



Facile purification of ultrathin Au nanowires: A solvent exclusion method

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ABSTRACT Ultrathin Au nanowires are always synthesized with 2 nm Au nanoparticles as the by-product, and their separation has been a long-standing problem. In this work, we show that high-purity (>99%) separation can be achieved using the solvent exclusion method: Adding hexane would cause water to be excluded from the initial water-THF mixture. The excluded water preferentially nucleates on the oleylamine-bilayer of the nanowires, transferring them into the water phase. Careful control experiments were conducted to establish the purification mechanism. The facile method can be generally applied to purify ultrathin Au nanowires with short lengths (470 and 130 nm), which has not been realized using the conventional methods.

Keywords: ultrathin Au nanowire, phase separation, separation, purification, solvent exclusion

INTRODUCTION

Ultrathin gold nanowires (UAuNWs) with a diameter of 2 nm are a special class of nanowires. They possess remarkable characteristics, including ultra-high specific surface area, chemical inertness, and excellent optical, electrical, thermal, and mechanical properties [1–3]. These attributes render them promising applications in catalysis [4,5], energy storage [6], sensing [7,8], flexible electronic devices [9,10], surface-enhanced Raman spectroscopy [11,12], and biomedicine [13].

To date, there are three methods for synthesizing UAuNWs. The most widely employed method is the oleylamine (OAm) assisted synthesis in non-polar solvents, where OAm plays multiple roles as a co-solvent, reducing agent, and capping agent [11,14–19]. Using this method, UAuNWs of varying lengths have been synthesized [20–23]. A less prevalent method employs cetyltrimethylammonium bromide (CTAB) [24] or benzyldimethylcetylammmonium chloride (BDAC) [25,26] as surfactants in aqueous solution, but the length control has yet to be established. Additionally, a special long-chain amidoamine derivative could produce UAuNWs in either polar or non-polar solvents [27–29]. It is important to note that all these methods yield a significant proportion of Au nanoparticles (AuNPs) as undesired by-products.

Without a pure form of the UAuNWs, there is always an ambiguity in establishing the structure-property correlation in applications, for example, in terms of assessing the relative contributions from the nanowires, the side-effects from the AuNPs or the comparison among the UAuNWs of different

lengths.

To obtain high-purity UAuNWs and enhance their performance, efforts have been made to improve the synthesis. For example, the use of hexane as a solvent and tri-isopropylsilane as a reducing agent has been explored to improve the yield of UAuNWs [11,30]. Moreover, a special form of 2-nm Au seeds were introduced into the growth solution to reduce the inhomogeneity of nucleation [5].

Purification methods are a vital alternative to synthetic improvements. The purification of nanomaterials, like the purification of organic compounds, is the necessary step towards multi-step synthesis and sophisticated applications. At present, there are only a few methods known for isolating UAuNWs, all of which are derived from density gradient ultracentrifugation. It could separate UAuNWs in multiple layers of density gradients [31–33], but the method only works for long UAuNWs (2–4 μm), and complete removal of small (2 nm) AuNPs remains a challenge.

Despite many attempts, large-area images of high-purity UAuNWs are extremely rare in the literature [31,33–35]. Scalable synthesis and purification are additional challenges. Finally, the existing methods cannot be applied to purify short UAuNWs (<1 μm). This field is in great need of out-of-box explorations.

Here, we developed a solvent exclusion method to separate UAuNWs from AuNPs, achieving a nearly perfect separation efficiency. In contrast to the traditional methods relying on high-speed centrifugation, the solvent exclusion method is facile and easily scalable. It can be used to purify short UAuNWs (470 and 130 nm), which is unattainable using the conventional methods. Given the new method, we focus our study on the separation mechanism by carrying out extensive control experiments. We believe that the insights are essential for improving purification methods.

EXPERIMENTAL SECTION

Chemicals

All chemical reagents were used as purchased without further purification. Hydrogen tetrachloroaurate(III) hydrate ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$, 99.9%, and metal basis Au 49%) and tri-isopropylsilane (TIPS, 98%) were purchased from Sigma-Aldrich. Oleylamine (80%–90%) was purchased from Acros. Ethanol (EtOH), toluene, xylene, methanol, acetonitrile, *n*-butanol, *tert*-butanol, dichloromethane, *N,N*-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), and ethyl ether were purchased from Sinopharm Chemical Reagent. Tetrahydrofuran (THF),

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n-hexane, cyclohexane, *n*-octane, and cyclooctane were purchased from Energy Chemical. Ultrapure deionized water (18.2 M Ω /cm) was used.

Measurements and characterizations

Transmission electron microscopy (TEM) images were collected on a Hitachi HT7800 (Japan) operated at 100 kV. Gas chromatography-mass spectrometry was collected on a Thermo-Fisher (USA).

Preparation of ultrathin Au nanowires

UAuNWs (length = 130, 470 nm and >1 μ m) were synthesized following established procedures described in Ref. [22]. The synthesis process involved adding 30 mg of HAuCl₄ into a vial, then introducing 1 mL of OAm, and subsequently adding 25 mL of THF as the solvent, along with 1.5 mL of triisopropylsilane as the reducing agent. The mixture was left to react at room temperature for 24 h, resulting in UAuNWs with lengths exceeding 1 μ m.

To synthesize UAuNWs of different lengths, the addition of water was necessary. For the 470-nm UAuNWs, 1 mL of water was added. To produce the 130-nm UAuNWs, 2 mL of water was added while keeping other conditions constant. These variations in water addition allowed for the controlled synthesis of UAuNWs with different lengths.

The solvent exclusion method to separate ultrathin Au nanowires and nanoparticles

For the 470-nm UAuNWs, 2 mL of hexane was directly added to 0.5 mL of the as-synthesized UAuNWs solution. Then, hand-shaking was used to ensure thorough mixing and small black droplets appeared in the mixture. After allowing the solution to stand undisturbed for 6 h, clear phase separation occurred, forming two distinct layers. The bottom layer exhibited a black hue, indicating the presence of UAuNWs free of AuNPs, whereas the upper layer appeared light brown, exclusively comprising AuNPs. To characterize the products of the upper and bottom layers, 0.5 mL of the upper layer liquid was transferred into a centrifuge tube, and 1 mL ethanol was added (ethanol/upper layer liquid >2) [36]. Concurrently, 0.5 mL ethanol was introduced into the bottom layer liquid (estimated 30 μ L, or diluted to 0.5 mL) to induce precipitation. The mixtures were subsequently centrifuged at 8000 r min⁻¹ for 5 min. The supernatant was discarded, and the resulting precipitates (estimated 10 μ L) were then dispersed in 1 mL THF for sample preparation.

For the 130-nm UAuNWs, 4 mL of hexane was directly added to 0.5 mL of the as-synthesized UAuNWs solution while maintaining other conditions constant.

Regarding the UAuNWs (>1 μ m), initially, 20 μ L of water was added to 0.5 mL of the as-synthesized UAuNWs solution, followed by the addition of 1 mL of hexane, while keeping other conditions constant.

RESULTS AND DISCUSSION

UAuNWs with different lengths were synthesized by following a reported method [22], where OAm acted as the capping agent, THF as the solvent, and tri-isopropylsilane as the reducing agent for HAuCl₄. A varying amount of water was added to modulate the length of UAuNWs, followed by the reaction at room temperature for 24 h. The final product exhibited a dark brown

color (Fig. 1a and Fig. S1a).

With no additional water, the product UAuNWs were more than 1 μ m in length; when the volume fraction of water was 3.85%, UAuNWs with an average length of 470 nm were obtained; at water fraction of 7.7%, the product UAuNWs were even shorter with a length of 130 nm. It is important to note that the products in all of the above experiments contained a substantial amount of small AuNPs (Fig. 1e and Fig. S1). No exception was found in multiple rounds of experiments.

Given the above-mentioned challenges in separating short UAuNWs, we use the 470-nm UAuNWs as a model for the following discussion on the separation method. To assess the initial state of the as-synthesized products, excess ethanol was directly added to cause precipitation, which was then isolated by centrifugation. The resulting supernatant was colorless, indicating precipitation of all metal products. The precipitate was then re-dispersed in THF and used for sample preparation. A survey of the resulting TEM images (Fig. 1e and Fig. S2) shows that the small AuNPs (2 nm diameter; 91% out of the 807 AuNPs/UAuNWs) greatly outnumber the UAuNWs (2 nm diameter; 466 \pm 59 nm in length), though the latter is much longer. These AuNPs packed orderly among the parallel UAuNWs, likely a result of self-ordering among the weakly interacting NPs/NWs during the drying process [21,22,30,37,38].

A series of experiments were conducted by adjusting the centrifugation velocity and amount of ethanol. The isolated UAuNWs always contained a significant amount of AuNPs (Figs S3, S4), highlighting the challenges in differential centrifugation. Indeed, all of the UAuNWs reported in the literature contained similar by-product AuNPs (except in small-area images, Figs S5, S6) [2,23,30–35,37,38], consistent with our observations.

As shown in Fig. 1g, high-purity UAuNWs could be obtained by our solvent exclusion method: To reduce the solvent polarity and induce phase separation, 2 mL of *n*-hexane was added to 0.5 mL of the as-synthesized UAuNWs solution. Tiny black droplets appeared in about 30 s, and they eventually merged into a black solution at the bottom (Fig. 1a–d). To collect all products and remove excess OAm, excess ethanol (ethanol/the corresponding layer liquid > 2) was used to cause precipitation in both the upper and bottom layers. The resulting TEM images show that the separation was extremely clean, with the upper layer containing mostly AuNPs (>99%, Fig. 1f), and the bottom layer containing mostly UAuNWs (>99%, Fig. 1g). More specifically, in our survey over 10 million AuNPs (approximately 200,000 AuNPs per image; multiple experiments; Fig. S7), only three UAuNWs were observed. Similarly, in our survey of over 6000 UAuNWs (200+ per image, Fig. S8), only 45 AuNPs were found. Direct sample preparations without ethanol precipitation gave sparser AuNPs, but the results were the same (Fig. S9).

After extensive experiments, we realized that the isolation of UAuNWs is always associated with the process of phase separation, and the separation efficiency depends on the solvent ratio. The amount of hexane was varied while keeping the as-synthesized UAuNWs solution constant at 0.5 mL. At low hexane amounts (<30%; final volume ratio; same below), the solution stayed a single phase (Fig. 2a). At 38%, phase separation occurred (Fig. 2b) and pure UAuNWs were found in the black bottom layer (Fig. S10b), but residual UAuNWs were still found in the upper layer (Fig. 2h). Increasing the hexane content led to a progressive decrease of UAuNWs percentage in the upper

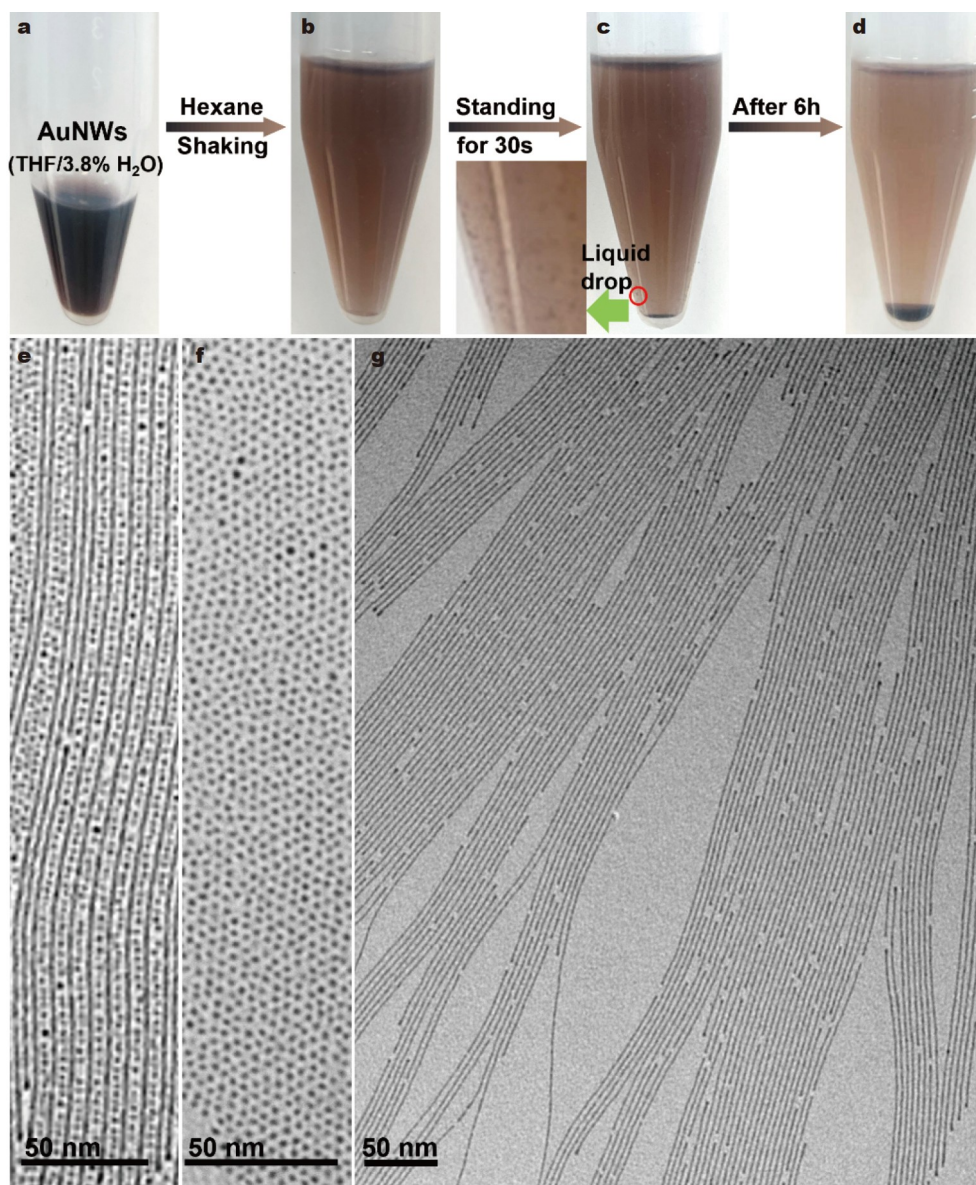


Figure 1 (a–d) Photographs illustrating the solvent exclusion process. TEM images of (e) the unpurified products, (f) AuNPs in the upper layer after solvent exclusion, and (g) UAuNWs in the bottom layer.

layer (Fig. 2i and j), indicating improved separation efficiency. When the hexane ratio reached 80%–90%, UAuNWs disappeared in the upper layer (Fig. 1c and Fig. S11b). Further increase in the hexane ratio led to compromised separation efficiency (Fig. 2g and Fig. S11c), likely due to the increased solubilization of water in the larger hexane domain. Notably, while the separation efficiencies vary, so long as the bottom layer of the solution displayed a black color, the obtained UAuNWs were always free of AuNPs (Fig. S10).

To investigate the phase separation, a blank experiment was conducted: a THF solution that contained a 3.85% volume fraction of water was prepared and mixed with an equal volume of hexane. After the mixture settled down, clear phase separation was observed (Fig. S12a), indicating that the solutes of the UAuNWs solution, namely the UAuNWs and AuNPs, are not the dominant factors. In particular, the amphiphilic molecule OAm is commonly expected to stabilize interfaces, but it is clearly not indispensable. Instead, phase separation depends on

the inherent physical properties of the three solvents. As shown in Fig. 3a, the ternary liquid-liquid phase diagram of water-THF-hexane demonstrates the presence of a non-miscible binary pair, that is, water and hexane [39].

Gas chromatography-mass spectrometry characterization (Fig. S12) was employed to identify the constituents within the phases. The upper layer consisted of hexane and THF, whereas the bottom layer comprised water and THF, with a roughly water/THF = 2:1 ratio. While water and hexane are immiscible, THF can dissolve in both phases and dramatically reduces the rate of phase separation. The order of mixing ensures that the three solvents form a homogeneous solution before the water phase is gradually excluded as tiny droplets.

Conversely, the purification of UAuNWs does not occur in the absence of phase separation. The prerequisites for phase separation involve the presence of water and a bad solvent to exclude water (Table 1). The 470-nm UAuNWs were firstly centrifuged to remove water and then re-dispersed in THF and

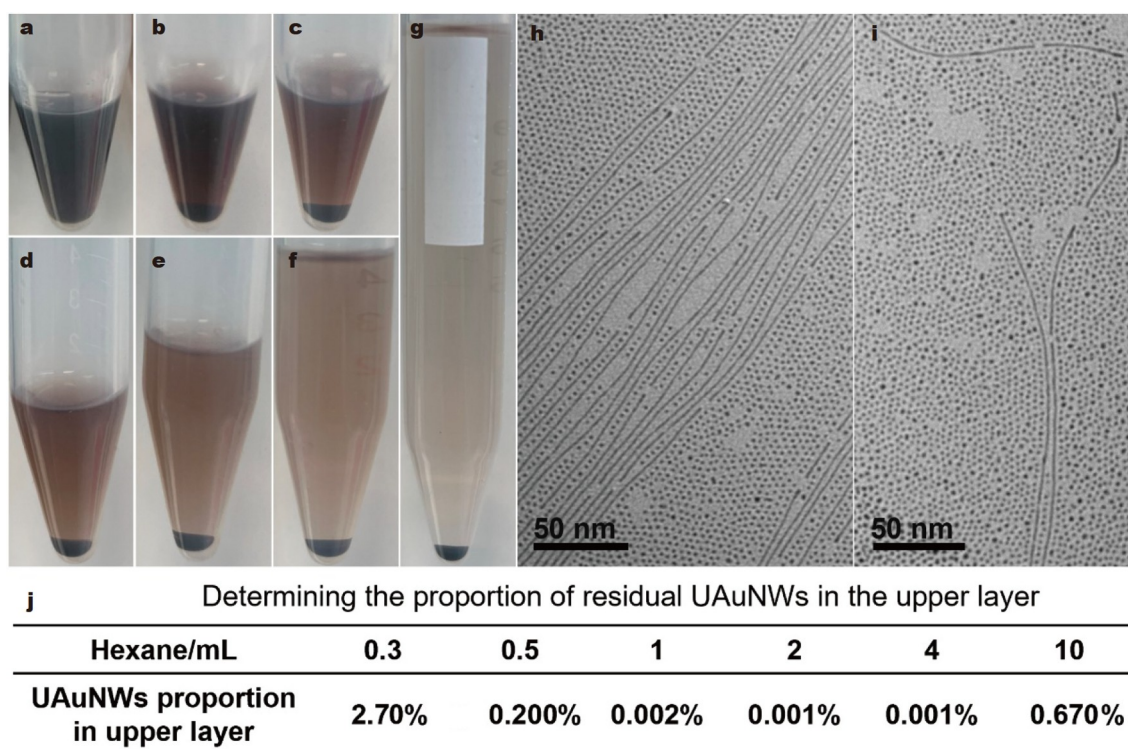


Figure 2 (a–g) Photographs of the UAuNWs dispersion after solvent exclusion method, with increasing the amount of hexane, where the amount of hexane was (a) 0.2 mL, (b) 0.3 mL, (c) 0.5 mL, (d) 1 mL, (e) 2 mL, (f) 4 mL, and (g) 10 mL. TEM images of the upper layer when the amount of hexane was (h) 0.3 mL and (i) 0.5 mL. (j) Summarizes the proportion of residual UAuNWs in the upper layer.

Table 1 Summary of results under different experimental conditions

entry	UAuNWs synthesized in (THF or hexane)	UAuNWs length	Constituents during separation process				Separation ?
			THF	H ₂ O ^a	OAm	hexane	
1	THF	130 nm	✓	0.89 v%	✓	✓	✓
Standard	THF	470 nm	✓	0.8 v%	✓	✓	✓
2	THF	470 nm	✓	✗	✓	✓	✗
3	THF	470 nm	✓	0.8 v%	✗	✓	✗
4	THF	> 1 μm	✓	✗	✓	✓	✗
5	THF	> 1 μm	✓	1.33 v%	✓	✓	✓
6	THF	Identical to above entry 5 , but water was added at the final step					✗
7	THF	Identical to above entry 6 , but with 24 h agitation					✓
8	hexane	> 1 μm	✗	28.5 v%	✓	✓	✗
9	hexane	Identical to entry 8 , but with 24 h agitation					✗
10	hexane	> 1 μm	✓	2 v%	✓	✓	✓

a) The table values denote water percentage in the overall volume, either inherent in the UAuNWs solution or added afterward. There was no water added during UAuNWs (> 1 μm) synthesis in either hexane or THF. Short UAuNWs (470, 130 nm) were synthesized in THF by adding water, adjusting UAuNWs length by varying the water amount.

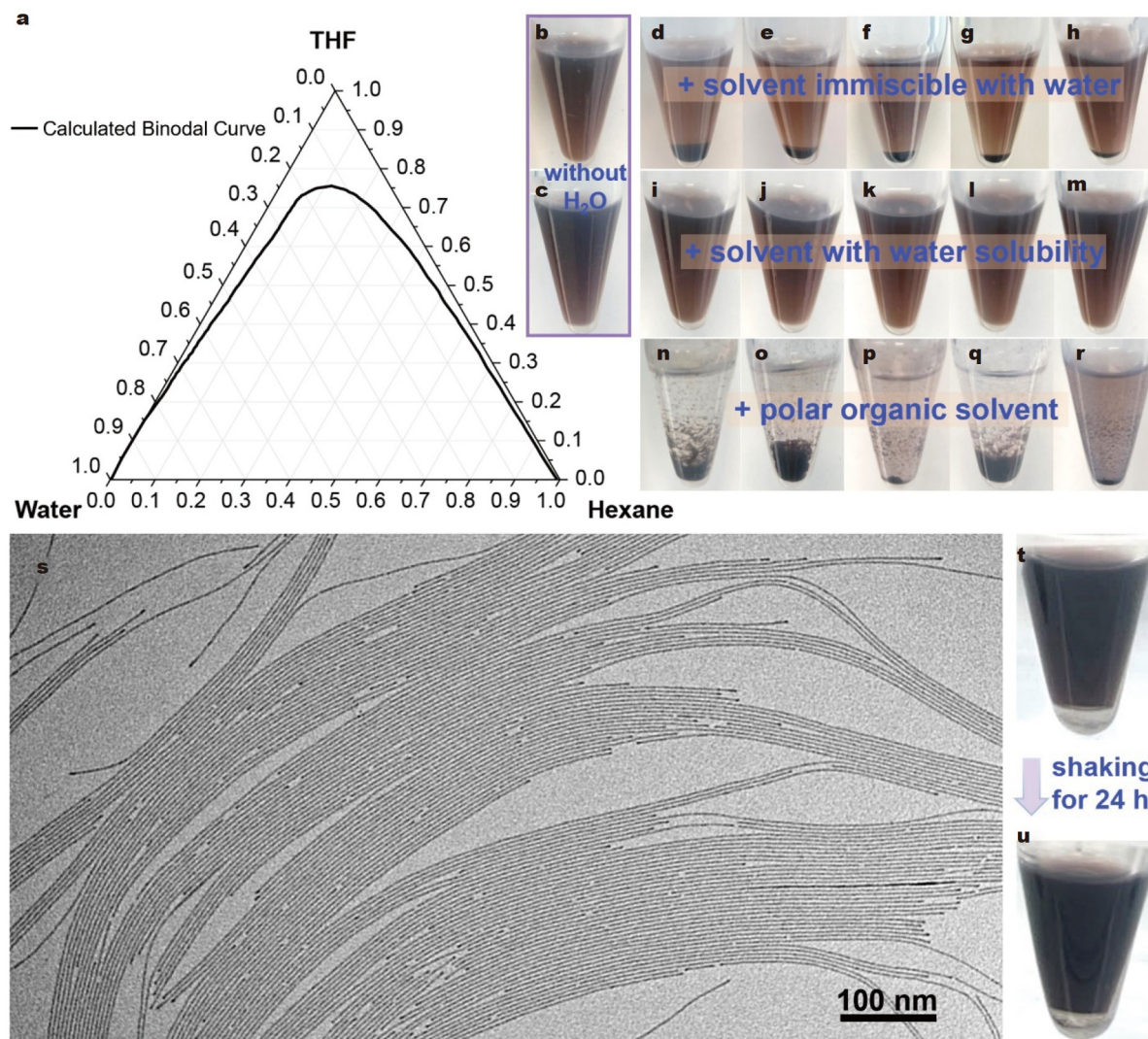


Figure 3 (a) Ternary liquid-liquid phase diagram at 301 K for *n*-hexane-THF-water mixtures. The binodal curve is the boundary between the two-phase and single-phase domains. The two-phase domain is inside the binodal curve (Reprinted with permission from Ref. [38], Copyright 2007, Elsevier). (b, c) UAuNWs mixtures without water do not undergo phase separation. (d–h) Addition of a bad solvent that is immiscible with water led to phase separation: (d) *n*-octane, (e) cyclooctane, (f) cyclohexane, (g) xylene, and (h) toluene. Addition of a solvent with sufficient water solubility did not give phase separation: (i) ethyl ether, (j) dichloromethane, (k) *n*-butanol, (l) *tert*-butanol, and (m) acetone. Addition of a miscible polar solvent did not lead to phase separation but gave precipitates: (n) DMSO, (o) acetonitrile, (p) DMF, (q) methanol, and (r) ethanol. (s) Representative TEM image of the bottom layer of sample (e). (t) The as-synthesized UAuNWs in THF (without water) were firstly mixed with hexane and then added water, and (u) after shaking sample (t) for 24 h in an orbital shaker.

OAm. Upon addition of hexane, the solution remains single-phase (Fig. 3b). This is in clear contrast to the standard reaction where phase separation readily occurs in the presence of water (Fig. 1d). In another control experiment, we used long UAuNWs (>1 μm) which were synthesized without adding water. Direct hexane addition did not induce a phase separation (Fig. 3c).

Many other solvents could replace hexane as the bad solvent. Basically, the bad solvent should be miscible with THF but not with water so that the competition for THF would cause the exclusion of the water phase. Using the same batch of 470-nm UAuNWs, we tested a series of organic solvents. Solvents like cyclohexane, *n*-octane, cyclooctane (water solubility <0.001 wt%), toluene (0.045 wt%), and xylene (0.028 wt%) are miscible with THF but have a low solubility of water. They readily resulted in phase separation (Fig. 3d–h and Fig. S13),

with successful purification of UAuNWs in the dark bottom layer (Fig. 3s and Fig. S14). On the other hand, acetone and *tert*-butanol are miscible with both water and THF; *n*-butanol (water solubility 20.1 wt%), ethyl ether (1.5 wt%), and dichloromethane (0.15 wt%) are miscible with THF but have a solubility of water. They did not induce phase separation due to the minimal water content in the system (Fig. 3i–m).

The role of other non-polar organic solvents is similar to that of *n*-hexane. The difference lies mainly in the varying solubility of water. The volume of the bottom dark layer varied for the above experiments, displaying an inverse relationship with the solubility of water in the bad solvent (Fig. 3d–h). Both *n*-hexane and cyclohexane exhibit extremely low water solubility, less than 0.001 wt%. Consequently, the final volume ratio of cyclohexane is equivalent to that of *n*-hexane when optimal separation effi-

ciencies are achieved (Fig. S13). In contrast, water solubility is relatively higher in xylene (0.028 wt%) and toluene (0.045 wt%). Hence, it becomes evident that the volumes of the bottom layer liquid in xylene (Fig. 3g) and toluene (Fig. 3h) are less than that in *n*-hexane (Fig. 2c), signifying a less effective separation. However, as long as phase separation occurred, the excluded water phase always contained pure UAuNWs (without AuNPs, Fig. 3s and Fig. S14). The efficiency of separation, i.e., the amount of residual UAuNWs in the upper layer, depends on the extent of phase separation.

In addition, there are several solvents that are miscible with both water and THF but cause precipitation, as opposed to phase separation, including polar organic solvents such as DMSO, acetonitrile, DMF, methanol, and ethanol (Fig. 3n–r). TEM images show that the precipitate contains both UAuNWs and AuNPs (Fig. S15), and it is thus not an effective separation. The precipitation occurred regardless of the water content (0–7.7%) in the UAuNWs solution. The precipitation occurred almost immediately for high-polarity solvents such as DMSO and acetonitrile, but it took more than a day for low-polarity solvents such as ethanol.

To further investigate the solvent exclusion mechanism, we designed a set of control experiments. When the as-synthesized UAuNWs in THF (without water) were first mixed with hexane and then added water, the water phase was directly collected at the bottom. It was nearly colorless, indicating the absence of UAuNWs (Fig. 3t). This is in clear contrast to the standard reaction (Fig. 1d), highlighting the importance of the solvent exclusion process for the isolation/purification of UAuNWs. After mixing for 24 h in an orbital shaker, the water phase was only slightly grey (Fig. 3u), indicating that it was difficult for the UAuNWs to penetrate an existing hexane-water interface.

Moreover, for UAuNWs synthesized in hexane (without THF), the direct addition of water also led to a nearly colorless bottom layer of water. It was still colorless after 24 h mixing in an orbital shaker (Fig. S16). When THF was initially added to the sample, the UAuNWs could then be purified with the solvent exclusion (Fig. S17). It appears that THF, being miscible with both water and hexane, is probably a “phase transfer catalyst” for promoting materials exchange between the two phases.

It was surprising that UAuNWs were soluble in water after the solvent exclusion, given that they were soluble in the non-polar solvent of the as-synthesized solution (THF or hexane). This bottom solution was diluted by water (10–20 times), and the UAuNWs were perfectly stable without any precipitation (Fig. S18). TEM images show that the UAuNWs were not

aggregated, with a ligand layer (4 nm gap) among the neighboring UAuNWs. Normally, OAm-stabilized UAuNWs are considered hydrophobic, and in previous studies, when applying UAuNWs to aqueous environments, a ligand exchange process was always necessary to render them water-soluble [13]. In the above paragraph, the UAuNWs in hexane (without THF) were obviously hydrophobic enough to resist being “extracted” by the water phase.

When we removed excess OAm from the purified UAuNWs in water by precipitation with ethanol, the precipitate cannot be re-dispersed in water but can be re-dispersed in hexane (Fig. S19). On these bases, we assign the former to UAuNWs being protected by a bilayer of OAm, whereas the latter have only a monolayer of OAm. In the literature, small-angle X-ray scattering (SAXS) has shown that the as-synthesized UAuNWs have a bilayer structure [21,37]. There is no contradiction: Such bilayer-protected UAuNWs just cannot be extracted by water or easily cross the hexane-water interface. They could be isolated as is by the gentler solvent exclusion process. Normal methods for isolating UAuNWs always involve precipitation by polar solvents (ethanol, etc.), which could easily remove excess OAm and disrupt the OAm bilayer structure [37].

The core issue of purification revolves around shape selectivity between UAuNWs and AuNPs. They both have a diameter of 2 nm and are capped by OAm. Hence, there is no ground for speculating drastically different surface properties. We speculate that during a solvent exclusion, the excluded water may nucleate on the OAm bilayer, given the hydrophilic $-NH_2$ groups facing outwards (Fig. 4). The much longer UAuNWs are expected to collect more water droplets. Their coalescence helps the UAuNWs to move into the water phase. The AuNPs with much smaller surfaces are expected to collect smaller and fewer water droplets, which may be lost in the competition and disappear *via* Ostwald ripening among the water droplets.

To test this hypothesis, we tried to isolate UAuNWs and AuNPs by centrifugation before the water droplets on their surface had time to coalesce. The water phase collected at the bottom contained a significant proportion of AuNPs (44.5%, or 55.5% UAuNWs, Fig. S20), in clear contrast to the high-purity samples of standard purification (>99%). This is fully consistent with the above hypothesis that there were initially water droplets on the surface of AuNPs, allowing them to be isolated by centrifugation. Normally, the 2-nm AuNPs are too small to be isolated by our centrifugation conditions (70 g).

Moreover, when we removed the excess OAm from the as-synthesized UAuNWs, the solvent exclusion process under the

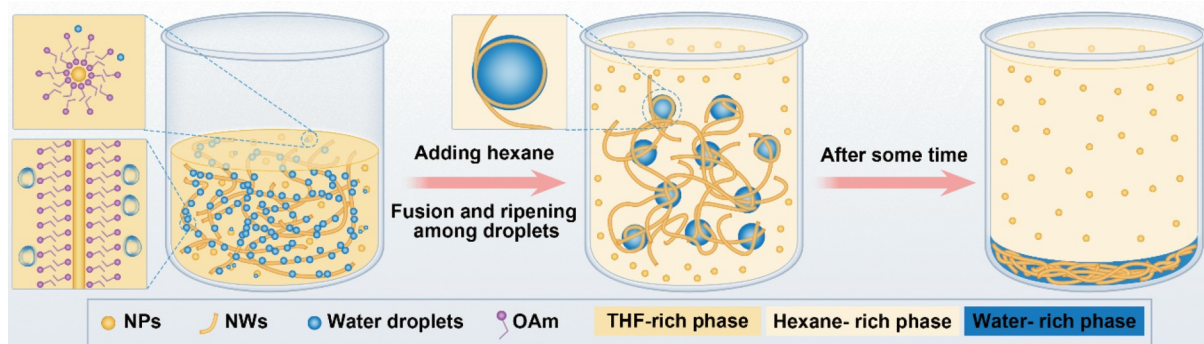


Figure 4 Schematics illustrating the separation mechanism of UAuNWs *via* solvent exclusion.

otherwise identical conditions gave only a light-grey color of the water phase (Fig. S21). Hence, the OAm bilayer is a necessary condition for the nucleation of water droplets on their surface and, thus, for transferring the UAuNWs to the water phase. When we purposely mixed UAuNWs of different lengths, the longer UAuNWs ($>1\ \mu\text{m}$) were preferentially enriched in the water phase (Fig. S22). It is thus consistent with our hypothesis, that the longer UAuNWs may collect more water droplets.

The solvent exclusion method could be applied to both long and short UAuNWs. The synthesis of long UAuNWs ($>1\ \mu\text{m}$) does not involve water, and so the addition of a small amount of water (3.85 v%) is necessary. After the hexane-induced solvent exclusion process (67 v%), the dark solution collected at the bottom showed a high-purity sample of the UAuNWs (Fig. 5b and Fig. S23). For 130-nm UAuNWs, as their synthesis involves water (7.7 v%), the direct addition of hexane led to solvent exclusion and gave a high-purity sample of the UAuNWs (Fig. 5d and Fig. S24). More careful studies show that, in order to minimize the residual UAuNWs in the organic phase (complete separation), 67 v% of hexane is required for the $>1\text{-}\mu\text{m}$ UAuNWs; 80 v% hexane is required for 470-nm UAuNWs; and

89 v% hexane is required for 130-nm UAuNWs. In other words, longer UAuNWs are, in general, easier to transfer into the water phase by solvent exclusion.

As the method only involves the mixing of solvents, it can be easily scaled up. For 10 mL of 470-nm UAuNWs (20 times scale-up from 0.5 mL), the solvent exclusion method was still very effective in terms of the operation and of the high-purity UAuNWs product (Fig. S25).

CONCLUSIONS

In summary, we have developed a facile and effective solvent exclusion method for purifying UAuNWs from the 2-nm AuNPs, which is the common by-product of the synthesis. The high-purity samples ($>99\%$) cannot be achieved by centrifugation-based methods, and in particular, the purification of short UAuNWs (470 and 130 nm) has not been previously demonstrated in the literature.

The purification mechanism is investigated. The solvent exclusion depends on the solvent miscibility and polarity, and thus, a wide variety of solvents could be used. So long as water is excluded from the colloidal solution, it would preferentially

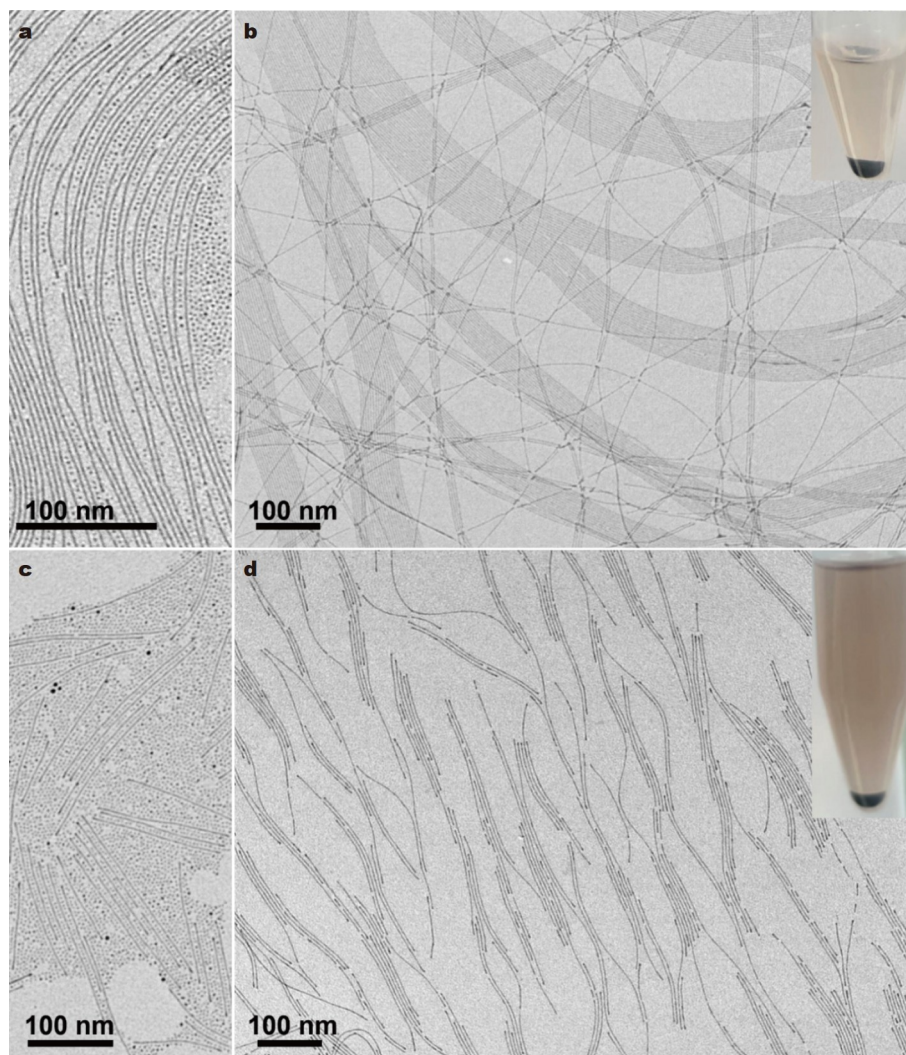


Figure 5 TEM images of (a) unpurified sample with long UAuNWs ($>1\ \mu\text{m}$) and (b) UAuNWs in the bottom layer after solvent exclusion. TEM images of (c) unpurified sample with short UAuNWs (130 nm) and (d) UAuNWs in the bottom layer after solvent exclusion. Insets: photographs of the UAuNWs samples after solvent exclusion.

nucleate on the OAm bilayer of UAuNWs, transferring them into the water phase. Thus, the purification would fail if the OAm bilayer is disrupted or when the water droplets on the UAuNWs do not have enough time to coalesce.

Exploration of new purification methods is critical for advancing nanotechnology. Our solvent exclusion method is distinctively different from the conventional purification methods. We believe that it would open new possibilities in designing separation/purification methods and for using ultrathin nanowires in various applications.

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Author contributions Yang Q and Chen H conceived the idea and explained the mechanism of separation, contributing to the writing and revision of the manuscript. Yang Q conducted the syntheses, separations, and

characterizations of the nanomaterials. Wang R and Cheng X participated in discussions, providing valuable insights into the mechanisms.

Conflict of interest The authors declare that they have no conflict of interest.

Supplementary information Supporting data are available in the online version of the paper.



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简单地纯化超细金纳米线的方法: 溶剂排除法

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摘要 在超细Au纳米线的合成过程中常伴随着2 nm Au纳米颗粒这一副产物, 两者如何分离是长期存在的问题. 本研究通过使用溶剂排除法实现了高纯度(>99%)分离: 加入正己烷会导致水从最初的水-THF混合物中被排除. 被排除的水优先在纳米线表面吸附的油胺双分子层上成核, 从而将纳米线转移到水相中. 本文通过仔细地对照实验建立了纯化机理. 这种简便的方法可以用于传统方法无法实现的短的超细金纳米线(470和130 nm)的纯化.