Systematic Parameter Screening for Capillary Electrophoresis Monitoring of Surfactants on Silicon Wafer Surfaces by Designed Experiments

S.C. Hoo[1], T. Wen[1], S.F.Y. Li[2,*], and **Th. Ehmann[3]**

[1]Siltronic Singapore, Metrology, 10 Tampines Industrial Ave 5, Singapore 528820; [2]Department of Chemistry, National University of Singapore, 3 Science Drive 3, Singapore 117543, Republic of Singapore; and [3]Siltronic AG, Metrology, Johannes-Hess-Straße 24, 84489 Burghausen, Germany

Abstract

The surface purity of silicon wafers is an important parameter to monitor for yield improvement of semiconductor devices in a production line. Surfactants are used to reduce the surface potential in order to facilitate the removal or cleaning of particles and metals. Traces of surfactant residues from the cleaning bath may still be present on the wafer surface after the final cleaning step. In this report, two capillary electrophoresis (CE) methods for the analysis of dodecylbenzene sulfonate (DBS) are developed for monitoring the surfactant residues in the wafer manufacturing process. One method is developed for the sensitive determination of all DBS homologues and isomers in one single peak. Another method is developed for the fingerprint analysis of the homologues and isomers of DBS. The Taguchi methodology was used as a systematic optimization tool for the DBS analysis by CE. The experiments were evaluated by calculating the signal-to-noise ratio values with four responses. The lowest detection limit for DBS was 15 µg/L at 95% confidence level. The percent recovery of surfactant was between 90% and 110%.

Introduction

Surface preparation is one of the many key processes in a wafer manufacturing plant. Good quality wafers undergo a series of cleaning processes designed to maintain surface purity by removing contaminants such as metals and particles. Surfactants are used to enhance the wettability of the wafer surface, thus improving the removal efficiency of contaminants. However, traces of surfactant retained on the wafer surface will cause detrimental effects such as increased leak current and poor gate oxide performance of electronic devices (1). Therefore, a routine method for monitoring surfactants is necessary. Dodecylbenzene sulfonate (DBS) was chosen as a model surfactant because anionic linear alkyl benzene sulfonates (LAS) are widely used in surfactant formulations because of their biodegradability. LAS can be determined by chemical titration analysis methods and separation techniques such as electrophoresis using aqueous gel (2), gas chromatography–mass spectrometry (3,4), high-performance liquid chromatography (5), and capillary electrophoresis (CE) (6–13). CE is becoming more widely used as a microanalytical technique, especially for anions. Compared with other chromatographic techniques, CE offers several advantages for this purpose (e.g., low consumption of chemicals and analytes, rapid separation, and high resolution). Moveover, CE is easy to operate, automated, and robust. Routine analysis for anions and cations on silicon surfaces has been successfully achieved using CE techniques (13,14).

The systematic statistical approach to design experiments developed by Genichi Taguchi et al. (15–17) has been accepted widely and used in many companies to characterize and to optimize complicated multiresponse processes with a minimum of experiments (18–22). The reduction of time-consuming tests not only increases productivity, but also produces savings in materials and manpower costs and reduces wasted materials (19). Taguchi used a systematic statistical approach to design experiments for robust products or processes (20). It is based on quality engineering principles in which experiments are performed on product or process designs rather than on process operation (21). When dealing with simultaneous optimization of more than one response in the same process, this required "engineering judgments" on the confirmation results.

In this report, a systematic approach based on Taguchi's method was used to develop methods based on CE for DBS analysis to monitor surfactant residues on wafer surface. Two methods are developed in one design of experiment. One method is designed to serve as a fingerprint analysis of the homologues and isomers in the qualitative analysis of DBS on silicon wafer surface, and the other method is to produce a single compound peak for quantitative analysis of all the residual DBS present on the silicon wafer surface. Based on the quantitative single-peak

* Author to whom correspondence should be addressed: email chmlifys@nus.edu.sg.

method developed, the limit of detection (LOD) is calculated at a 95% confidence level.

Experimental

Apparatus

All designed experiments were carried out with an HP3D CE instrument from Agilent Technologies (Waldbronn, Germany) at a constant temperature of 20°C. Fused silica capillaries (50-µm i.d, 350-µm o.d.) were obtained from Polymicro Technologies (Phoenix, AZ). The length used in this experiment was 64.5 cm (56 cm to the detection window). Detection wavelength was at 194 nm with a bandwidth of 6 nm.

Reagents

Chemicals were purchased from various suppliers and were of analytical grade or better. The ultrapure water (UPW) fulfilled the requirement of the SEMI F61-0301 guidelines for pure water in semiconductor processing (i.e., cationic contamination less than 5 ng/L and anionic contamination less than 20 ng/L) (22). Dodecylbenzene sulfonate (DBS) sodium salt was obtained from Fluka (Seelze, Germany). Quality control (QC) standard DBS was obtained from Aldrich (Bellefonte, PA). Both α- and β-cyclodextrin (CD) were obtained from Wacker Chemie (Munchen, Germany). All buffer solutions and standards were prepared in a cleanroom of ISO Class 4 (ISO 14644-1 by ISO Technical Committee 209, 1999). The pH values of all prepared buffer solutions were measured with a daily calibrated pH meter (Mettler Toledo M235, Columbus, OH). Anionic surfactant used in the wafer fabrication process was diluted 100 times from stock for further analysis. The wafer sample was extracted by wetting the entire surface with 2 mL of UPW.

Procedures

Taguchi methodology: L18 layout

The methodology of Genichi Taguchi was applied for the design of screening experiments because of its advantages of the inherent robustness and reduced number of necessary experiments for investigating parameters at more than three different levels (15–21). Taguchi L18 refers to an orthogonal design with 18 experiments for optimization using seven control factors with three levels and only one factor with two levels.

The experimental screening matrix is given in Table I, which summarizes the parameters and their levels. In the Taguchi methodology, the optimization of a process involves determining the best control factor levels so that the output will be at the target value. Continuous experimental data can be processed and converted to signal-to-noise ratios (s/n) (23), which are log functions of desired outputs. The contribution of factors assigned to the inner array of the orthogonal array is considered as signal, and all other factors are considered as noise, which are present in the process but have no effect on the output. The relationship between signal, noise, output, and control factors is illustrated in Figure 1.

The experimental screening matrix was superimposed with a noise factor, which takes into account a wafer surface preparation step known as vapor phase decomposition (VPD) (24). In a VPD preparation, the silicon wafer surface is exposed to gaseous hydrofluoric acid (HF) in order to dissolve the native oxide of the wafer surface. It is assumed in dissolving the native oxide, the van der Waals interaction between polar compounds such as surfactants and the wafer surface was eliminated, allowing the surfactants to be removed from the surface.

Several s/n are commonly calculated in the Taguchi methodology (15–21): (*i*) s/n for larger-the-better (LTB) is computed based on equation 1, where y_i is the raw data corresponding to a particular control factor, and n is the number of experiments carried out at this control factors combination.

$$\text{s/n} = -10 \times \log 1/n \times \sum_{i=1}^{x} 1/y_i^2 \qquad \text{Eq. 1}$$

(*ii*) s/n for smaller-the-better (STB) is computed based on equation 2.

$$\text{s/n} = -10 \times \log[1/n \times \sum_{i=1}^{x} y_i^2] \qquad \text{Eq. 2}$$

(*iii*) s/n for nominal-the-best is computed based on equation 5, where sensitivity (S_m) and sample variation (V_e) are calculated using equations 3 and 4, respectively.

$$S_m = 1/n \times [\sum_{i=1}^{x} y_i^2] \qquad \text{Eq. 3}$$

$$V_e = 1/n\ [\sum_{i=1}^{x} (y_i - y)^2 \qquad \text{Eq. 4}$$

$$\text{s/n} = 10 \times \log[(S_m - V_e)/(n \times V_e)] \qquad \text{Eq. 5}$$

where:

$$y = \sum_{i=1}^{x} y_i/n \qquad \text{Eq. 6}$$

The s/n value for each response factor is calculated depending on individual response factor. For example, in the analysis for fingerprint of DBS, the larger the number of peaks, the better the separation. In this case, LTB calculation is chosen to compute the s/n value. The responses monitored in this report are given in the following list: number of peaks, LTB for a unique finger print pat-

Table I. Parameters and Their Levels of the Screening Experiment for DBS*

Parameter	Level 1	Level 2	Level 3
A Type of electrolyte	Phosphate	Borate	–
B Conc. of electrolyte (mmol/L)	25	50	75
C pH	7.0	8.0	9.0
D Type of organic solvent	Acetonitrile	Methanol	THF
E Conc. of solvent (%)	0	10	25
F Conc. of SDS (mmol/L)	0	25	50
G Electrical field (V/cm)	233 (15 kV)	349 (22.5 kV)	465 (30 kV)
H CD	0	10 mmol/L α-CD	20 mmol/L β-CD

* Noise matrix c(HF) (mmol/L): 0, 0.5, 1.0, 5.0, and 10.

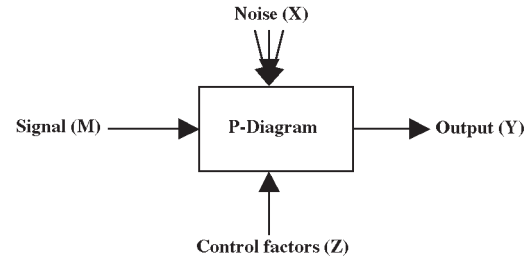

Figure 1. The relationship between signal, noise, output, and control factors is illustrated in the P-diagram, where P is the process or product.

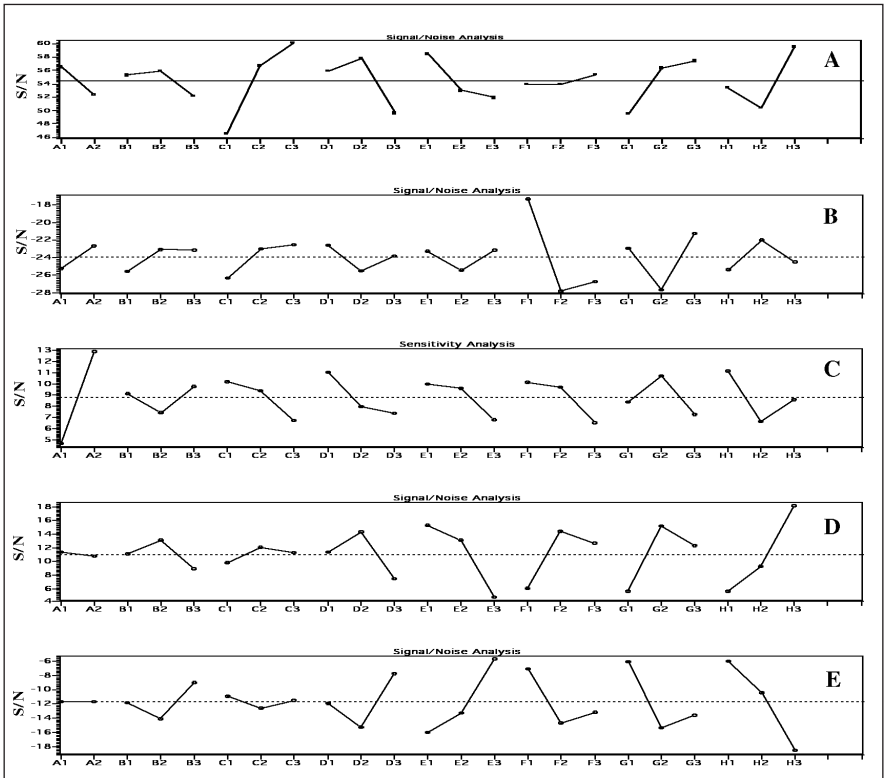

Figure 2. Result of s/n values calculated by analysis of variance for the screening of parameters for DBS: response factor for area, LTB (A); migration time, STP (B); and symmetry, nominal the best (C). Response factor for number of peak, LTB (D) and fingerprint analysis and single peak analysis, STB (E). The line (—) represents the average s/n values. For the *y*-axis, A, B, C, D, E, F, G, and H represent the eight parameters and 1, 2, and 3 represent the three levels (see Table I).

tern; number of peaks, STB for all homologues and isomers in single-peak quantitation analysis; corrected peak area, LTB for sum of all peaks of homologues and isomers evaluated (the higher the total peak area, the higher the sensitivity); symmetry of (last) peak, nominal-the-best for both finger print analysis and single peak analysis to determine whether there was overloading of the electrolyte system; migration time of (last) peak, STB for a faster separation.

Parameter design of experiments

Large test arrays of experiments were carried out to provide an overall view of the analysis method. The set of experiments (Table I) was the so-called L18 with the combination of one factor at two levels and seven factors at three levels. A total of 18 experiments were designed in which each column of the array contains up to three levels for every factor. Each array row represents a factor combination. A noise factor was included becasue the real sample may contain some HF.

Results and Discussion

Effect of parameters in DBS analysis

The parameters influencing separation and detection of anions by CE are well-documented (25–40). The main parameters affecting the performance of an appropriate electrolyte system are the type of electrolyte and its concentration; pH and additives, solvents, or chemicals used to improve the overall stability of the system; and good separation of the anions analytes.

The silanol groups of a capillary are ionized by a high pH electrolyte replacing Si-OH with $Si\text{-}O^-$ at the inner surface of the capillary. The positive cations will be attracted to the capillary, forming the first layer called the fixed layer, and the mobile layer is the diffuse double layer. As an electric field is applied across the capillary, the mobile layer is

Table II. Tabulation of the Choices of Factor Levels for the Best Combination of s/n Values and Mean Values

	A Type of electrolyte	B Conc. of electrolyte (mmol/L)	C pH	D Type of organic solvent	E Conc. of solvent (%)	F Conc. of SDS (mmol/L)	G Electrical field (kV)	H CD
Area	1	1 or 2	3	2	1	2 or 3	3	3
MT	2	2 or 3	2 or 3	1 or 3	1 or 3	1	3	2
Symmetry	2	1 or 3	1 or 2	1 or 2	1 or 2	1 or 2	2	1
No. of peak (LTB)	1	1 or 2	2 or 3	2	1	2 or 3	2 or 3	3
No. of peak (STB)	1	1 or 3	1 or 3	1 or 3	3	1 or 2	1	1
Choice for "fingerprint"	2	1	3	1	1	2	3	3
Choice for one peak	2	1	3	1	1	1	3	1

pushed toward the negatively charged cathode, resulting in an electroosmotic flow (EOF) (i.e., "electrically-driven pump"). Thus, the change of pH value will have an effect on EOF (26). At low pH (4,5), the EOF is nearly suppressed. At higher pH (8,9) the EOF mobility is increased, hence reducing the analysis time.

Phosphate and borate buffers are commonly and successfully used as electrolytes for the separation of alkyl benzene sulfonates (27–31). Phosphate and borate differed in their buffering range, affinity to the capillary wall, and contribution to Joule heat. Increases of buffer concentration lead to increased migration times and peak resolutions of the DBS isomers because of the slower EOF. The concentrations of the electrolytes influenced Joule heat and electrodispersion. Joule heat increases with increasing electrolyte concentration. However, the effect of electrodispersion decreases because the disturbance of the sample zone by the electrical field of the electrolyte can be neglected.

Organic solvents were added to modify the resolution of mixtures of surfactants because the micelle formation and the interaction between surfactant molecules were reduced and suppressed (32). The degree of influence depended on the type of solvents (25). Organic solvents added to the electrolyte may also reduce the EOF mobility, thus increasing peak resolution. It may also reduce the conductivity of the electrolyte, therefore decreasing Joule heat. However, the organic solvent may result in a higher tendency for bubble formation within the capillary while applying high electrical fields.

Sodium dodecylsulfate (SDS), an anionic nonchromophoric surfactant, was added to the electrolyte in order to introduce an interaction with the alkyl chains of the analyte surfactant to improve peak resolution (33–35). The solvent concentration affected not only the formation of the analyte surfactant (dodecylbenzene sulfonate) micelle, but also the interaction of the additive surfactant (i.e. SDS) with the analyte surfactant. Anionic analyte surfactant has the tendency to interact with SDS rather than with the solvent. This association is called solvophobic interaction (33). Different lengths of hydrophobic alkyl chain linked to the phenyl ring of alkylbenzene sulfonate forms association complexes of different hydrophobicity and different electrophoretic mobilities. This produced good separation of isomers of DBS homologous. At lower concentration of organic solvent, the association between the SDS monomer and the anionic surfactant is preferred (33). Thus, these interactions improve peak resolution.

CD is used as a steric selector in many applications (8,36–39). The isomers and homologues of DBS are resolved because of the formation of inclusion host–guest complexes with DBS molecules. The long alkyl chain of DBS could interact with the small hydrophobic cavity of α-CD, while the aromatic ring could interact with the larger cavity of β-CD. The addition of CDs was reported to reduce or disrupt surfactant aggregate or micellization in the buffer system and capillary surface (8).

Table III. Optimized System for Fingerprint Analysis

Factor level	Factor name	Choice level
A2	Type of electrolyte	Borate
B1	Conc. of electrolyte (mmol/L)	25
C3	pH	9.0
D2	Type of organic solvent	–
E1	Conc. of solvent (%)	0
F2	Conc. of SDS (mmol/L)	25
G3	Electrical field (kV)	30
H3	Cyclodextrin (mmol/L)	20 β-CD

Table IV. Optimized System for One-Peak Analysis

Factor level	Factor name	Choice level
A2	Type of electrolyte	Borate
B1	Conc. of electrolyte (mmol/L)	25
C3	pH	9.0
D1	Type of organic	–
E1	Conc. of solvent (%)	0
F1	Conc. of SDS (mmol/L)0	
G3	Electrical field (kV)	30
H1	Cyclodextrin (mmol/L)	0

Optimization of buffer system by Taguchi L18 layout

Figure 2 shows the results obtained from the 18 experiments. The sum of peak area and migration time could be improved by increasing the pH value to 9.0. The higher pH resulted in higher EOF mobility. Consequently, DBS interacted more strongly with the surface of capillary rather than with the electrolyte. This hampered the separation of homologues and isomers. At high pH, peak symmetry was poorer, but the number of peaks was not affected. Considering peak symmetry, borate showed a better performance than phosphate. The concentration of the electrolyte has one of the lowest effects on the observed responses. A lower concentration was chosen to minimize the potential problem of Joule heat. Peak area was increased without the addition of organic solvent (SDS and CD) because DBS, which was moved along with EOF, was not resolved from its homologues and isomers. This also corresponded to the response criterion STB for the number of peaks response factor. Without any additives, a shorter analysis time was obtained in which the isomers and homologues were combined into nearly one peak and, thus, apparently improved the sensitivity. Adding SDS and CD lengthened analysis time, but it produced better resolution of the

Table V. Summary of the Parameter Settings for Fingerprint and Single Peak DBS System

System	Injection	Length capillary/i.d.	Electrolyte	Conc. of electrolyte	Volt (kV)	UV λ (nm)	pH	SDS (mmol/L)	Temp (°C)	CD (mmol/L)
Fingerprint	50 mbar at 40 s	64.5 cm/50 µm	Borate	25 mmol/L	30	194	9	50	20	β-CD 20
Single peak	50 mbar at 40 s	64.5 cm/50 µm	Borate	25 mmol/L	30	194	9	0	20	0

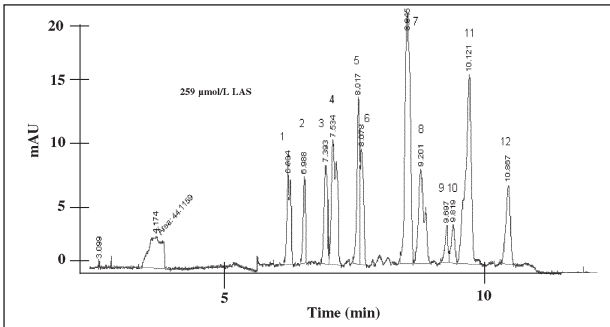

Figure 3. Conditions were: 25 mmol/L borate, pH 9, 50 mmol/L SDS, 20-b-CD, 50 mbar at 40 s, 64.5 cm × 50 µm i.d., 30 kV, 20°C, 194 nm (for parameters, see Table V). The sample was 250 µmol/L DBS.

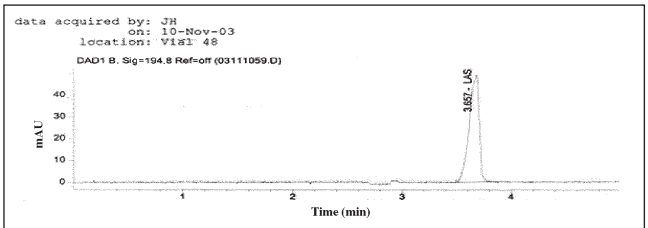

Figure 4. Conditions were: 25 mmol/L borate, pH 9, 30 kV, 50 mbar at 40 s, 64.5 cm × 50-µm i.d., 20°C, 194 nm (for parameters, see Table V). Sample was 50 µmol/L DBS.

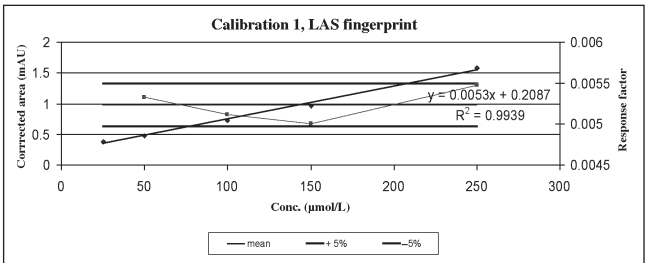

Figure 5. A linear calibration curve for DBS as "fingerprint". The left axis shows the total absorbance of the area under the all the peaks. The linearity is from 50 to 250 µmol/L, and R^2 is 0.9939. The right axis shows the response factor of the absorbance peak areas against the concentrations. These responses are limited to ± 5% deviation. The lowest concentration within 95% confidence is 50 µmol/L.

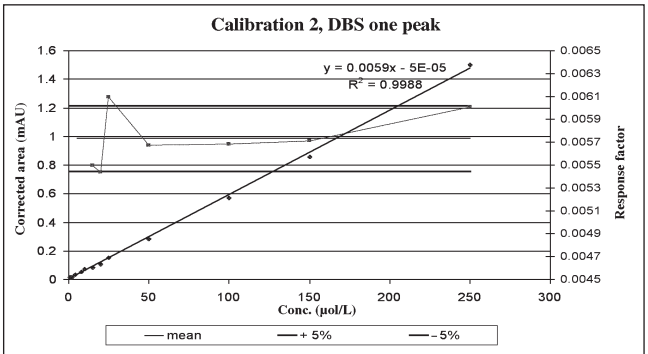

Figure 6. A linear calibration curve for DBS as "one peak" method. The left axis shows the absorbance of the single peak corrected area in mAU. The linearity is from 50 to 250 µmol/L, and R^2 is 0.9988. The right axis shows the response factor of the absorbance peak areas against the concentrations. These responses are limited to 5% deviation. LOD within 95% confidence is 50 µmol/L.

isomers and homologues. In the absence of organic solvent, SDS forms a more stable association complex with the hydrophobic dodecyl chain because of stronger solvophobic interaction with the hydrophobic micellar core, while β-CD, with its large cavity, forms an inclusion complex more effectively with the aromatic ring of DBS, resulting in better resolution of the isomers and homologues. This offered some benefits for "fingerprint" analysis. In an optimized system, all effects were carefully balanced to obtain an optimum performance. The choices of parameters for analyses of DBS as "fingerprint" and as single-peak are summarized in Table II. In Table III and IV the optimized systems of "fingerprint DBS" and "single-peak DBS" are summarized, respectively.

DBS fingerprint analysis

Confirmation analyses were performed in order to validate the chosen levels of parameters. Table V summarizes the testing parameter settings for both fingerprint and single-peak systems. Figure 3 shows an electropherogram of a standard of DBS for fingerprint analysis. Based on this optimized system, the linear curve shown in Figure 4 was obtained with standards of DBS ranging from 25 to 250 µmol/L. The left axis displays the absorbance of the single-peak area in mAU. The linearity is from 50 to 250 µmol/L; R^2 is 0.9939. The right axis displays the response factor of the absorbance peak areas against the concentrations. These responses were limited to ± 5% deviation. The LOD within linearity of 0.9988 at 95% confidence was 50 µmol/L (15 µg/mL), and the limit of quantitation (LOQ) was 150 µmol/L (45 µg/mL). The percent recovery of the QC standard of DBS (50 µmol/L) was within 100% ± 10%. The recovery of DBS standard obtained from Aldrich at a concentration of 15 µg/mL was within 100% ± 10%.

Single-peak DBS analysis

In Figure 5, a running buffer system is shown in which all homologues and isomers of DBS were analyzed as one single peak. This could be explained by the absence of organic solvent, SDS and CD. These additives improved peak resolution by interacting selectively with the homologues and isomers of DBS. Without these additives the homologues and isomers were not resolved and migrated as one single peak. For the optimized single-peak buffer system, a linear calibration curve was obtained as shown in Figure 6. The DBS concentration ranged from 2.5 µmol/L to 250 µmol/L (2.5, 5, 10, 25, 50, 100, 150, 200, and 250 µmol/L). Similar to Figure 5, the left axis displays the absorbance of the single-peak area in mAU, while the right axis displays the response factors at ± 5% deviations of the absorbance peak areas against the concentrations. The linearity is from 50 to 250 µmol/L, and R^2 is 0.9988. Thus, LOD within the linearity of 0.9988 at 95% confidence is 50 µmol/L (15 µg/mL). Similarly, the percent recovery of the QC standard of DBS (50 µmol/L) in the single-peak system was also within 100 ± 10%.

Wafer sample and recovery

A 200-mm silicon wafer was extracted with 2 mL UPW by completely wetting the surface and swiveling the wafer for 2 min. A sample of 900 µL was taken from the wafer surface and 100 µL of 10 µmol/L of HF was added. Another wafer from the same batch

lot was intentionally contaminated with 15 µg/mL DBS by completely wetting the surface with 2 mL of spiking solution. The wafers are dried in a class 4 (ISO 14644-1) cleanroom over night. Samples were analyzed by using the single-peak electrolyte system, and no DBS was detected on the sample wafer surface. The percent recovery from the spiked wafer was in the range of 100% ± 10%.

Conclusion

A systematic screening and optimization study was performed for CE determination of alkyl benzene sulfonates by applying the methodology of G. Taguchi. Seven parameters at three levels and one parameter at two levels were investigated by an array of 18 experiments. By using different response criteria, different electrolyte systems could be obtained for different purposes without any additional work. One electrolyte system was developed for the sensitive determination of all DBS homologues and isomers in one single peak. Another electrolyte system was also developed for the fingerprint analysis of the homologues and isomers of DBS. Quantitative analysis of an unknown technical grade DBS sample was performed. The LOD of these two systems are 15 µg/mL of DBS and the percent recovery for the QC standard and spiked samples was in the range of 90–110%, which proved the applicability of the method.

If any surfactant residues remain on the bare Si wafer surface after cleaning, the contamination will be in the ultratrace analytical concentration range. In a future designed experiment, the quantitation limit must be further reduced.

Acknowledgments

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