Determination of the Optimal Chemical Dosing Concentration in Reverse Osmosis Using Design of Experiments: A Case Study of Semiconductor Fabrication Factory in Chacheongsao, Thailand

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***Abstract*— The objective of the study was to explore the effects of chemical dosage concentration with respectively to specification of treated Reverse Osmosis water and imply to simulate the optimal amount of chemical in the Reverse Osmosis (RO) process. The study was implied Water Treatment Plant in one semiconductor fabrication in Chacheongsao, Thailand as the case study. The experiment was conducted in the real pilot scale by variation of chemical concentration. Design of Experiment was used to evaluate in term of Analysis of Variance in order to validate the obtained result. Conductivity and % Salt Rejection are key performance indicators. The results developed the optimal range of treated chemical concentration of 10% Antiscalant with 10% Microbiocide solution with RO water conductivity in the range of 5.05-8.51 µS/cm with % Salt rejection in the range 95.12-98.52. The optimal condition could operate approximately to the calculated value from Excel Solver. This condition could reduce feeding chemical consumption amount and help monitoring performance of RO unit.**

***Keywords*— Reverse Osmosis, Antiscalant, Microbiocide, Conductivity, Analysis of Variance**

I. INTRODUCTION

The continuous and accelerated development of information and communication technology is affected to the growth of semiconductor manufacturing industry. In manufacturing semiconductor products, semiconductor chips are performed on silicon wafer substrate involving oxidation, diffusion, ion implementation, deposition of conductors and insulators, photolithography and etching [1]. During the fabrication, chips may contaminate by various impurities e.g. chemical solution, machine, operator, thus the chips are particularly being cleaned prior undergoes to assembly as one part of electronic devices. Cleaning process is one of the critical process in order to rinse the impurities from chips, thus the quality of rinsing water is significantly to be qualified with respectively to chip specification requirements. Ultrapure water is typically consumed in semiconductor manufacturing according to the specification defined in term of resistivity of 18 Megaohm.cm as the inverse of conductivity value [2, 3].

In this research, Ultrapure Treatment Plant of one Semiconductor Fabrication Factory in Chacheongsao province, Thailand was implied as a case study. The treatment processes are consisted of Pretreatment process and Desalination process. Pretreatment process is fundamental process of water treatment, consisted of Sand Filtration to trap suspended solid, Carbon Filtration to absorb colour, odour and organic substance and Cartridge Filter Housing to trap suspended particulate matter before undergoes to Desalination process. [4, 5]. Desalination process is correspondingly a fundamental process for Ultrapure water production, the processes are consisted of Reverse Osmosis and Ion Exchanger [2]. Reverse Osmosis or RO is purified water by rejecting soluble molecules, ions and biological substance by semipermeable membrane, purified water from RO undergoes to Ion Exchanger to remove ion containment in water as significantly affect to Resistivity of water and eventually become Ultrapure water supplying to manufacturing process.

In this research, Reverse Osmosis or RO was studied. As the fundamental treatment of Ultrapure water, RO is rejected chemical and biological substances by semipermeable membranes at high driving pressure with aided dosing chemical solutions. Product or RO water has lower ionic content than feeding water referred in term of conductivity. Chemical solutions are Antiscalant for controlling scale precipitation and Microbiocide for inhibiting the growth of microorganism on membrane [6,7]. After operating for a period of time, scale and slime could be formed on RO membrane. This effect could result in decreasing in flux, operation performance, and RO water quality. To mitigate this effect, the concerned factors are reasonable design, proper treatment and correct operation [8]. In this study RO water treatment with chemical dosing solution, Antiscalant and Microbiocide were implied to evaluated.

The objective of the research is to explore the effects of dosing concentration of Antiscalant and Microbiocide with respectively to quality of RO water which defined in the term of conductivity (µS/cm) and correspondingly optimize the appropriated chemical dosing concentration for process improvement and cost reduction. Conductivity and pH of feeding water and RO water are monitored with automatically driving pressure from RO unit. The experimental results were evaluated by using Design of Experiment concept.

II. EXPERIMENT AND METHOD

The actual operation of RO Unit was investigated. Factory's Treatment Plant consumes water from the industrial estate reservoir as feeding water to the system. The measurement of feed water and RO water was daily conducted, concerned parameter are pH and conductivity. pH was measured by pH meter (Yokogawa brand, model: PH72), and Conductivity was measured by Conductivity meter (Oakton brand, model: CON400). RO Membrane used for this study was a brackish water RO Membrane from Dow Filmtec. Commercial Antiscalant (pH range: 2.2 – 4) and Microbiocide solution (pH range: 8.5-9.5) were prepared and adjusted at 5%,10%,15%,20%,25% and 30% concentration by volume. Supplied feed water had pH in the range of 6.15-8.56 and conductivity at 216-369 µS/cm, the system was controlled driving pressure at 4 Bar by RO Unit controller. A schematic diagram of the experiment is shown in Fig.1

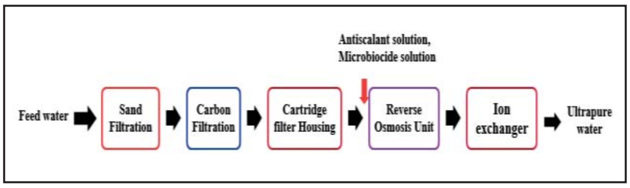

Fig. 1 Experimental apparatus

A. The Study of % Antiscalant dosing concentration variation

The experiment was conducted by varying dosing concentration of Antiscalant into 5%,10%,15%,20%,25% and 30% by volume with dosing constant concentration of Microbiocide at 20% by volume. Analysis of Variance (ANOVA) was used to analyse the test result by Minitab 16 software as shown in Fig.2 and Fig.3.

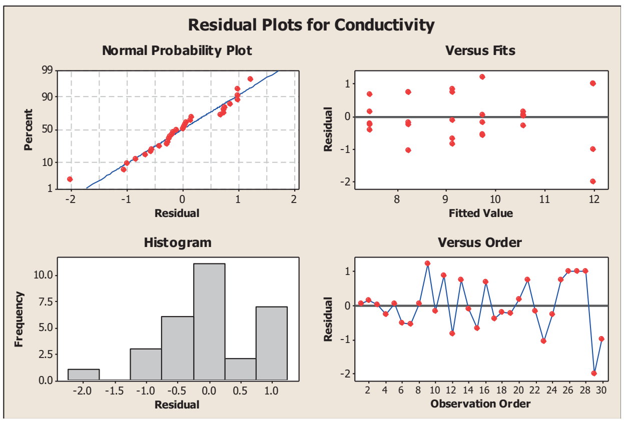

Fig. 2 The Residual Plot for Conductivity Value with respectively to variation concentration of % Antiscalant dosing solution.

One-way ANOVA: Conductivity versus % Antiscalant

```
Source        DF      SS      MS      F      P
% Antiscalant  5  67.654  13.531  20.45  0.000
Error         24  15.881   0.662
Total         29  83.536

S = 0.8135   R-Sq = 80.99%   R-Sq(adj) = 77.03%

                              Individual 95% CIs For Mean Based on
                              Pooled StDev
Level  N    Mean  StDev  --------+---------+---------+---------+-
 5     5  10.568  0.157                      (----*----)
10     5   9.728  0.731                (----*---)
15     5   9.110  0.788           (----*----)
20     5   7.422  0.439  (---*----)
25     5   8.220  0.760       (---*----)
30     5  11.996  1.421                               (----*----)
                         --------+---------+---------+---------+-
                               8.0       9.6      11.2      12.8

Pooled StDev = 0.813


Grouping Information Using Tukey Method

% Antiscalant  N     Mean  Grouping
30             5  11.9960  A
 5             5  10.5680  A B
10             5   9.7280    B C
15             5   9.1100    B C
25             5   8.2200      C D
20             5   7.4220        D

Means that do not share a letter are significantly different.
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Fig. 3 Result from ANOVA for %Antiscalant dosing concentration variation.

According to the result of Fig.2 and Fig.3, it was found that at significance level of 0.05, % Antiscalant dosing concentration was significantly affected conductivity of RO water (P-value < 0.05). The residual plot was distributed nearly in left-skewed and independently fitted.

B. The Study of % Microbiocide dosing concentration variation result

The experiment was conducted by varying dosing concentration of Microbiocide into 5%,10%,15%,20%,25% and 30% by volume with dosing constant concentration of Antiscalant at 20% by volume. Analysis of Variance (ANOVA) was used to analyse the test result by Minitab 16 software as shown in Fig.4 and Fig.5.

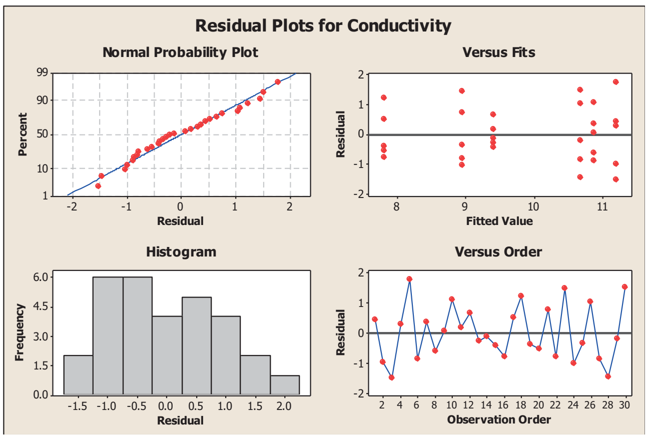

Fig. 4 The Residual Plot for Conductivity Value with respectively to variation concentration of % Microbiocide dosing solution.

One-way ANOVA: Conductivity versus % Microbiocide

```
Source         DF      SS      MS     F      P
% Microbiocide  5  44.031  8.806  8.94  0.000
Error          24  23.644  0.985
Total          29  67.675

S = 0.9926   R-Sq = 65.06%   R-Sq(adj) = 57.78%


                                 Individual 95% CIs For Mean Based on
                                 Pooled StDev
Level  N    Mean  StDev  ----+---------+---------+---------+-----
 5     5  11.204  1.299                       (-----*-----)
10     5  10.878  0.786                    (------*-----)
15     5   9.398  0.429            (-----*-----)
20     5   7.796  0.843  (-----*-----)
25     5   8.938  1.066        (------*-----)
30     5  10.668  1.255                   (-----*-----)
                         ----+---------+---------+---------+-----
                           7.5       9.0      10.5      12.0

Pooled StDev = 0.993


Grouping Information Using Tukey Method

% Microbiocide  N     Mean  Grouping
 5              5  11.2040  A
10              5  10.8780  A
30              5  10.6680  A B
15              5   9.3980  A B C
25              5   8.9380    B C
20              5   7.7960      C

Means that do not share a letter are significantly different.
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Fig. 5 Result from ANOVA for %Microbiocide dosing concentration variation.

According to the result of Fig.4 and Fig.5, it was found that at significance level of 0.05, % Microbiocide dosing concentration was significantly affected conductivity of RO water (P-value < 0.05). The residual plot was distributed nearly in right-skewed and independently fitted.

Referred to the results from Experiment No.1 and No.2 implied the residual distribution, the plots were not normally distributed and tended to be skewed. To investigate this phenomena, Experiment No.3 was conducted.

C. The Study of the combination of % Antiscalant and % Microbiocide dosing concentration variation

The experiment was conducted by varying dosing concentration of Antiscalant 10%, 15%, 20%, 25% and 30% by volume with dosing constant concentration of Microbiocide at 10%, and 15% by volume. General Linear Model was used to analyse the test result by Minitab 16 software as shown in Fig.6 and Fig.7.

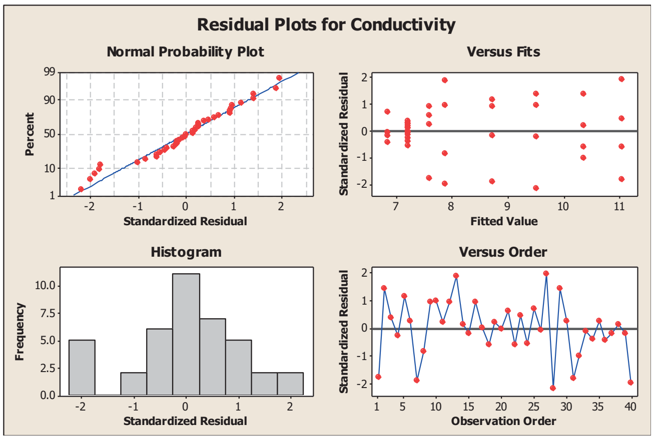

Fig. 6 The Residual Plot for Conductivity Value with respectively to variation concentration of % Microbiocide and % Antiscalant dosing solution.

General Linear Model: Conductivity versus %Antiscalant, %Microbiocide

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Factor        Type   Levels  Values
%Antiscalant  fixed       5  10, 15, 20, 25, 30
%Microbiocide fixed       2  10, 15


Analysis of Variance for Conductivity, using Adjusted SS for Tests

Source                      DF    Seq SS    Adj SS   Adj MS      F      P
%Antiscalant                 4   75.1687   75.1687  18.7922  25.52  0.000
%Microbiocide                1    0.7868    0.7868   0.7868   1.07  0.310
%Antiscalant*%Microbiocide   4    3.5091    3.5091   0.8773   1.19  0.335
Error                       30   22.0907   22.0907   0.7364
Total                       39  101.5553


S = 0.858112   R-Sq = 78.25%   R-Sq(adj) = 71.72%


Unusual Observations for Conductivity

Obs  Conductivity     Fit  SE Fit  Residual  St Resid
 38        7.8900  9.5100  0.4291   -1.6200    -2.18 R

R denotes an observation with a large standardized residual.
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Residual Plots for Conductivity

Main Effects Plot for Conductivity

Fig. 7 Result from General Linear Model Simulation for %Microbiocide dosing concentration variation

According to the result of Fig.6 and Fig.7, it was found that at significance level of 0.05, % Antiscalant dosing concentration was significantly affected conductivity of RO water (P-value < 0.05), whereas % Microbiocide dosing concentration and interaction between % Antiscalant and % Microbiocide dosing concentration was not significantly affected conductivity of RO water (P-value > 0.05). In addition to the residual plot in Fig.6, the residuals were normally distributed with zero mean and nearly constant variance and independently fitted. To prove the relation among these factors, Main Effects Plot and Interaction Plot were shown in Fig.8 and Fig.9.

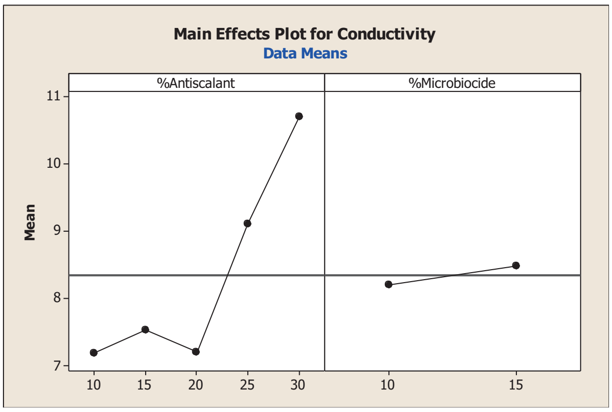

Fig. 8 Main Effects Plot for Conductivity with respectively to % Antiscalant and % Microbiocide.

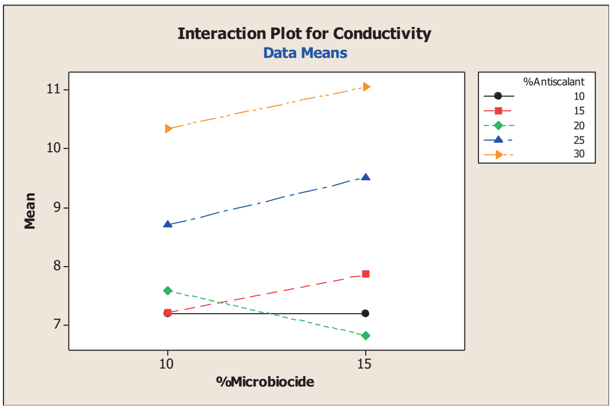

Fig. 9 Interaction Plot for Conductivity with respectively to % Antiscalant and % Microbiocide.

D. *Response Function*

To develop the response function, Regression Analysis was used with the deducting the non significant factor. The model was showned in Fig.10.

Regression Analysis: Conductivity versus %Antiscalant, %Microbiocide

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The regression equation is
Conductivity = 0.222 %Antiscalant + 0.297 %Microbiocide


Predictor          Coef  SE Coef     T      P
Noconstant
%Antiscalant    0.22201  0.02289  9.70  0.000
%Microbiocide   0.29674  0.03810  7.79  0.000


S = 1.30892


Analysis of Variance

Source          DF      SS      MS        F      P
Regression       2  3471.4  1735.7  1013.09  0.000
Residual Error  48    82.2     1.7
Total           50  3553.7


Source         DF  Seq SS
%Antiscalant    1  3367.5
%Microbiocide   1   104.0
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Fig. 10 Response Function of Conductivity with respectively to % Antiscalant and % Microbiocide by Regression Analysis.

The regression equation is

Conductivity = 0.222 % Antiscalant + 0.297 %Microbiocide

E. *Optimal Condition of the Parameters*

To determine the optimal condition by optimizing the parameter in RO Water Treatment, Excel Solver in Microsoft Excel Program was simulated. The optimal condition of these factors are summarized in Table I.

TABLE I: Result of Optimal Condition of Factors in Reverse Osmosis Process

Factors	Optimal Level
% Antiscalant	10
%Microbiocide	10

The dosing of Antiscalant and Microbiocide at 10% concentration by volume are optimal value and could yield RO water conductivity at 5.19 µS/cm.

III. CONCLUSION

To confirm the result, 30 samples were performed under the optimal condition at 10% concentration for Microbiocide and Antiscalant with the 2 results of standard concentration of 20% Microbiocide and Antiscalant at pH 6.15-8.56, conductivity at 216-369 µS/cm of feed water and controlled driving pressure at 4 Bar by RO Unit controller. Conductivity and % Salt rejection of RO water are the key performance indicators, the results are shown in Fig.11 and Fig.12.

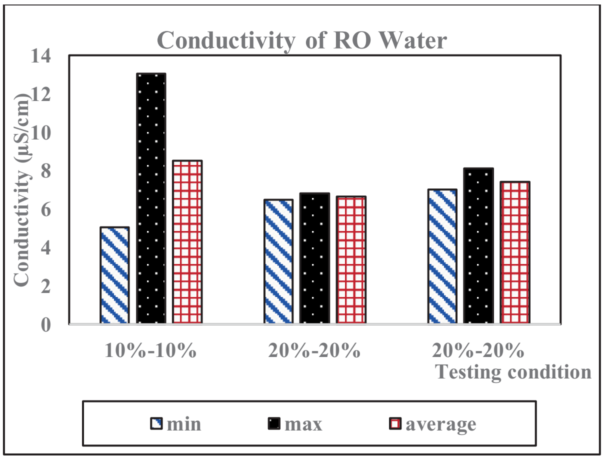

Fig. 11 The comparative Result of RO Water Conductivity between optimal condition and standard condition

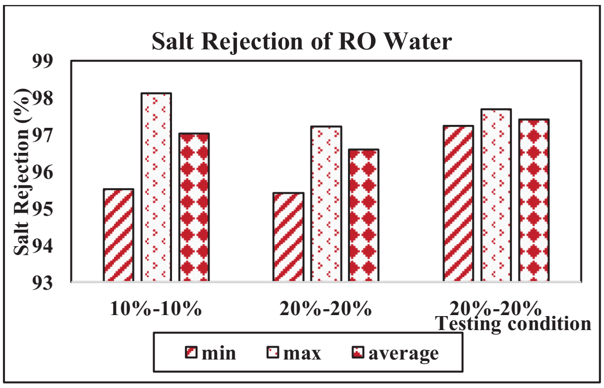

Fig. 12 The comparative Result of Salt Rejection of RO Water between optimal condition and standard condition

According to the result in Fig.11 and Fig.12, conductivity of RO water in optimal dosage concentration in the range 5.05-8.51 μS/cm with % Salt rejection in the range 95.12-98.52, whereas the standard condition yield conductivity in the range of 6.49-6.80 μS/cm and 7.01-8.11 μS/cm with % Salt Rejection in the range of 95.42-97.22 and 97.23-97.68. The optimal condition could operate approximately to the calculated value from Excel Solver. This condition could reduce chemical consumption amount and help monitoring performance of RO unit.

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