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Wastewater treatment and recycle from a semiconductor industry: A demo-plant study

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Abstract

The objective of this study was to establish a demo-scale plant with 1,000 cubic metres per day (CMD) capacity to recycle industrial wastewater from a semiconductor industry. In this study, two wastewater streams from continuous electrodeionization (CEDI) and local scrubber (LS)/controlled decomposition and oxidation (CDO) with flow rate of 1,335 CMD and 1,012 CMD respectively were chosen to be recycled. For the CEDI reject reclaim system, boron selective resin (BSR) and activated carbon filter (ACF) were utilized to remove boron and total organic carbon (TOC) respectively. The water quality was good enough to be used as ultrapure water (UPW) supply. For the CDO reclaim system, the combination of ACF + ultrafiltration (UF) + reverse osmosis (RO) under high pH condition was implemented to recycle the local scrubber wastewater (LSW) for cooling tower top up. Product water from both treatment systems was able to meet the target water specifications. The average cost saving was S$0.91/m^3 of reclaimed water produced.

Key words: activated carbon filter, boron selective resin, reverse osmosis, ultrafiltration

INTRODUCTION

Semiconductor industries have become one of the most crucial contributors to Singapore's economy in recent years due to the increasing global demand for electronic products (Bang *et al.* 2016; Liang *et al.* 2016). For example, the semiconductor industry contributed US$63.4 billion in 2013 to Taiwan's economy and was predicted to reach US68.35 billion in 2015 as reported by the Taiwan Semiconductor Industry Association (Liang *et al.* 2016). Nevertheless, this rapid development of the semiconductor industry also prompts environmental threats such as generation of wastewater and high consumption of clean water (Huang *et al.* 2010). The wastewater produced often contains high levels of anions and organic pollutants like ammonia-nitrogen, phosphate and fluoride (Xiao *et al.* 2014). The phosphate and ammonia-nitrogen are major contributors to water eutrophication (Huang *et al.* 2017). In addition, skeletal fluorosis occurs if too much fluoride is consumed by the human body (Deng *et al.* 2016), and the wastewater contains heavy metal ions, acid and bases which are toxic and harmful to the environment (Ryu *et al.* 2008). Therefore, the wastewater from the semiconductor industry must be treated and disposed of appropriately. The semiconductor industry normally reuses its treated wastewater for cooling towers, scrubbers and other facilities (Currier *et al.* 2008). These applications can greatly reduce water consumption and costs (Lee *et al.* 2008).

In the semiconductor industry, there are many product processes. The wastewater rejected from reverse osmosis/continuous electrodeionization systems (RO/CEDI) is considered to be relatively

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clean and easy to treat and recycle. An RO/CEDI system is designed for producing ultra-pure water (UPW) from any feed water stream. Raw water should first go to the RO system to remove various types of large molecules and ions from feed water by applying pressure to the feed when it is on one side of a selective water permeable membrane. The recovery rate of RO can reach about 60–80%. CEDI is a continuous electrochemical process that is applied after RO. A CEDI system purifies the RO product water to a high quality (ultra) pure water. During this process most of the remaining ions are removed from the RO product water, including weakly ionized materials, like silicates and CO_2. The recovery rate of CEDI can be as high as 95%. Normally, the reject from CEDI could be recycled to the feed of the RO system because of low total dissolved solids (TDS) (not more than 25–30 ppm). However, it should be noted that the CEDI reject may contain a high concentration of ions, such as boron (B) that requires further treatment before recycling.

As the reject from the CEDI system contains a high ion concentration, an ion exchange system could be applied to produce high purity deionized water for reuse. Ion exchange is the reversible interchange of ions between a solid (ion exchange material) and liquid in which there is no permanent change in the structure of the solid (Alexandratos 2009). Furthermore, it is a proven cost-effective method of recycling selected wastewaters and metal from semiconductor processing activities. The ion exchange process involves resin beads, which are used to trap and exchange ions. There are two types of ion exchange resins: cation resins, which are used for the purpose of trapping positively charged ions; and anion resins, which exchange negative ions. Also, a mixed bed resins, or the combination of cation and anion, are used for the purpose of achieving purified water that is free of ions. This is an efficient ion exchange method for exchanging positive and negative ions simultaneously. By using selected resins, it is possible to recover expensive materials from waste and wastewater (e.g., recovery of heavy metals).

In the semiconductor manufacturing processes, both the chemical vapor deposition (CVD) and etch process use perfluorinated compounds (PFCs), which have a high global warming potential and must be removed from emissions into the atmosphere. The abatement of PFCs is carried out through use of a local scrubber (LS) and is applied at the point of use in the semiconductor manufacturing process. A large amount of wastewater is generated from the LS system due to the use of water to wash the byproduct gases from thermal or plasma treatment of PFCs. The total water usage of the LS process can reach 30% of whole plant water usage for a 300 mm wafer semiconductor manufacturing plant. Therefore, local scrubber wastewater (LSW) offers a strong opportunity for water reclamation. It is important to note that LSW may contain high concentrations of fluoride (F^-), chloride (Cl^-) and organic compounds. Thus, LSW has to be treated appropriately before recycling and reusing.

Currently, an advanced oxidation process (AOP) would be applied to remove the organic compounds from water, which is then followed by precipitation or ion exchange to remove other ions by the addition of new chemicals. This type of treatment process would introduce new pollutants and create new environmental issues. Therefore, an effective treatment process which employs activated carbon filter (ACF) and reverse osmosis (RO) had several advantages compared to other conventional methods (Huxstep & Sorg 1988; Kegel *et al.* 2010). ACF not only can remove total organic carbon (TOC) and suspended solids (SS) in the mixed wastewater, but also can act as an absorbent for adsorption of trace metals due to its large surface area, high porosity and low material cost, compared to strong-base resins. The RO could then remove ions and other impurities and further polish the effluent from ACF for subsequent reuse.

This study investigated the feasibility of reclaiming CEDI reject water and LSW for reuse as UPW and cooling tower top up, respectively. A demo-scale plant was designed and constructed to tackle these wastewater streams, aiming to save water input in the semiconductor company.

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METHODS

Flow characterization

The flow rate of water streams was measured using flow meters. All flow meters were checked to ensure proper calibration and maintenance were done. Conditions of all relevant pipes network were checked to ensure no leakage. Flow measurement was recorded every day and water consumption was monitored.

Water quality characterization

Qualitatively, there were 37 parameters to be monitored to evaluate the quality of the water streams. The main parameters to be monitored were the concentrations of the following: fluoride (F^-), phosphate (PO_4^{3-}), TOC, chemical oxygen demand (COD), biochemical oxygen demand (BOD_5), ammonia (NH_4^+), TDS, hydrogen peroxide (H_2O_2), boron (B), chloride (Cl^-), total and dissolved silica (SiO_2), pH and M-alkalinity. The data were then recorded and interpreted.

In this study, TDS was estimated from conductivity based on the equation $TDS = k^*Conductivity$. A typical value of 0.7 was selected as the k factor (Walton 1989).

RESULTS AND DISCUSSION

Selection of raw water stream

The flow rate and water quality of potential raw water streams were determined and shown in Table 1. The water quality specifications of potential reuse points are shown in Table 2.

Previously, CEDI reject was reused for cooling tower make up while LSW was disposed and discharged as wastewater in the company. However, as shown in Table 1, the quality of CEDI was relatively good and could be used for UPW uptake if treated appropriately. By comparing the water quality of CEDI reject and UPW uptake requirements, it was noted that all parameters of CEDI reject had met the requirements except TOC and boron. Hence, proper treatment process

Table 1 | Flow and quality of potential raw water streams

Main parameters	CEDI reject	LSW
Flow rate	1,335 CMD	1,012 CMD
F^-	ND*	3.84–120.41 mg/L
PO_4^{3-}	0–2.0 mg/L	0–100 mg/L
TOC	0–0.3 mg/L	0.76–24.5 mg/L
COD	0–13.0 mg/L	0–133.71 mg/L
NH_4^+	0–1 mg/L	0–91.90 mg/L
TDS	12.0–32.0 mg/L	50–353 mg/L
H_2O_2	ND*	0–162 mg/L
B	0.02–0.37 mg/L	0–1.6 mg/L
Cl^-	0–2.3 mg/L	0–92.6 mg/L
Total-SiO_2	0.02–0.41 mg/L	1.27–36.8 mg/L
Ca	ND*	0–0.27 mg/L
pH	8. 2–10.5	3.0–9.9

*ND indicated non-detectable.

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Table 2 | Target treated quality of potential reuse point

Main parameters	UPW uptake	Cooling tower make up
F-	<0.5 mg/L	–
TOC	<0.05 mg/L	<5 mg/L
NH_4^+	<1 mg/L	–
TDS	<150 mg/L	<500 mg/L
B	<0.05 mg/L	–
Cl^-	<20 mg/L	<250 mg/L
Total-SiO_2	<3 mg/L	<50 mg/L
pH	7.0–8.5	6.0–8.5

must be implemented to remove TOC and boron in CEDI reject before reusing. Additionally, in order to replace the loss of CEDI reject flow to cooling tower, LSW which had the similar flow rate showed potential for reuse in cooling tower. A series of treatment processes might be required for the LSW to meet the standards for cooling tower make up.

Process train design

Two systems were designed, namely a CEDI Reject Reclaim System and LS Reclaim System (also known as CDO Reclaim system) for the treatment of CEDI reject and LSW, respectively.

CEDI reject reclaim system

For the CEDI Reject Reclaim System, the major concerns were boron and TOC concentrations. Boron is a p-type impurity in silicon or germanium, and hence creates the positively charged holes, which are deficient in valence electrons, to the semiconductor's valence band. It will cause the inversion of n-type semiconductor, silicon, which can affect the concentration of charge carriers (Wen *et al.* 2005). Hence, the process water used in the production of submicron devices must not contain boron. In addition, for water used in wafer fabrications plants, TOC is to be of a concentration not more than 0.1 mg/L, with a requirement of 0.05 mg/L for recycle water to enter the UPW plant. In this case, ion exchange process and activated carbon could be implemented to remove boron and TOC respectively. In order to ensure effective boron removal, boron selective resin (BSR) from Mitsubishi Chemicals was selected.

Regeneration will be required when the resins are exhausted. Before regeneration, a backwash is conducted to remove any trapped particles and to remix the bed to remove any channels formed during service. The bed is fluidized during backwash, and the larger and denser particles will be reoriented to the bottom. The arrangement produces a good hydraulic flow pattern, and improves resistance to fouling via suspended solids. The resin bed is then treated with the regenerant solution. After regeneration, the bed is rinsed with sufficient water to remove excess regenerant. Regeneration depends on the nature of the liquid phase, and aims to extend the lifespan of the resins (Velizarov 2010). In this study, sodium hydroxide and sulfuric acid were used as the regenerants.

Activated carbon was applied in the CEDI Reject Reclaim System to remove unwanted TOC. It was reported that activated carbon has a large surface area (of approximately 500–1,500 m^2/g) for the adsorption of contaminants (Ibanez *et al.* 2007). The large surface area is required for a practical residence time (Kucera 2010) and thus able to adsorb TOC effectively. The process train of the CEDI Reject Reclaim System is shown in Figure 1.

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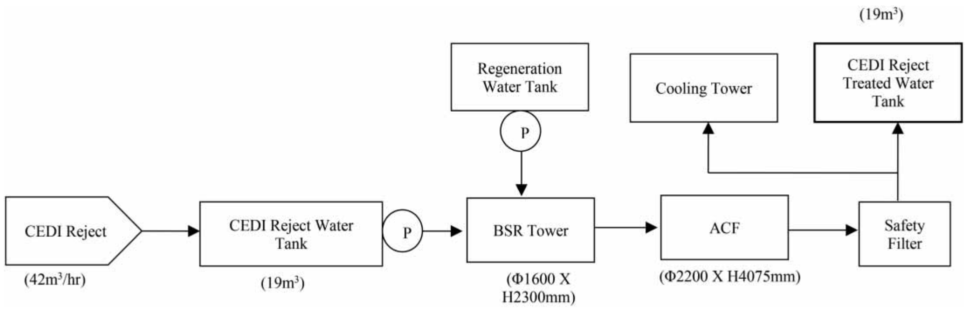

Figure 1 | Process train of CEDI Reject Reclaim System.

CDO Reclaim System

According to measurement results, major concerns for CDO reclamation are TDS, TOC, F^-, Cl^-, SiO_2 and H_2O_2. In this case, RO, which is able to remove constituents less than 0.001 microns and ionic constituents smaller than the pore size of the membrane, is suitable to treat the LSW and to achieve target water quality. RO processes typically work at a recovery rate of 75–85% (Bartels *et al.* 2010). In the treatment of semiconductor wastewater, it was reported that a RO system could remove TDS, which refers to any minerals, salts, metals, cations or anions dissolved in water, such as nitrates, nitrites, phosphates, silica, TOC, organic nitrogen (Bartels *et al.* 2010), and boron in the form of boric acid. RO can remove at least 95–99% of TDS (Ibanez *et al.* 2007). Among these contaminants, it is important to remove ionic material such as hardness, silica and phosphates due to the possibility of scaling. Therefore, anti-scalant must be dosed into the RO system and pretreatment of raw water must be implemented to minimize the scaling issue.

It was reported that Ca^{2+} could be precipitated as CaF_2, which might cause scaling of a RO membrane. Although Ca^{2+} concentration was low (<0.5 mg/L) in LSW, scaling still needed to be minimized by anti-scalant or pH adjustment as F^- is inactive in an alkaline environment (pH 10.5–11). Additionally, total-SiO_2 might cause RO membrane scaling. Low concentrations of SiO_2 could be removed by RO at high pH. Therefore, in order to minimize the fouling and scaling concerns of the RO membranes, a high pH of 10–11 was maintained in the process train. Furthermore, high pH of 10.5 is able to kill or inactivate the microorganisms and bacteria which have high potential to cause biofouling in the RO membrane. In this study, 50% NaOH was dosed into the raw water to increase the pH to 10.5–11.

Furthermore, hydrogen peroxide (H_2O_2), with concentration ranging in 0–162 mg/L in the LSW stream, could result in degradation of membrane media due to oxidation. H_2O_2 could be decomposed by activated carbon through the exchange of the hydrogen peroxide anion and a hydroxyl oxygen group on the solid surface. In addition, chlorine and TOC which might cause damage to the RO membrane could also be removed by activated carbon via redox reaction and adsorption, respectively. Therefore, ACF was incorporated into the CDO Reclaim System.

Before RO, ultrafiltration (UF) units are required to serve as pretreatment to achieve the desired purity required for RO influent. UF has been utilized for the removal of dissolved BOD, dissolved organics (Cartwright 1988) and particulates, such as colloids and silt, from the soluble constituents in a carrier solvent. Microorganisms such as bacteria and viruses might also be removed, but not ions and small molecules (Tong & Nwaoha 2012). Compared to conventional chemical and mechanical filtration processes, the UF process provides a more reliable treatment outcome. This is particularly useful for systems which receive feed water of variable quality, and of high organic levels (3M Company 2006). Hence, UF played an important role in treating the LSW with fluctuating quality.

Lastly, a neutralization process is necessary to reduce the pH of treated water to the target of 6.0–8.5 for cooling tower reuse purpose. Hence, 65% of sulfuric acid was used and dosed into the

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RO permeate before reusing in cooling tower. The process train of the CDO Reclaim System is shown in Figure 2.

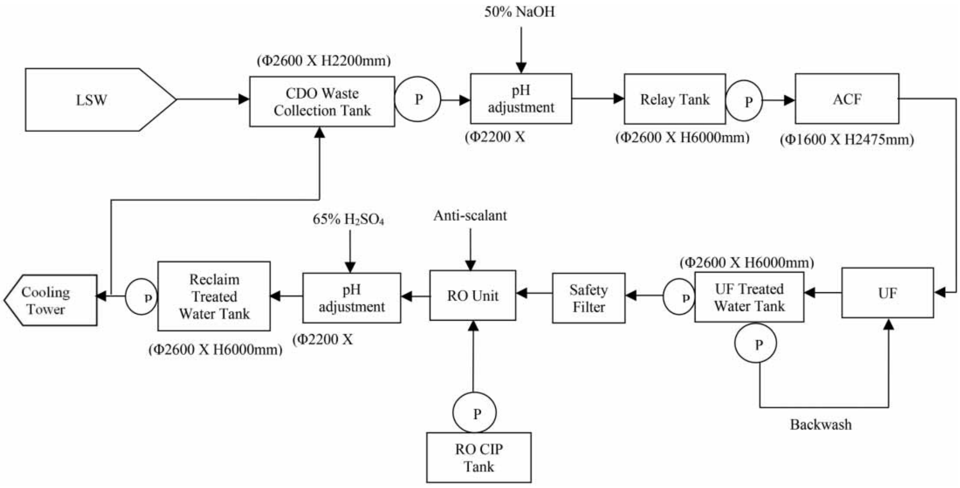

Figure 2 | Process train of CDO Reclaim System.

Performance evaluation of demonstration plant

A demonstration plant with two treatment systems (CEDI Reject Reclaim System and CDO Reclaim System) was constructed. The design capacity was 1,000 CMD for both treatment systems with 90% and 67% recovery for the CEDI Reject Reclaim System and CDO Reclaim System, respectively.

CEDI Reject Reclaim System

In order to evaluate the treatment efficiency of the CEDI Reject Reclaim System, water samples were collected three times per week at three sampling points. Briefly, the first sample was taken before the boron removal step and marked as IE-1, the second was collected after boron removal and named IE-2 while the last sample, IE-3, was collected after ACF (see Figure S1).

As determined by water characterization, the major concerns of the CEDI Reject Reclaim System were boron and TOC. Thus, the efficiency of the CEDI Reject Reclaim System in removing both boron and TOC was investigated and closely monitored. The average concentrations of boron over five months of operation were 0.35, 0.04 and 0.02 for IE-1, IE-2 and IE-3, respectively.

This indicated that the BSR was effective in removing boron from the feed stream. It was also noted that the BSR was able to remove boron to less than the maximum allowable limit of 0.05 mg/L, suggesting that BSR was suitable for boron treatment in this study. Other than boron, the concentration of TOC was also investigated. As the TOC of product water was solely concerned, only the TOC of IE-3 was examined. The average outlet TOC of the CEDI Reject Reclaim System was 0.027 mg/L, which was lower than the target of 0.05 mg/L. Hence, it was concluded that the CEDI Reject Reclaim System was able to produce treated water that met target specifications.

CDO Reclaim System

For the CDO Reclaim System, water samples were collected from five different sampling points, namely LS-1, LS-2, LS-3, LS-4 and LS-5 (see Figure S2). LS-1 refers to LSW raw water, LS-2 was collected after ACF, LS-3 was collected after UF while LS-4 and LS-5 refers to RO permeate and RO reject, respectively. Water samples were collected three times per week to monitor the performance of each treatment process.

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The results of main parameters of each sampling point are summarized in Table 3. The results reported were averaged over five months of data. It is clear that ACF played an important role in removing H_2O_2 and subsequently reduced damage to the RO membrane. The concentration of H_2O_2 after ACF (LS-2) was less than 0.5 mg/L. The plant was also effective in reducing the concentration of TOC to below 3 mg/L, which is lower than the maximum 5 mg/L for use as cooling tower make-up water.

Table 3 | Water sample analysis of CDO Reclaim System (averaged over five months of data)

	LS-1	LS-2	LS-3	LS-4	LS-5
pH	3.24	9.83	9.76	10.33	9.02
TDS (mg/L)	461.60	521.08	498.77	156.43	1,904.95
M-alkalinity (mg/L)	0	117.65	118.55	83.45	304.33
H_2O_2 (mg/L)	5.29	0.16	0.09	–	–
TOC (mg/L)	5.47	3.56	3.48	2.31	16.96
COD (mg/L)	11.30	5.99	4.93	2.04	27.68
NH_4^+ (mg/L)	15.87	13.07	13.07	9.22	27.74
F^- (mg/L)	60.66	59.33	58.14	0.47	109.26
Cl^- (mg/L)	8.12	7.97	7.84	0.19	35.04
Ionic-SiO_2 (mg/L)	22.36	–	–	0.89	97.79
Total-SiO_2 (mg/L)	22.88	–	–	0.95	104.88

RO membrane units were critical in removing both Cl^- and F^- ions. It is shown in Table 3 that the concentration of chloride in RO permeate was less than 1 mg/L which indicated that most of the chloride had been removed by the RO process. In this project, even though there is no requirement for fluoride concentration for cooling tower make-up water, the concentration of F^- was still closely monitored as it might corrode metals and piping of the cooling tower. As shown in Table 3, the RO system was able to remove F^- ions to less than 1 mg/L, which would minimize corrosion of metal and piping in the cooling tower system.

In addition, the concentration of SiO_2 in RO permeate was lower than the limit of 50 mg/L. The concentration of dissolved silica was measured, and it was noted that most of the total silica detected in the water samples was in the form of dissolved silica. In fact, both total and dissolved silica were less than 1 mg/L in RO permeate, which reflected high silica removal when RO was operating at high pH. According to Table 2, the maximum TDS allowed for the cooling tower make-up water is 500 mg/L. The results show that the conductivity was less than 200 mg/L in RO permeate, which fell within the targeted range and was suitable for reuse as cooling tower.

Before reusing treated water as cooling tower make-up water, a final pH adjustment was carried out to minimize cooling tower corrosion. Sulfuric acid was dosed into the RO permeate to reduce the pH to the target of 6.5–8.0 (results are not shown). All parameters had achieved the targeted standards. This indicated that the product water was suitable for reuse as cooling tower make-up water.

Water and cost saving

The amount of water saved after implementation of both the CEDI Reject Reclaim System and CDO Reclaim System was determined and calculated. The amount of saved water can be determined by calculating the amount of product water produced and reused. The average monthly water saving index for the four-month operation of the demo plant was determined to be 7.6%.

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Cost saving was determined by deducting the operation cost from the cost of saved input water. The average operating cost after implementation of the demo plant was S$1.28/$m^3$, while the cost of input water was S$2.19/$m^3$. Hence, it was calculated that the average cost saving achieved was S$0.91/$m^3$.

CONCLUSIONS

For the CEDI Reject Reclaim System, BSR and ACF were implemented to remove boron and TOC respectively. It was noted that BSR had high efficiency of boron removal and ACF was able to remove the TOC to less than 0.05 mg/L. Therefore, it was concluded that the treated water from the CEDI Reject Reclaim System was suitable for UPW intake.

For the CDO Reclaim System, treated water from the combination of ACF + UF + RO under high alkaline condition was able to achieve the targeted water quality. The product water was suitable to be reused in cooling tower.

After implementation of the demo plant, an average monthly water saving of 7.6% and average cost saving of S$0.91/$m^3$ were achieved.

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