Simultaneous Multidrop Creation with Superhydrophobic Wells for Field Environmental Sensing of Nanoparticles

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ABSTRACT: Facile creation of multiple drops at appropriate volumes on surfaces without the use of sophisticated instrumentation facilitates downstream evaporative preconcentration of liquid samples for analytical purposes. In this work, a superhydrophobic (SH) substrate comprising wells with a perforated mesh base was developed for simultaneous drop creation in a quick and convenient manner. In contrast to the method of pouring liquid directly over the SH wells, consistent liquid filling was readily achieved by a simple immersion approach. This method works well even for challenging situations where well diameters are smaller than 3.4 mm. Despite the poor liquid-retention properties of SH surfaces, inverting the wells did not result in liquid detachment under gravitational force, indicating strong pinning effects afforded by the well architecture. The perforated base of the well allowed the liquid to be completely removed from the well by compressed air. High-speed camera image processing was used to study the evolution of drop contact angle and displacement with time. It was found that the liquid body was able to undergo strong oscillations. Optical spectroscopy was used to confirm the ability of evaporative preconcentration of silver nanoparticles.

INTRODUCTION

The attractive physicochemical properties of nanoparticles, in particular, their extremely small size, large specific surface area, and unique optical properties, have led to growing interest in the development of diverse applications in the fields of medicine, electronics, chemical, and clean energy production. However, these nanomaterials can enter human and animal systems through many pathways and exert harmful effects at the cellular, tissue, and organ level.1,2 The exact underlying mechanism of this toxicity remains to be clearly established, but recent studies have indicated the involvement of oxidative stress induction,3,4 proinflammatory gene activation,5,6 and ATP deprivation leading to autophagy and apoptosis.7 In view of the potential toxicological effects associated with nanoparticle exposures, various entry routes of nanoparticles from emission sources to the natural environment, including atmospheric outfalls, solid surface leaching, and industrial or urban emissions from municipal wastewater treatment plants are of major concern.8 Effective implementation of remediation efforts, an area of recent intense pursuit,9–11 relies heavily on the availability of sensitive instrumental tools for environmental nanomonitoring. Although nanoparticles are ubiquitously found in natural systems, their presence at trace concentrations represents a major impediment to their detection and monitoring. To address this issue, highly sensitive (and normally expensive) methods of detection are required.12,13 Effective approaches to preconcentrate the nanoparticles in solution prior to sensing can ameliorate this.

Effective preconcentration with minimal sample loss by entity deposition on surfaces is achieved by minimizing the area of contact between a solid substrate and the liquid sample. Superhydrophobic (SH) surfaces, now created by a wide variety of surface treatment methods,14–16 offer the ideal means to accomplish this. Liquid bodies on SH surfaces assume drops that exhibit high contact angles and low surface attachment. Such wetting behaviors are governed by two-phase (liquid and air) and three-phase (liquid, solid, and air) interactions.17 Recently, a “manhole” preconcentration approach18 based on evaporation19 and suitable for field application was reported. To attain higher detection sensitivity, the preconcentration process is performed by creating multiple drops where volume reduction is achieved in successive stages (see Figure 1A). In this work, we describe a scheme whereby the key objectives are to create multiple drops at preselected volumes simultaneously, quickly, and conveniently. This scheme which relies on SH well architectures is also designed to operate seamlessly with the preconcentration approach to achieve minimal sample loss by surface deposition reported.

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Figure 1. To obtain a high degree of preconcentration, a series of drops can be reduced in size by evaporation so that they fall through under the manhole effect and can then be collected to form a cascading process (A). Drops for pre-evaporation are to be created simultaneously by immersion into water through SH well architectures that can have a solid (B) or mesh (C) base.

The key functions pursued include the ability to attain consistent liquid well-filling by immersion and for the formed drops to be dispensed from the wells readily. Two well designs, one with a solid (Figure 1B) and the other with a meshed base (Figure 1C), are investigated.

## MATERIALS AND METHODS

### Substrate Preparation

Holes of various sizes were drilled through 2 and 4 mm thick copper plates to form the side walls of the wells. Holes with diameters of 2.4, 2.8, and 3 mm were created in 2 mm thick copper plates, whereas larger holes with diameters of 3.4, 3.7, 3.8, 4.1, and 4.6 mm were made in 4 mm thick plates. The base of the wells was made of either a thin copper plate (thickness = 1 mm) or a copper mesh (wire thickness = 0.1 mm, spacing = 0.3 mm). All of the plate samples were polished until they were shiny (the mesh base was not polished). They were then ultrasonicated in ethanol (70 v/v %) and acetone for 3 min and subsequently in deionized (DI) water for another 15 min. The substrates were oxidized in a solution containing 2 M NaOH and 1.5 M (NH₄)₂S₂O₈ for 5 min. The samples were then allowed to synthesize in an oven at 180 °C for 120 min to complete the phase transfer from hydroxides to oxides. At the end of the reaction, the substrate was removed from the solution, rinsed several times with DI water and ethanol, and then dried using compressed air. In the final step, the substrates were silanized using flame absorption spectroscopy (FAS) (1H,1H,2H,2H-perfluorodecyl-triethoxysilane) to obtain a low-surface energy material with good corrosion resistance and thermal stability. The substrate was immersed in FAS–ethanol solution for 30 min and dried in the oven at 150 °C for 10 min. Finally, the base of the wells was formed by using epoxy to attach either the silanized thin copper plates or copper mesh to the silanized thick copper plates with holes.

### Characterization of Surfaces

Representative samples of the silanized copper plate and mesh were placed on stubs using conductive adhesive and examined using a scanning electron microscope (FEI, Nova Nano SEM 430). Optical profilometry was also used to examine the surface of the copper plate. Images of the copper surface located 1 mm from the hole was obtained. The sample was attached onto a flat silicon surface that served as the reference surface. Optical scans obtained from the profilometer (Bruker Contour GT-I) were processed and analyzed using the instrument’s accompanying software (Contour Elite). Because of the relatively large surface area to be examined, multiple images were recorded and the software-stitching capability was used to construct an integrated map over an extended field of view.

The contact angles of 10 μL sessile water drops dispensed on the substrate were measured using the Kruss DSA100S system. The mean value from 10 separate readings was found to be 158° (σ = 0.80°). The inclination angle needed for the displacement of 10 μL drops was found to be 3°.

### Liquid Filling

The SH samples (wells facing upward) were placed on a holder and lowered slowly into a beaker of water to a depth of 20 mm below the water surface (Figure 2A) to allow the well liquid filling by hydrostatic pressure alone. The substrate was held in position for 10 s and then raised up slowly.

![Figure 2](image_url)

**Figure 2.** (A) Immersion method was conducted by using a holder to slowly lower the SH sample (with wells) into a beaker to a specific depth \( h \) below the water surface. The amount of water retained in the well was then evaluated. (B) Propensity of the liquid drop to be displaced from the well was determined by inverting the substrate. In the case of wells with a meshed base, a jet of compressed air was delivered by placing the distal end of the air tube at a fixed distance \( h' \) from the superhydrophobic base of the well. The air pressure was controlled by a valve, and the pressure applied was measured by a manometer.

The volume of liquid collected in the well was determined using two methods. For large wells on the 4 mm thick substrate, liquid drop volume was determined by mass measurement using a weighing scale (Ohaus, SE2020) with 0.01 g resolution before and after the liquid was completely removed by absorption onto the paper towel. In the case of more challenging smaller wells on the 2 mm thick substrate, a glass capillary tube (inner diameter = 0.58 mm) was used to completely drain the liquid from the well, and the drop volume was then determined from the length of the liquid column in the capillary tube.

The liquid retention characteristics of the drops in wells were evaluated by inverting the SH substrate (Figure 2B), and side views were recorded using a camera (Motincam 3) with diffuse back-illumination.

### Liquid Displacement

In the case of wells with a meshed base, liquid drops were displaced by delivering compressed air from a tube placed at a fixed distance \( h' \) (10 mm) from the base (Figure 2B). Valve-controlled air pressure was measured via a digital manometer (Digitron, 2002P) placed in the airflow line. The discharged air pressure was correlated with the force exerted through a calibration procedure where a weighing scale (OHAUS, SE2020) with 0.01 g resolution was placed at the same distance \( h' \) away from the distal end of the tube. The
manner in which the drops were displaced from the wells was observed by high-speed side-view images recorded using a camera (Fastec) at 250 frames per second.

**Spectroscopic Measurement of Silver Nanoparticle Concentration.** Silver nanoparticles (40 nm average diameter; Sigma-Aldrich) was suspended at a concentration of 20 μg/mL in aqueous buffer containing sodium citrate as a stabilizer. This sample was diluted with DI water to obtain a nanoparticle concentration of 0.032 μg/mL. The evaporative concentration approach described in this work was used to create preconcentrated samples. The concentration of silver nanoparticles in the preconcentrated sample was determined from their UV light absorbance spectra. Sample absorbance was measured in a 10 cm path-length quartz cuvette placed between a UV LED light source (Jaycar, ZD0260; emission maximum at 400 nm) and the distal end of an optical fiber that was connected to a spectrometer (Ocean Optics).

## RESULTS AND DISCUSSION

SEM images of the SH plate and mesh surfaces are shown in Figure 3A,B, respectively. The hierarchical microscale and nanoscale structures are predominantly prismatic. Apart from this, no other artefacts were found that could have contributed to the predominant Cassie wetting characteristics observed. Figure 4A,B shows the corresponding optical profilometry traces of the SH plate and mesh surfaces. This provided further confirmation that even at a larger length scale no other surface artefacts were present that could have contributed to the predominantly Cassie wetting characteristics. Additionally, the low-adhesion characteristics of the substrate were corroborated by the displacement of 10 μL drops off the surface with inclinations as low as 3°.

Liquid well filling using the immersion approach is depicted in Figure 2A. The volume of liquid in the wells of various sizes is shown in Figure 5 and is consistently demonstrated to be higher than the corresponding theoretical well volume indicated as red dots on the plots. Evidently, overfilling of the wells occurred whereby a convex meniscus was formed over the mouth of the well. The volume of liquid filling the wells showed only small variations in repeat measurements, indicating that the immersion approach was suitable for use in the evaporative preconcentration scheme, as outlined in Figure 1A.

As an alternative to the immersion approach, attempts were also made to fill the wells by tilting the substrate to an angle of 5° to the horizontal and pouring liquid down the inclined surface from the raised end. The measured volumes collected in the wells showed significantly higher variations than the immersion method (see Figure 6). This can be attributed to the dynamic nature of of liquid-well interaction which in turn affects the ability of the three-phase contact lines to pin adequately. The lack of sufficient hydrostatic pressure as liquid moving over the top of the well is also another contributing factor for poor liquid filling. It is noteworthy that liquid filling did not occur when the well diameters were smaller than 3.4 mm. This may be attributed to the strong nonwetting characteristics of the surface that would tend to prevent liquid from entering the well. This behavior is exacerbated by the dynamic actions occurring at the three-phase contact line and the lack of hydrostatic pressure to aid the filling process. Capillarity can be ruled out as a factor because of the relatively large diameters of the wells involved.

Surprisingly, liquid was retained within the well when the SH substrate with a solid base was inverted (Figure 7A). This phenomena could be attributed to some initial air entrapment during filling. It is then possible that the liquid is held in place because of a pressure differential between the entrapped air and that in the environment. However, a similar liquid retention behavior was also observed for wells with a meshed base (where the side-view image of the drop appeared exactly the same, as shown in Figure 7A). Hence, this explanation for liquid retention as a result of air entrapment cannot hold true because pressure equalization would occur in the latter case. The strong liquid retention is then likely caused by increased pinning offered by the edges of the well. Strong pinning from edges of hydrophilic surfaces is well-known and has been
demonstrated.\textsuperscript{21,22} That the same increased pinning effects are observed here using substrates that manifest predominant Cassie wetting correlates with recent findings of drops strongly retained on holes created on thin SH substrates.\textsuperscript{23} Liquid may be recovered from the well in two ways; by application of impact forces or by transference of the liquid to a substrate with higher wetting properties. The former results in poor movement control and potentially some sample loss, whereas the latter does not fit with the scheme of subsequent evaporative preconcentration on SH manholes. The meshed base architecture presents an opportunity to use a third method; that of compressed air delivered down toward the mesh base to displace the liquid from the well. The relative “gentleness” of air in actuating liquids on low wetting surfaces had been demonstrated previously.\textsuperscript{24} Figure 8 presents results showing the air pressure needed to just dislodge the liquid from the well. As expected, higher pressure is needed for wells with smaller diameters because liquid volumes are smaller and hence higher surface tension forces are exerted. It was observed that relatively small variations in air pressure were needed to completely remove liquid from wells of a specific diameter. This finding makes it possible to determine the minimum pressure needed to empty all of the wells consistently. For instance, the application of an air pressure of 12.5 mbar will remove liquid from the 2.4 mm diameter well all of the time. Hence, this air pressure will be more than sufficient to remove liquid from wells of larger diameters. The applicability of a fixed air pressure setting is advantageous in field applications, whereby standard air dusters can be used to deliver the required force for liquid dislodgement from the wells. However, this may have an impact on the speed at which...
the drop leaves the well and subsequently on the force with which the liquid impinges on its destination substrate. This has been extensively studied\textsuperscript{25,26} wherein the ability of the drop to execute elastic rebounds especially on increasingly hydrophobic destination substrates has received particular attention.\textsuperscript{27–29}

At this juncture, it is noteworthy that compressed air was delivered through a refrigerated compressed air dryer before use. In the unit, the inlet warm wet air was cooled to about 3 °C, where any water vapor that developed was condensed into water and then removed via a water trap. Subsequently, the cold air was reheated to room temperature. Hence, it was unlikely that any vapor in the compressed air contributed significantly to the detachment process.

High-speed camera images of the liquid body as it was displaced from the well showed an early stage axis-symmetric drop that evolved to an asymmetric form over time (Figure 7B). There was also an absence of any pinch-off that is typically observed for liquid dripping from a faucet.\textsuperscript{30} This is not surprising as the entire liquid body was essentially displaced out of the well without any continuum of liquid to fill the space behind it. With an increasing absence of liquid—solid interactions, there is propensity for the liquid to attach preferentially to one side of the well in the later stages because it is impossible to ensure equal wettability all around. In doing so, the axis-symmetric nature of the liquid body disappears. This has the effect of displacing the delivered drop from the central axis of the well, albeit the extent of this is not significant. Receptacles with geometries (e.g., truncated cones) that help to redirect the drop to specific locations on the destination surface will ensure that this is not a limitation. Alternatively, careful design of the edges of the well offers the possibility of directing the detaching drops toward more axis-symmetric deliveries. It is important to point out that because there is no pinch-off, there is no development of daughter drops which can contribute to any sample loss.

A quantitative analysis of the sequence of images in Figure 7B indicated that drop dislodgement occurred within a relatively short time of around 20 ms (Figure 9). The contact angle was initially at 60° but increased to 100° around the midpoint followed by restoration to 60° prior to dislodgement (Figure 9A). The trace of the nadir position of the liquid body (Figure 9B) relative to its origin, however, indicated an almost linear increase with time till dislodgement. This behavior is consistent with the lower portion of the liquid body moving in a downward direction initially, whereas the contact line was being pinned to increase the contact angle toward advancing. The continuing motive force of air delivery presents the liquid body with two options; either continue to manifest an increasing contact angle up to the point of pinching off or to move itself out of the well. The latter occurs because of the lack of a continuum of liquid being delivered as well as the propensity of the rear contact line (which is higher up) to move down. This causes the liquid body to assume a spherical shape to minimize its surface energy, thus resulting in a reduction in contact angle just prior to dislodgement.

Because the liquid body is subject to this form of perturbation, the surface tension forces involved can drive it to respond dynamically. This is also possible because of compressed air being used here which generates a more stochastic driving impetus as opposed to gravity forces acting alone.\textsuperscript{18,23} It is noteworthy that resonant behavior had previously been observed in the presence of stochastic perturbations\textsuperscript{31–33} and has been found to occur notwithstanding the small restoring forces developed from SH surfaces.\textsuperscript{32,33} Although some small extent of this was discerned, the relatively short time period leading to dislodgement does not furnish the means to confirm this.

The contention that the liquid body in the well exhibits oscillatory behavior prior to dislodgement as a result of restoring forces leads to the question of whether a more pronounced manifestation might be exhibited if the drop was actuated by air but not to the extent that it was dislodged from the well. Such a situation is depicted in the high-speed camera sequences given in Figure 10B. The trace of contact angle evolution with time (Figure 11A) shows the liquid body...
manifesting values that deviate from an initial 60° and reaching a maximum of 130°. Interestingly, periodic fluctuations were found along the way before the contact angle values settled close to 90° at the point at which the liquid body was detached and hanging outside the well. This indicated an ability of the liquid body to exhibit pronounced oscillations in the process. The extent of this behavior is better revealed by following the corresponding displacement-time trace of the nadir position of the liquid body (Figure 11B) from its origin. It is noteworthy that pendant drops actuated by air pressure driven by a loudspeaker have been shown to exhibit oscillations previously.34

It is important to note (from Figure 11B) that the liquid body is pushed out to its maximum extent at a very early stage of the actuation. This is illustrated by the fact that it does not return to its original position of Figure 10A even after air actuation has ceased. With a displaced position downward, the three-phase contact line is subject to an advancing state early and is thus strongly affected by the added pinning from the edges of the well. This then gives impetus for the liquid body to develop restoring forces, notwithstanding the continued downward impetus provided by the compressed air, such that oscillations are manifested. That the degree of liquid body deformation is more pronounced initially, gives rise to periods that were shorter initially (~17 ms) before lengthening gradually up to the stages before settling (~25 ms). It is also noteworthy that the fluctuations exhibited diminishing amplitudes (see Figure 11B). Taken together, this infers a mechanism in which the liquid body undergoes gradual (a matter of perspective because this occurs over only 350 ms) relaxation with time.

The absorbance scan of the original nanoparticle sample (20 μg/mL) in Figure 12 showed a UV–vis peak wavelength around 400 nm. This result is in agreement with that found in a previous study.35 When this sample was diluted 625 times, its optical spectrum was almost verbatim to that exhibited by water. Following preconcentration, peak absorbance at 400 nm was restored, indicating the feasibility of the evaporative approach. The absorbance values obtained from repeated experiments (see inset of Figure 12) also indicate that the approach is reproducible.

On a final point of application, the well filling approach here is highly amenable for application in the field. By creating multiple drops from an array of wells and releasing them into multiple holes for preconcentration, it is possible to attain results more conveniently and expeditiously, with the volume of each drop remaining consistent. It is also important to note that the SH surfaces are self-cleaning which obviates the use of solvents.

## CONCLUSIONS

The ability to form multiple drops simultaneously, quickly, conveniently, and at prespecified volumes on surfaces of a SH construct, such that it facilitates subsequent evaporative preconcentration, is demonstrated here. The immersion approach to fill the wells, which yielded recovery of consistent volumes of liquid, is an approach that can be conducted with...
ease in the field. That strong liquid retention within the well is observed even against gravitational forces when the substrate inverted is advantageous because it allows subsequent delivery of the liquid to a desired location on the destination surface. Actuation of liquid removal by compressed air allows preselection of wells from which the liquid body (from an array) is to be removed for collection. Complete removal of liquid from the well by a burst of compressed air delivered through the mesh base of the well is both convenient and gentle enough not to foment formation of daughter drops, which engenders material loss. The ability of the liquid body to undergo oscillations does not have practical implications in the application context described here. However, it illustrates a liquid behavior that has not been reported to date and may be useful in developing density gradients in liquids, from which convection can then cause changes to the diffusion rates of molecules present in the liquid sublayer.

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**Notes**

The authors declare no competing financial interest.

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