Electrically Controlled Formation and Release of Admicelles for Solid Phase Extraction

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ELECTRICALLY CONTROLLED FORMATION
AND RELEASE OF ADMICELLES
FOR SOLID PHASE EXTRACTION

A Thesis Presented to
The Faculty of the Department of Chemistry
Western Kentucky University
Bowling Green, Kentucky

In Partial Fulfillment of the
Requirements for the Degree
Master of Science

By
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May 2014
ELECTRICALLY CONTROLLED FORMATION
AND RELEASE OF ADMICELLES
FOR SOLID PHASE EXTRACTION

Date Recommended  April 11, 2014

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Acknowledgement

For this research there are so many people that I would like to thank. Without them I would have never made it this far. I give the biggest thanks to my father, Dr. Yong-ill Lee, who has lead me into the world of chemistry, and my mother, In-soon Jung, for the utmost support for walking the path to being a chemist.

For my research I would like to thank Dr. Conte and Dr. Burris for the endless support for the project and guidance in studying analytical chemistry. Also I am very thankful for the help other than research that I got from my research advisors, so that I could concentrate more on my studies. I would also like to thank Dr. Nee for accepting to be one of my thesis defense committee, Dr. Andersland for the wonderful SEM pictures and suggestions for coating the frit, Dr. Cao for the physical data of the frit, and all of the chemistry department faculty and staff for all the help I received.

This work was supported by the National Science Foundation (NSF-CHE RUI 1008356).
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Solid phase extraction is one of the most widely used methods to concentrate diluted compounds in a solution. Substances can be extracted into admicelles and hemimicelles, which are surface adsorbed micelles and surfactant monolayers, respectively. Investigations of the electrical control of surfactants on surfaces for the purpose of analyte preconcentration prior to chromatographic analysis are presented. The surfactant layer serves as the “stationary phase” in a solid phase extraction sorbent scenario. Analytes are adsorbed on this layer, and then released from the solid phase via surfactant removal. The attachment and removal of the surfactant are controlled by means of an electric field. Because the surfactant-analyte association is released by electrical control, organic solvents, which are used in conventional solid phase extraction, are not required. Therefore, this procedure is advantageous for method development and environmental concerns. Presented is the preconcentration of a test probe, 2-naphthol, using electrical control of the formation and release of dodecyl sulfate on planar gold, gold coated stainless steel, and a porous stainless steel frit, using impedance spectroscopy to observe the layer formation with various surfactant concentrations and applied potentials.
INTRODUCTION

Analytical chemistry applications of admicelles and hemimicelles are newly investigated phenomena. Admicelles are micelles formed on surfaces as bilayers and hemimicelles are surfactant monolayers. These formations are the result of the charged group on ionic surfactants being attracted to oppositely charged surfaces, in addition to surfactant chain-chain attractions. The variations between these two states occur by changing pH, ionic strength of the solution, and the type of surfactant added. As with micelles, admicelles and hemimicelles are aggregates of surfactants that are able to solubilize a wide variety of substances. [1] In numerous studies, changes in the concentration of surfactant, solution pH, electrolyte concentration, and surface type have been investigated to determine what variations among between these two assemblies (hemimicelles and admicelles) may be forming on surfaces. [2] These properties of surfactant micelles were used as a stationary phase in a variety of methods for the analysis of benzimidazolic fungicides [3], pesticides [4], estrogens [5], bisphenols [6], and quaternary ammonium herbicides. [7] Other applications of admicelles include carbon nanotubes being used as the base surface for micelle aggregation [8], and catalysts for a hydrolysis reaction [9].

Recently a study was conducted for applying this method to solid phase extraction (SPE) [5]. SPE is performed by many analytical laboratories, and is used to concentrate, purify, or separate an analyte of interest. SPE is a method that is capable of separating compounds in a solution from other compounds according to their physical and chemical properties. SPE uses the difference of the affinity of each components in the mixture
dissolved or suspended in a liquid (mobile phase) for a solid through which the sample is passed (stationary phase) to separate components of a mixture. The result is that either the desired analytes of interest or undesired impurities in the sample are retained on the stationary phase. The portion that passes through the stationary phase is collected or discarded, depending on whether it contains the desired analytes or undesired impurities, respectively. Analytes retained on the stationary phase are eluted by passing additional eluents through the stationary phase.

According to a study by J. Lipkowski et al. [9], it is possible to form a monolayer of dodecyl sulfate on a gold surface by controlling the applied surface potential. By adjusting the potential of a gold surface, various formations of dodecyl sulfate could be formed.

In this study, the formation of dodecyl sulfate layers on a gold surface from a sodium dodecyl sulfate (SDS) solution was investigated with a subsequent application for SPE. The main focus of the study was to use controlled electrical potentials for the formation of the surfactant layer over the metal surface and the release of the target analyte that has been adsorbed to the layer. This has an advantage of being cost effective and environmentally friendly because no organic solvents are needed for this method, unlike typical SPE. The test molecule, 2-naphthol, was adsorbed into the SDS layer formed at a charged surface and released it into the solution with the surfactant by changing the surface potential. For actual laboratory applications various materials were studied to increase the surface area of the surfactant layer.
EXPERIMENTAL

Chemicals

Fig 1 Chemical structure of 2-naphthol

Fig 2 Chemical structure of sodium dodecyl sulfate (SDS)

2-naphthol is a compound that is composed of two aromatic rings with a –OH group at the 2-position. 2-naphthol was used for the study because it has a similar chemical structure of environmental hazardous materials such as phenols and their derivatives, but has a simpler structure and is relatively non-toxic. The aromatic rings are hydrophobic, which makes it easy to get 2-naphthol adsorbed onto the SDS surfactant layer, and the –OH group on the 2-position shows some hydrophilic properties giving 2-naphthol some water solubility. (Fig 1) Compared to the carbon chain tail of the micelle, the sulfate head is hydrophilic. During the SDS surfactant layer formation, the attraction of the positively charged surface and the negative charge on the oxygen bonded to the sulfate facilitates the formation of the surfactant layer alongside with the affinity of sulfate with gold. (Fig 2)
Gold slide

Fig 3. Illustration of the experiment cell

Gold slides were obtained from the Evaporated Metal Films Corporation. The slide had a dimension of 25 mm x 75 mm x 1 mm with a 1000 Å Au layer over a 50 Å Ti coating on glass. SDS and 2-naphthol were purchased from Sigma-Aldrich. Impedance spectroscopy has been conducted with PARSTAT 2263 (Princeton Applied Research). For the investigation of the SDS surfactant being formed on the gold surface, a cell was constructed to allow contact with the gold slide. (Fig 3) The investigation was conducted over three stages: blank stage, SDS stage, and 2-naphthol stage. Fifty capacitance measurement cycles were taken at each stage. At the beginning of each experiment a series of cyclic voltammograms (CVs) were conducted to clean the gold surface. The voltages for the CVs were 0.2V to 0.9V, 0.2V to 1.2V, and 0.2V to 1.5V. All series of CVs were done at a rate of 100 mV/s, and were cycled 10 times. During each CV, the cell was filled with 0.1M sulfuric acid (H₂SO₄) prepared with 5 mM potassium chloride (KCl) solution.

The blank stage is the blank measurement of the capacitance of the surface without the molecules that were used for the experiment. The cell was filled with 0.1M KNO₃
solution. For the SDS stage, the concentration of SDS was varied from 0.33 mM to 16 mM. As the capacitance measurement for the SDS surfactant layer formation was finished, the solution inside the cell was exchanged to water. This procedure was needed to remove the unabsorbed SDS from influencing further experiments. A plastic transfer pipette was used to remove half of the solution from the cell and water was added to fill up the cell. This process was repeated 10 times. After the solution exchange, 2 mL of 0.5 ppm 2-naphthol was added to see if the surface formed during the SDS stage could adsorb 2-naphthol. For all stages the capacitance was measured at 0.8 V.

**Coating of stainless steel frit**

Hydroxylamine hydrochloride and gold chloride solution were purchased from Sigma-Aldrich. The frits used for the coating and the preconcentration experiment were purchased from Upchurch scientific. The pore size of the frit was 10 µm with a diameter of 1.27 cm and a height of 2.54 cm. All SEM images were taken with the SEM (JEOL 5400LV) in the Biology Department of Western Kentucky University. The coating was applied in a reaction cell containing a solution of 0.2% chloroauric acid (HAuCl₄), 0.4 mM hydroxylamine (NH₂OH) and water mixed in a ratio of 1:1:1. The frit was connected as the working electrode with a stainless steel wire and a Pt wire coil served as the counter electrode located around the frit. The applied voltage was 0.5 V. To facilitate the flow of the solution in and out of the frit, a syringe pump with a continuous cycle was connected. A stir bar was used to prevent precipitates from forming. The syringe pump was purchased from KD Scientific (Model 210) and was used in a continuous cycle mode with 3 mL volume being pulled and pushed at a rate of 5 mL/min.
Preconcentration

In the preconcentration experiment, the stainless steel frit was coated with gold. A similar setup from the gold coating reaction was used, except for the use of the stir bar. The preconcentration experiment consisted of three stages: DS layer formation, extraction, and surfactant release. 4mM SDS was used for the layer formation and the solution was exchanged with water after 40 EIS cycles. After the exchange of SDS solution to water, 2-naphthol solution was spiked into the cell to provide a solution concentration of 4 ppm 2-naphthol. When the extraction stage was finished, the potential that was applied to the cell was turned off in order to remove the DS layer and the absorbed 2-naphthol. In all but the release stage, 0.8V was applied to the frit, and a syringe pump was used all times to provide a flow of solution into and out of the frit. A sample was taken from the beginning of each stage to measure the 2-naphthol concentration change. The 2-naphthol concentration was analyzed by high performance liquid chromatography (HPLC). The HPLC was composed of a Varian 9012 pump, a C\textsubscript{18} column purchased from Varian, and a Varian Prostar 330 PDA detector. The mobile phase was a mixture of HPLC grade methanol and water, mixed in a 60:40 ratio and was pumped at a rate of 1.00 mL/min for 10 min. For detection, the wavelength range of the photodiode array detector was set at 200 nm to 400 nm with a monitored wavelength of 250 nm.
RESULTS AND DISCUSSION

Gold slide

![Capacitance measurement graph](image)

Fig 4. Capacitance measurement with 0.33mM SDS. Stage A, B, and C was measured in a solution of A: 0.1M KNO₃, B: 0.33mM SDS, C: Addition of 0.5ppm 2-naphthol 2mL.

The initial attempts to get the SDS surfactant layer onto the gold slide were conducted with 0.33mM SDS. Stage A was the capacitance measurement of the gold slide with 0.1M KNO₃ in the cell. This measurement is a blank measurement to be compared with stage B. Stage B is the measurement with the cell containing SDS solution, and stage C is the measurement of the cell when 2-naphthol was spiked in. The main purpose of these separate measurements was to see the difference of capacitance due to the presence of a layer formed on the surface. (Fig 4) Except for stage C in Fig 4, each stage had a decrease in capacitance for the first 20 cycles of measurements. This was due to the
time for the solution to reach equilibrium. After equilibrium there was a drop of capacitance after each stage, which means that each stage resulted in a new surface formation. From the results of these experiments, DS surfactant was formed onto the gold slide as evidenced by the observed capacitance drop. However, the decrease of capacitance of each stage was about 5 µF. With a low SDS concentration, there is not enough DS surfactant layer being formed to lower the capacitance of the gold slide. Also there was not much DS surfactant layer present on the surface for the 2-naphthol to get adsorbed. To achieve a bigger capacitance drop, the experiment continued with various SDS concentrations from 1mM to 16mM during the SDS surfactant layer formation stage.

![Capacitance graph](image)

**Fig 5. Capacitance change after turning the cell off**

An experiment with an additional cell off stage was conducted. The purpose of this experiment was to see if the capacitance of the gold slide would be higher than the initial capacitance value. Ideally, when the potential applied to the gold slide is turned off, the
interaction of the sulfate head of the SDS and the gold slide would be broken, DS would be released into the solution. The first curve in Fig 5 is the capacitance measurement of the gold slide with the same procedures as of Fig 4 and Fig 5, and the second curve is the capacitance measurement after the applied potential was turned off for 30 minutes. After this time, the capacitance increased to a level similar to the initial capacitance measurement with only KNO₃ solution. This experiment concluded that when the applied potential was discharged, the DS surfactant layer that was formed on the gold slide was released into the solution. Also it could be said that the increase in capacitance meant the gold slide is eligible to be used as a base surface for a new surfactant layer, because it regained its ability to hold an electrical charge.

Fig 6. Capacitance measurement with different SDS concentration
Fig 7. Capacitance after 80 cycles by different SDS concentration

To investigate the different surface surfactant layer formats on the gold surface, experiments with different SDS concentrations were performed on various concentrations of SDS up until two times the critical micelle concentration of SDS. Over the concentrations of 1 mM to 4 mM, the capacitance of the gold surface decreased. But at concentrations greater than 4mM, the capacitance of the surface started to increase. (Fig 6, Fig 7). From the results it was assumed that at concentrations lower than 4 mM, a hemimicelle layer of SDS surfactant started to form on the gold slide. At 4 mM the SDS hemimicelle surfactant momolayerlayer became saturated and caused the excess SDS to form a surfactant layer on top of the existing SDS hemimicelle surfactant layer to form an admicelle SDS surfactant layer on the gold slide. [10] The admicelle layer formation resulted in an increase in capacitance. In the hemimicelle layer of SDS, the hydrophobic chain of the surfactant faced the solution. However for the admicelle layer of SDS, the hydrophilic heads faced the solution in a bilayer. With this assumption for future
experiments 4 mM SDS was used for the SDS surfactant layer formation, because the gold slide with a SDS surfactant layer formed with 4 mM SDS is believed to give a hemimicelle layer, which would have a much higher probability of 2-naphthol to be adsorbed to the SDS surfactant layer.

Coating of the frit

Fig 8. SEM image of a stainless steel wool
Fig 9. Magnified SEM image of the area marked on Fig 8.

Fig 10. Elemental analysis of area 1 in Fig 9.
Before proceeding on to experiments to coat various materials for preconcentration experiments, experiments were done on stainless steel wool to verify the method for coating. The SEM image of the stainless steel wool from Fig 9 was magnified. According to the magnified image, it was observed that there were two distinctive surfaces: A dark surface, and a layer of bumps on top of the dark surface. (Fig 8) Elemental analysis was done with the SEM on area 1 and area 2 as shown in Fig 9. The dark surface marked as area 1 showed a great abundance of Fe and Cr, which are the main elements contained in stainless steel. The bright bumps on top of the dark surface have been marked as area 2. For this area, it resulted in a very high abundance of Au. With these SEM images the coating method was confirmed to produce gold a coating on stainless steel, and the observed bright bump that was formed on top of a dark surface was the gold coated on the stainless steel frit. (Fig 10, 11)
Fig 12. SEM image of the clean frit.

Fig 13. SEM image of the frit after the coating procedure.
Fig 14. Elemental analysis of give area of the SEM image in Fig 13.

With the success in coating gold onto stainless steel wool, further investigations were done on a HPLC solvent filter frit. The reason for selecting a HPLC solvent filter frit was because of the pores present in the frit that provide a higher surface area compared to a planar surface. The surface area was measured at the Institute for Combustion Science and Environmental Technology (ICSET). The sample was broken into 5 mm particles and then analyzed by a Micromeritics ASAP 2020 to evaluate the surface area by N\textsubscript{2} adsorption isotherms at 77 K and applying the BET equation in the relative pressure range of 0.05-0.3. As result a surface area of 0.28 m\textsuperscript{2}/g was calculated. The weight of the frit was measured at 8.8723g and gave a total surface area of 2.48 m\textsuperscript{2}. The frit showed a 8000 fold increase of surface area compared to the gold slide.

The uncoated frit had a clean and a flat surface with numerous pores. (Fig 12) However in the picture for the coated frit at a higher magnification, gold particles are show to have formed in lumps across the surface. Fig 14 is the elemental analysis taken with the SEM for the gold coated surface. At point 1 and 3, the analysis shows that gold is the element that is most present, compared to Fe, Cr, and Ni, which are elements that are contained in stainless steel. Point 2 is an area of a pore. (Fig 13, 14)
In addition to the SEM images, weights of the frit were measured to see if the inside of the frit was coated as well. The mass of the frit was measured at three points: Before the coating procedure, after the coating procedure, and after scratching off the gold coated on the surface. The mass of the coated frit was obtained after drying the frit in an oven overnight to remove the solution present in the frit. The gold coating on the frit was scratched off with a spatula until the bare frit surface was exposed and most of the dark yellowish color present on the frit due to the coating was barely visible. Three separate frits were used for this experiment. On average the frit gained 12.7 mg of gold from the coating procedure. Assuming the scratching the coating off the surface can remove more than 95% of the coating resulted from the gold coating procedure there were 4.4 mg difference between the clean frit and the frit with the coating being scratched off. However the statistical calculations with Student’s t test showed that the measurements of
the frits were about the same weight at 95% confidence level. Despite the average shows a weight gain from the gold coating procedure, this result cannot be accepted as a statistically significant result. (Fig 15)

Fig 16. SEM image of a cut section of a gold coated frit
Fig 17. Elemental analysis of box 1 in Fig 16.

Fig 18. Elemental analysis of box 2 in Fig 16.
Fig 19. Elemental analysis of box 3 in Fig 16.

Fig 20. Elemental analysis of box 4 in Fig 16.

To obtain visible evidence and to validate the measured weight increase, a SEM image was taken on a cut section of a gold coated frit. The frit was molded inside with plastic at high pressure so that the pores were completely filled. This was done to protect the coating inside the frit during the bisection. The frit was bisected horizontally with a water saw. From the SEM image of the bisected frit, white spots were observed on the
surface and inside of the bisected frit. (Fig 16) For further validation an elemental analysis was conducted on the dark surface and the white spots inside the frit, and a high abundance of Au was measured. (Fig 17, 18, 19, 20)

**Preconcentration**

With the frit obtained from the coating experiment, experiments were conducted to see if this gold coated frit made to hold a DS layer could extract and preconcentrate 2-naphthol.

![Preconcentration of 2-naphthol](image)

Fig 21. Relative 2-naphthol concentration change. 1: Beginning of extraction stage, 2: End of extraction stage, 3: End of release stage

Compared to the initial concentration of 4 ppm 2-naphthol at the beginning of the extraction stage, there was a 33 percent drop of 2-naphthol concentration as measured from the sample taken from the end of the extraction stage. This strongly suggests that
the 2-naphthol is being adsorbed to the frit. The 8000 fold increase of surface area compared to the gold slide provides 2-naphthol molecules more surface area to adsorb onto the DS surfactant layer. However, the 2-naphthol concentration stayed the same even after the potential was turned off in the cell. (Fig 21)

![Graph showing 2-naphthol concentration comparison of preconcentration experiment with blank experiments.](image)

**Fig 22.** 2-naphthol concentration comparison of the preconcentration experiment with blank experiments. 1: Beginning of extraction stage, 2: End of extraction stage, 3: End of release stage

To investigate the cause of this observation, two blank experiments were conducted. With every procedure being the same as the preconcentration experiment, one experiment was done with a frit with no gold coating, and another set of experiments with no added SDS. This was to see which condition is influencing the frit not releasing the 2-naphthol into the solution. Samples were taken from the same point and were compared with the preconcentration experiment. When there was no SDS present in the cell 2-naphthol
concentration dropped only by 7 percent. With no SDS being added, the intended formation of SDS surfactant layer did not occur. (Fig 22) From this experiment it could be said that the SDS surfactant layer is need for the extraction to happen with a great efficiency. When there was no gold coating of the frit, the extraction efficiency dropped to about half compared to the preconcentration experiment. This result implies that some of the SDS can form a hemimicelle layer on the stainless steel surface without gold. However the reaction is unfavorable compared to the whole preconcentration where a stainless steel frit has a gold coated surface. The affinity between sulfate and gold facilitates the DS surfactant layer formation on the gold surface. For all of the experiments the 2-naphthol concentration had a small change during the release stage of the experiment. The reason for not having the intended 2-naphthol concentration increase after the release stage has to be investigated further.

Fig 23. 2-naphthol destruction test 1: With no potential applied, 2: At 0.8 V, 3: At -0.8 V
To investigate if other variables that are preventing the release of 2-naphthol might be present further experiments were conducted. One of the possibilities were if the potential being applied is destroying 2-naphthol. The same set up as the preconcentration experiment was performed, except not using SDS and directly starting with a 4 ppm 2-naphthol solution. Samples were taken at the start of the experiment, after an hour of potential of 0.8 V applied to the frit, and after an hour of potential of -0.8 V applied to the solution. Samples were analyzed with the HPLC and the same methods were used as the preconcentration experiment. The peak area of the 2-naphthol for all three samples are statistically the same. It was concluded that the potential being applied is not destroying or altering 2-naphthol. (Fig 23)

Fig 24. Tube sticking test

Another possibility was if the 2-naphthol was sticking to the Teflon tubing that connects the syringe pump and the frit. To investigate this possibility the syringe pump
was connected with the frit using new Teflon tubing with the same length and diameter of the Teflon tube used for the preconcentration experiment. The 2-naphthol concentration was measured before pumping the solution in and out of the frit, and after an hour of pumping the solution. There was no change in the 2-naphthol concentration. It was concluded that 2-naphthol does not stick onto the Teflon tubing. (Fig 24)

![Graph showing peak areas for first and second rinses](image)

**Fig 25. Frit rinse test with methanol**

In order to investigate the possibility of 2-naphthol being trapped inside the pores, a rinse test was done on the frit after a preconcentration experiment. Three consecutive rinses were done with 2mL of undiluted methanol injected a syringe. The eluent from the rinse was collected and the concentration was analyzed with a HPLC. The peak areas from the first and the second rinse were 20293 and 5334 respectively. The sample obtained from the third rinse did not shown a peak. From this result the concentration of 2-naphthol washed from the first and second rinse were 2.22 ppm and 0.58 ppm, giving 4.44 μg and 1.16 μg respectively. (Fig 25) The sum of these values (2.80ppm) were similar to the concentration
that remained in the frit after extraction (2.71ppm). From the series of tests to investigate the problem of the failure to increase the 2-naphthol concentration, it could be concluded that 2-naphthol does not stick to Teflon tubing or gets destroyed the potential applied to the frit, but is trapped inside the frit.
CONCLUSION

In this study the formation of a DS surfactant layer on a gold surface was demonstrated. The capacitance measurements have shown that the layer was formed, and subsequently by discharging the potential, the surfactant layer is released together with the adsorbed analyte. To obtain a monolayer of DS surfactant layer the capacitance of the gold slide was monitored as the SDS concentration was varied. 4mM SDS for the SDS surfactant layer formation step was studied due to the lowest capacitance measurement indicative of a hemimicelle surface formation. Various materials in different forms were investigated to increase the surface area of the SDS layer for better performance of extraction. With a Coating gold onto the surface of a porous HPLC mobile phase solvent filter frit was successful. Preconcentration experiments were conducted with this frit and 2-naphthol concentration changes with HPLC were measured. The frit exhibited a 33% extraction of 2-naphthol. However the expected increase in 2-naphthol concentration did not occur. Several control experiments were conducted to see if a variable was affecting this problem. However these experiments proved inconclusive. Further study is necessary to model the chemical environment difference of the SDS surfactant layer in plainer surface and porous surface, and find a solution to facilitate the release the 2-naphthol into the solution after the preconcentration procedure.


8. Li, L.; Huang, Y.; Wang, Y.; Wang, W.; Hemimicelle capped functionalized carbon nanotubes-based nanosized solid-phase extraction of arsenic from environmental water


