]. In particular, cloud point technology (CPT) has recently attracted intensive attention because of its high extraction efficiency and reliability for separating trace-level substances[21,22], as well as its environmental safety, low cost, and improved analytical characteristics compared with other techniques [23-26]. Nonetheless, this method requires several specific factors to be satisfied before separation can be attained, including temperature[27], pressure[28,29], pH environment[30], and the presence of assisting agents[31-33], which vary across systems. Thus, developing a strategy for each specific case of contaminant treatment based on CPE is tedious, time-consuming, and labor-intensive. Moreover, these factors often interfere with one another, posing significant difficulties for successful separation.

There is a growing global demand for analytical methods that enable rapid on-site monitoring of environmental contaminants, especially in scenarios where access to centralized laboratory facilities is limited or immediate decision-making is required. Existing approaches often fall short in portability, operational simplicity, or the ability to simultaneously handle both metal ions and engineered nanoparticles at trace levels. Conventional approaches for metal ion detection typically include ICP-AES, ICP-MS and atomic absorption spectroscopy (AAS). These techniques exhibit low detection limits and allow the simultaneous measurement of multiple ions. Nevertheless, they are generally requiring large-scale, costly equipment, specialized operators, and complex pretreatment steps. As a result, portability, cost-effectiveness, and high integration have become key focuses in the field of metal ion sensing. Optical sensors, which demonstrate high sensitivity and selectivity for heavy metal ion detection, have been used to construct portable sensors.

Here, we describe a simple and efficient general method for the direct separation and concentration of trace-level substances. This was achieved using a molecularly designed amphiphilic compound consisting of a hydrophobic polyhedral oligomeric silsesquioxane (POSS)[34] head covalently linked to a functional hydrophilic complexing agent tail. The hydrophilic tail has abundant active groups that capture specific pollutants in water[35-36]. Due to their amphiphilic nature and rigid hydrophobic head with extraordinary dimensional stability, these POSS derivatives self-assemble in aqueous systems to form stable large-scale suspensions and allow facile precipitation without the need for elevated temperature, pressure control or the addition of a "salting out" agent in acidic, alkaline or neutral media. By immobilizing the hydrophobic POSS head on different complexing agents, target analytes can be readily customized. These features provide a large concentration factor, high extraction efficiency, and excellent selectivity of POSS derivatives for trace-level substances. Furthermore, we present a novel, field-deployable analytical platform based on self-developed stereoscopic amphiphilic materials that enables direct and efficient preconcentration and separation of analytes such as Cu^{2+}, Pb^{2+}, Ag nanoparticles, and ZnO nanoparticles from complex aqueous matrices. The amphiphilic POSS derivatives employed in this study not only simplify the enrichment process but also allow rapid on-site spectrophotometric detection, eliminating the need for sophisticated instrumentation or harsh operational conditions. Importantly, the analytical performance, including the detection limits and sensitivity achieved by this method, is comparable to that of laboratory-based AAS, thereby bridging the gap between field analysis and high-end laboratory techniques. This study provides a practical and versatile solution for environmental monitoring and offers significant advantages for point-of-need applications in analytical chemistry.

EXPERIMENTAL

Synthesis of modified POSS derivatives. Aminopropylisooctyl polyhedral oligomeric silsesquioxane (AP-POSS) and octaammonium-POSS (OA-POSS) were purchased from Hybrid Plastic Co. All other reagents were purchased from Aladdin Reagent Co. Shanghai, China. The modification of POSS was achieved by a simple amidation reaction between PCAC, PCAA, TA, and SA modifier, and amino-functionalized POSS (NH_2-POSS). A magnetically stirred solution of 0.001 mol modifier in 100 mL of ethanol was prepared, to which a solution containing NH_2-POSS (0.87 g), DCC (0.21 g), DMAP (0.0051 g), and chloroform (30 mL) was added. The reaction mixture was stirred vigorously for 10 h at room temperature. After air-drying, the crude product was washed with distilled water until the filtrate was colorless, and the corresponding POSS derivatives were obtained under an infrared lamp.

General methods. The test solution containing trace copper and lead ions, as well as Ag and ZnO NPs, was placed in a 40 mL centrifuge tube. The following reagents were sequentially added, with mixing between additions: buffer solution, POSS derivatives solution in absolute ethanol, diluted to the mark with deionized water. The separation of the two phases was achieved by centrifugation for 25 min at 10000 r/min. The aqueous supernatant was discarded. The separated POSS-rich phase was diluted with absolute ethanol and analyzed using spectrophotometry and flame atomic absorption spectrometry (FAAS). The morphologies of the composites were analyzed by Transmission electron microscopy

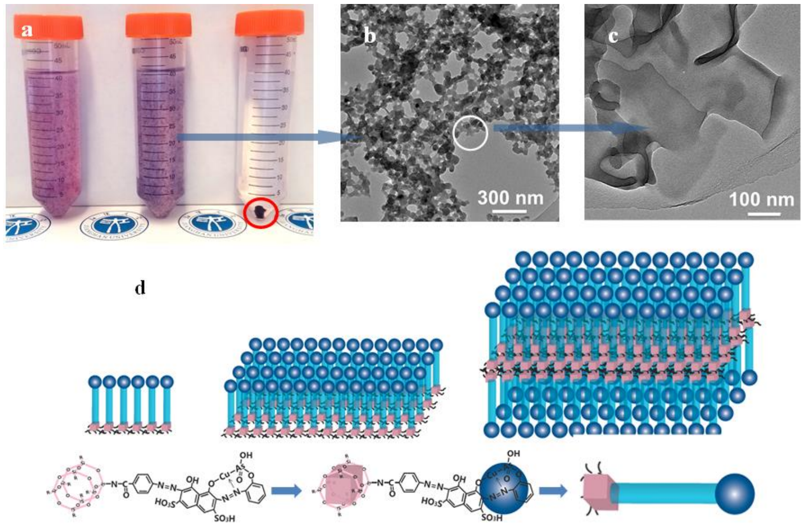


Fig. 1 Self-assembly of POSS derivate in aqueous solution: a, from left to right: the PCAA-POSS aqueous solution, the PCAA-POSS aqueous solution containing Cu^{2+} and centrifuged solution of PCAA- POSS; b and c, the TEM of PCAA-POSS aqueous solution; d, schematic illustration of modified POSS self-assembly in solution.

(TEM) (JEOL 2000FX) operated at an acceleration voltage of ~120 kV.

Photometric determination. A precise volume of the 100 µg/mL Cu standard solution was transfer into a 10 mL colorimetric tube. 3.0 mL of 0.1% PCAA-POSS ethanol solution and 3.0 mL of pH 4.5 HAc-NaAC solution in sequence was added, then top up to 10 mL with 100% ethanol. A 1.0cm cuvette was used to measure the absorbance wavelength at 612 nm with a blank reagent as the reference.

RESULTS AND DISCUSSION

Preparation and self-assembly of modified POSS molecules. Phase separation, which involves physical changes in a solution, is crucial for modern extraction systems. Conventional surfactants, consisting of hydrophilic head groups and hydrophobic segments such as hydrocarbon chains, typically self-assemble into small (submicron-scale) micellar structures in solution. These micelles cannot spontaneously separate from the aqueous phase because they are unable to grow further or form large coacervates and interconnected networks. Achieving phase separation and analyte concentration through large-size self-assembly in these systems often requires external stimuli, such as changes in temperature, pH, or pressure, or additives, making the extraction process tedious and energy-consuming.

In this study, a series of water-insoluble amphiphilic molecules was designed and synthesized using cubic POSS groups as hydrophobic units covalently linked to hydrophilic functionalities. Unlike conventional rod-like water-soluble surfactants, POSS derivatives spontaneously form large molecular clusters because of their bulky, stiff cubic cage-like heads and dumbbell-like molecular structures. This facilitates large-scale assembly (micron scale, approximately 100,000 times the size of POSS nanomolecules), resulting in stable suspensions in aqueous media that efficiently absorb or react with analytes and readily precipitated without external stimuli. Functionalization of POSS derivatives allows customization toward specific target analytes, enabling direct phase separation without heating or the addition of pH or electrolyte additives and avoiding interference among chelation, adsorption, and isoelectric point conditions.

Upon the addition of modified POSS derivatives (*e.g.*, PCAA-POSS) into aqueous solution at any temperature between 0 and 100 °C, the solution turns turbid instantly due to the formation of abundant, loosely structured floccules that remained stably suspended. In the presence of analytes such as heavy metal ions or nanoparticles, these floccules remained physically stable, and the suspensions exhibited different responses depending on reactions with the functional groups of the POSS derivatives, including color changes. After centrifugation, the suspended substances were rapidly enriched at the bottom of the solution, forming microvolume precipitates that were easily collected. The visual appearance, colorimetric response, and formation of precipitates in the PCAA-POSS system are shown in Fig. 1a. The large lamellar structures formed by the self-assembly of the POSS derivatives provided open access to guest analytes, enabling facile precipitation and phase separation. TEM images revealed that these aggregates were loosely stacked and interconnected multilamellar structures with micron-scale lateral sizes (Fig. 1b). The particle size distribution further confirmed the formation of micelles up to 500 μm in size (Fig. 1c). The self-assembly process is illustrated schematically in Fig. 1d. This unique self-assembly behavior established the foundation for efficient sample pretreatment prior to atomic spectrometry analysis. The surface chemical bonding states of PCAA, POSS, and PCAA-POSS were investigated using FT-IR (Fig. S1), indicating the successful preparation of PCAA-POSS.

Separation and concentration of Cu^{2+} with PCAA-POSS. Most ligands or chelates with high selectivity and sensitivity toward metal ions are unsuitable for separating and extracting trace substances from aqueous solutions because of their high-water solubility. However, when specific hydrophilic ligands are covalently linked to hydrophobic POSS molecules, they can directly extract metal ions. For instance, divalent copper ions, which strongly interact with arsenazo groups, were efficiently captured using PCAA-POSS bearing arsenazo functionalities.

PCAA-POSS is much less hydrophilic than conventional water-soluble surfactants and was only sparingly soluble in water, although it dispersed well in ethanol. When mixed with water, PCAA-POSS quickly self-assembled into lamellar aggregates, achieving spontaneous phase separation. TEM images confirmed the formation of these aggregates both before and after Cu^{2+} addition, (Fig. 2a and b), with no significant change in morphology. Upon the introduction of Cu^{2+} ions, the aggregates exhibited an immediate color change from red to blue due to the chelation, which could be quantitatively monitored by visible absorbance spectra. PCAA-POSS and the resulting Cu-PCAA-POSS complexes were easily separated from the aqueous phase by centrifugation.

Extraction efficiency and enrichment factor were determined spectrophotometrically using FAAS as the reference method. The effects of various parameters, such as pH, medium, PCAA-POSS concentration, and equilibration time, were investigated (Fig. S2). Extraction efficiency reached a plateau after 5 min. The amphiphilic POSS derivatives employed in this study not only simplify the enrichment process, but also permit rapid on-site spectrophotometric detection, eliminating the need for sophisticated instrumentation or harsh operational conditions. Under optimal conditions, the extraction yield of Cu^{2+} ions were 99.0% in the concentration range of 0.002 to 0.50 μg/mL. The enrichment factor was determined to be 495. After preconcentration, the LOD for Cu^{2+} by spectrophotometry was reduced to 0.63 μg/L, representing a 24-fold sensitivity improvement compared with direct measurement. The method exhibited high precision, with a relative standard deviation (RSD) of 3.7% for five replicate measurements at 0.050 μg/mL Cu^{2+}. The linear regression calibration equation was $A = 4.56C + 0.024$ (C: μg/mL) with a correlation coefficient of 0.9976. High selectivity was observed, with tolerance limits for common interfering ions listed in Table 1.

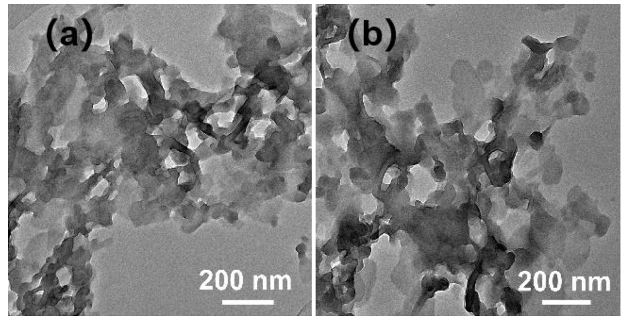


Fig. 2(a) TEM of PCAA-POSS before centrifugation; (b)TEM of Cu-PCAA-POSS before centrifugation.

Table 1. The effect of potential coexisting ions on the determination of 0.050 g/mL of copper

Coexisting ions	Tolerance limits (μg)	Coexisting ions	Tolerance limits (μg)
Na^+	10000	Zn^{2+}	40
K^+	10000	Cd^{2+}	10
Mg^{2+}	100	Mn^{2+}	50
Pb^{2+}	700	Sr^{2+}	50
Ba^{2+}	10	Cr^{3+}	50
NO_3^-	10000	SO_4^{2-}	10000
Fe^{3+}	0.2	Al^{3+}	0.2

Table 2. Cu contents found in Sanjiao lake water samples (n=5)

Sample	Found/(μg/40mL)		Cu added/μg	Total (μg/40mL)	Recovery (%)	RSD (%)
	FAAS	This method				
1	2.25	2.05	5	7.46	108%	3.8%
			10	11.95	99%	3.1%
2	6.04	5.85	5	10.56	94%	1.1%
			10	15.40	96%	2.5%

Application to environmental water samples demonstrated excellent agreement with FAAS results, with recoveries of 94%-108% and RSDs of 1.1-3.8% (Table 2). These results confirmed that the present method was accurate, reliable, and provided an efficient pretreatment platform for atomic spectrometric analysis of trace Cu^{2+} in environmental samples.

Separation and concentration of Pb^{2+} with PCAC-POSS. The extraction temperature is a key parameter influencing efficiency, as heating can disrupt heat-sensitive coordination reactions of complexing agents with metal ions. Some hydrophilic chelate reagents, such as PCAC, produce highly sensitive color reactions with Pb^{2+} at room temperature but are unsuitable for extraction at elevated temperatures. In this study, a high-efficiency method for Pb^{2+} separation was developed using PCAC-POSS, which enabled coordination-fading reactions at room temperature without heating.

Upon the addition of 3 mg of PCAC-POSS to a 40 mL solution,

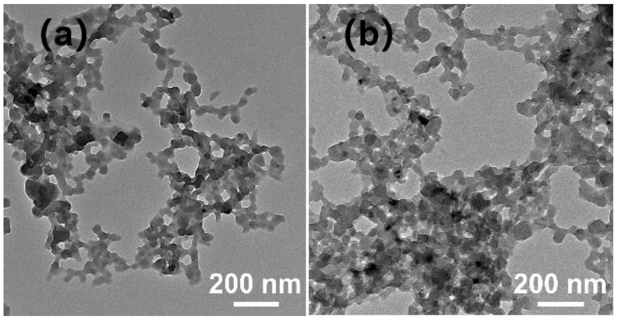


Fig. 3 TEM images of the self-assembled PCAC-POSS (a) before and (b) after introducing Pb ions.

Table 3. The effect of potential coexisting ions on the determination of 0.50 g/mL of lead

Coexisting ions	Tolerance limits (μg)	Coexisting ions	Tolerance limits (μg)
Na^{+}	10000	Zn^{2+}	500
K^{+}	10000	Cd^{2+}	350
Mg^{2+}	15	Al^{3+}	20
Cl^{-}	10000	Cr^{3+}	200
NO_3^{-}	10000	SO_4^{2-}	10000
Fe^{3+}	0.2	Cu^{2+}	0.2
Ba^{2+}	0.2	Sr^{2+}	0.2

Table 4. Pb contents found in Sanjiao lake water samples (n=5)

Sample	Found/(μg/40mL)		Pb added/μg	Total (μg/40mL)	Recovery (%)	RSD (%)
	FAAS	This method				
1	2.95	2.87	5	7.91	101%	4.1%
			10	12.71	98%	3.3%
2	5.38	5.21	5	10.48	105%	4.8%
			10	14.34	91%	2.6%

self-assembly into large-scale aggregates was observed. TEM images indicated that the morphology of PCAC-POSS remains similar before and after Pb^{2+} addition (Fig 3a and b). A high enrichment factor of 495 was obtained. Extraction yields exceeded 99.0% for Pb^{2+} concentrations ranging from 0.002 μg/mL to 0.625 μg/mL. The selectivity for PCAC was retained or improved after grafting onto POSS (Table 3). The method exhibited good precision, with an RSD of 4.2% for five replicate measurements at 20 μg/40 mL Pb^{2+}. The LOD for Pb^{2+} was significantly decreased to 11.25 μg/L, which is ten times lower than without separation.

Application to environmental water samples yielded recoveries of 91%–105% with RSDs of 2.6-4.1% (Table 4). The Pb^{2+} concentrations determined by this method were in excellent agreement with FAAS, confirming its suitability for atomic spectrometric analysis of trace Pb^{2+} in real water samples.

Separation, concentration, and recovery of Ag and ZnO nanoparticles. The potential toxicity of nanoparticles necessitates the development of effective separation, concentration, and recovery techniques. Existing surfactant-based preconcentration methods require heating and electrolytes, making them time-consuming and complex. In this study, two POSS derivatives (TA-POSS and SA-POSS) were synthesized by combining NH_2-POSS with short-chain 2,3-dihydroxysuccinic acid (TA) and 2-hydroxy-5-sulfobenzoic acid (SA), respectively, to facilitate encapsulation and aggregation.

TEM images of TA-POSS and SA-POSS in aqueous solution are shown in Fig. 4a and 4b (SA-POSS with Ag NPs) and Fig. 5a and 5b (TA-POSS with ZnO NPs). Both derivatives formed large-scale planar multilayer structures through a self-assembly process, similar to PCAA- and PCAC-POSS. After the addition of Ag or ZnO NPs, small particle agglomerations (highlighted in white circles) were observed in the TEM images, which were absent in unchelated POSS. The diameter of Ag NPs was approximately 5-20 nm, whereas ZnO measured 15-20 nm. The absolute values of the zeta potentials of SA-POSS and TA-POSS were considerably smaller than those of PCAA- or PCAC-POSS, facilitating self-assembly and separation. Extraction efficiencies reached 95% for both Ag NPs (0.002-0.3 μg/mL) and ZnO NPs (0.002-0.3 μg/mL), with enrichment factors of 475 for each (Tables 5). After preconcentration, the LODs were reduced to 0.056 μg/mL for Ag NPs and 0.065 μg/mL for ZnO NPs, representing substantial improvements in sensitivity over direct analysis. The results were consistent with FAAS data, confirming the applicability of this method for atomic spectrometric analysis of nanoparticles.

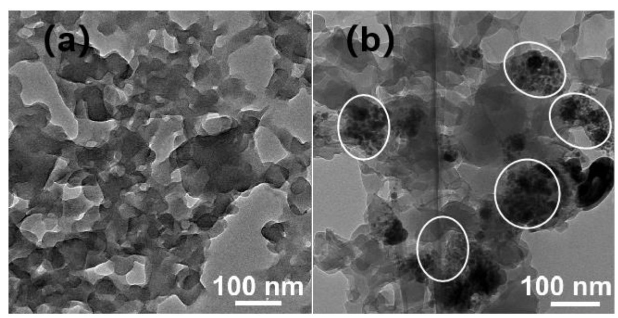


Fig. 4 TEM images of the self-assembled SA-POSS (a) and introducing Ag NPs ions (b).

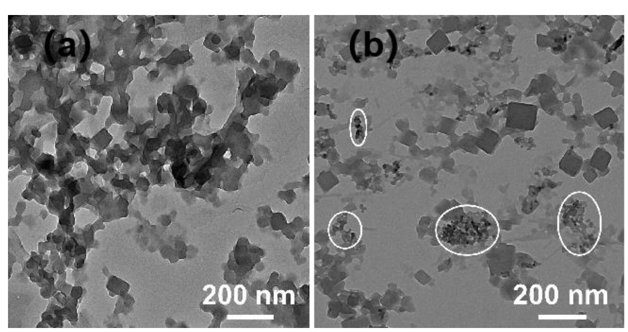
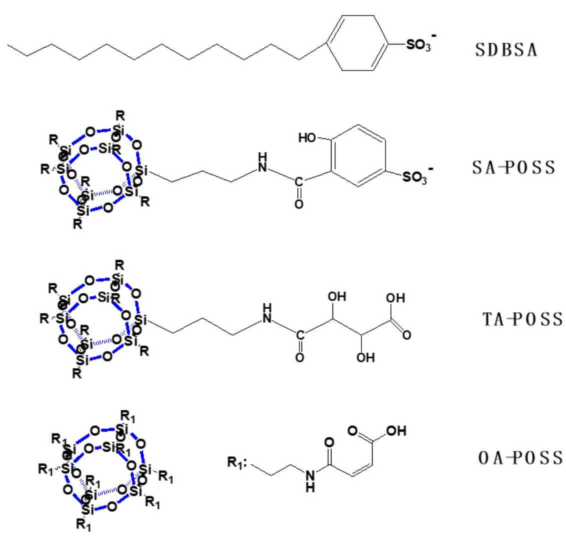


Fig. 5 TEM images of the self-assembled TA-POSS (a) and introducing ZnO NPs ions (b).

Table 5. Extraction efficiency of Ag NPs by SA-POSS and ZnO NPs by TA-POSS

Ag NPs by SA-POSS					
Concentration /(μg/mL)	0.056	0.11	0.16	0.22	0.34
Extraction efficiency /(%)	96	94	93	95	92
ZnO NPs by TA-POSS					
Concentration /(μg/mL)	0.065	0.13	0.19	0.26	0.35
Extraction efficiency /(%)	99	97	96	94	90



Scheme 1. Molecular structures of SDBSA, SA-POSS, TA-POSS, and OA-POSS molecules

All the prepared POSS derivatives with amphiphilic structures exhibit distinctive water insolubility; therefore, we investigated the solubility of molecules with structures analogous to those of the POSS derivatives. For example, sodium dodecylbenzene sulfonate[37] (SDBSA; Scheme. 1), a commonly used anionic surfactant, comprises a hydrophobic dodecyl hydrocarbon chain linked to a hydrophilic benzene sulfonate group. The hydrophilic segment of SDBSA is analogous to that of SA-POSS. However, unlike SA-POSS, the SDBSA is water soluble, and does not phase-separate spontaneously from aqueous solution. The main difference between the two molecular structures lies in their hydrophobic groups-namely, the linear hydrocarbon chain versus the cage-like POSS. Although both are hydrophobic, the linear chain with flexible carbon-carbon bonds tends to fold back and curl in polar aqueous solutions, similar to other linear soft siloxane-based surfactants[38], whereas the cage POSS is rigid and resistant to deformation. As a result, the rigid amphiphilic SA-POSS molecules form large-scale supramolecular aggregates with lamellar structures. These structural advantages directly contribute to the high extraction efficiency and compatibility of the method with atomic spectrometric detection.

The computer-simulated packing factor (g), which is typically used to predict the morphology of assembled aggregates of hydrocarbon chain-containing amphiphiles, was applied to investigate the structure of the supramolecular aggregates of the POSS derivatives.

$$g = \frac{v}{a_0 l_c} \tag{1}$$

Here, V is the volume of the hydrophobic chain, a_0 is the mean cross-sectional area of the head groups in the aggregate, and lc is the length of the fully extended chains.

The calculated value (g = 0.23) for SA-POSS corresponds to a cylindrical micelle structure rather than a lamellar structure with a higher packing factor (g ~1.0), indicating that the rigid three-dimensional group differs substantially from conventional hydrocarbon chains. POSS derivatives, including TA-POSS, PCAA-POSS, and PCAC-POSS, all adopt a lamellar morphology owing to the steric effects of the POSS structure (Scheme 1). In contrast, octamaleamic acid POSS (OA-POSS; Scheme 1), which is fully functionalized with maleamic acid groups despite containing a cage POSS, is water soluble because of the presence of hydrophilic functional groups at all eight symmetric vertices. These results imply that the suspension behavior of POSS derivatives in aqueous systems originates from the hydrophobicity, steric effects, and structural rigidity of the POSS head.

The efficient integration of POSS-based extraction with deployable spectrophotometric detection offers distinct advantages for trace metal ion and nanoparticle analyses. Owing to the unique amphiphilic design, self-assembly behavior, and chemical tunability of POSS derivatives, this method enables high enrichment factors, exceptional selectivity, and compatibility with a wide spectrum of analytes, even in complex environmental samples. Compared with conventional extraction strategies, POSS-based materials achieve rapid phase separation and analyte concentration under mild ambient conditions without the need for auxiliary reagents or stringent controls, thereby simplifying on-site procedures. Notably, this approach allows direct, rapid spectrophotometric measurements in the field, providing sensitive, reliable, and immediate results that approach the performance of laboratory-based techniques such as FAAS. This universal platform not only facilitates routine and emergency environmental monitoring but also expands the analytical toolkit for atomic spectrometry, bridging laboratory and field applications, while offering significant benefits in efficiency, environmental sustainability, and analytical versatility.

CONCLUSION

Based on the results of this study, a novel and efficient approach for the separation and preconcentration of trace metal ions and nanoparticles from aqueous solutions was established using stereoscopic amphiphilic nanohybrids constructed from polyhedral oligomeric silsesquioxane (POSS) cores covalently linked to functional hydrophilic ligands. These amphiphilic POSS derivatives spontaneously self-assembled into micron-scale aggregates, enabling rapid and effective extraction and phase separation under mild conditions without the need for heating, pH adjustment, or auxiliary agents. This method demonstrated high extraction efficiencies (up to 99% for metal ions and 95% for nanoparticles), excellent selectivity, and remarkable enrichment factors (495 for metal ions and 475 for nanoparticles). Quantitative spectrophotometric analysis before and after enrichment revealed significantly improved detection limits, with the LOD for Cu^{2+} reduced to 0.63 μg/L and for Pb^{2+} to 11.25 μg/L, representing substantial enhancements in sensitivity. This approach also showed strong reproducibility and accuracy, with recoveries ranging from 91% to 108% and relative standard deviations below 5% in environmental water samples. The reliability of the results was further confirmed by their excellent agreement with FAAS. Overall, this POSS-based amphiphilic extraction platform provides a simple, rapid, and environmentally friendly solution for sample pretreatment and enrichment, greatly facilitating the atomic spectrometric analysis of trace contaminants and offering significant potential for practical applications in environmental monitoring and analytical chemistry.

ASSOCIATED CONTENT

The supporting information (Figs. S1−S2) is available at https://www.at-spectrosc.com.

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Notes

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The authors declare no competing financial interest.

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