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Communication

Pilot-Scale Experiences with Aerobic Treatment and Chemical Processes of Industrial Wastewaters from Electronics and Semiconductor Industry

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Abstract: TMAH is quaternary ammonium salt, consists of a methylated nitrogen molecule, and is widely used in the electronics industry as a developer and silicon etching agent. This substance is toxic and fatal if ingested. It can also cause skin burns, eye damage, and organ damage. Moreover, TMAH exhibits long-lasting toxicity to aquatic systems. Despite this known toxicity, the authorities currently do not provide emission limits (i.e., discharge concentrations) for wastewater by EU regulation. The current scenario necessitates the study of the processes for industrial wastewater containing TMAH. This work aims to present a successful example of the treatment process for the degradation of TMAH waste solutions of the E&S industry. Research was conducted at the pilot scale, and the process feasibility (both technical and economic) and its environmental sustainability are demonstrated. This process, which treats three exhausted solutions with a high concentration of toxic substances, is considered to be innovative.

Keywords: tetramethylammonium hydroxide; industrial and organic wastewater; E&S industry; pilot scale activity; aerobic treatment; chemical precipitation; safeguard and groundwater quality

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1. Introduction

The microelectronic industry produces a series of devices as memory and high-performance chips. These devices are produced on silicon wafers, which are integrated circuits for special applications, such as LEDs/OLEDs [1–4].

Different production processes involve the consumption of several chemicals, some of which are toxic and/or highly flammable. These substances should not be released into the environment without a specific treatment able to reduce the toxicity of wastewater.

The main substances contained in the residual effluents are organic ones, such as acetic acid, tetramethylammonium hydroxide (TMAH), and mineral acids, including nitric and hydrofluoric acid. All these compounds are hazardous materials and, with the exception of TMAH, can be removed by traditional processes [5]. TMAH is used as high-alkaline matter for the microelectronic devices. TMAH has toxic properties and can cause poisoning and, in some cases, death, even in diluted solutions. Moreover, TMAH is classified as ecotoxic, according to the ECHA [6]: "Hazardous to the aquatic life with ling lasting effects (H411). No observed Effect Concentration (NOEC) is 0.02 mg/L, max value to prevent chronic toxic effects on invertebrate organisms."

At the moment, EU regulation does not provide discharge limit values for TMAH. Despite this, TMAH is classified in the "Substances which have an unfavorable influence on the oxygen balance" and "Substances which contribute to eutrophication (in particular, nitrates and phosphates" in accordance with the Directive 2010/75/EU and EU Water Framework Directive 2000/60/EC (Annex VIII).

Regarding these aspects and NOEC values, the Italian National Institute of Health recommends 0.4 mg/L and 0.2 mg/L for TMAH concentrations discharged into sewage and water bodies, respectively [7].

The problem of TMAH wastewater treatment is of European importance, considering that several industries produce and use TMAH for different industrial applications.

Unlike other pollutants, the removal of TMAH remains an ongoing challenge, and researchers have only recently began to find a sustainable method to treat effluents containing TMAH. Both biological and chemical treatments can be adopted. Regarding biological treatments, anaerobic treatment has been further analyzed [8–10], achieving 95% of TMAH degradation and producing methane and carbon dioxide.

Alternately, chemical oxidation can be applied [11], which releases a poisonous gas containing NOx. Thus, it is necessary to insert a further operation to dissociate the NOx into nitrogen, carbon dioxide, and water [12]. Chemical oxidation can be combined with biological processes to improve the removal efficiency [13]. Other techniques have been studied, such as adsorption with activated carbon [14], zeolites [15], and ultrafiltration [16].

Considering the current scenario of TMAH classification and treatment, the Life Bitmaps project was proposed. The project studies a technical and economical approach to treat TMAH solutions from a microelectronic manufacturing plant, LFoundry Srl (Avezzano, Italy), to meet the Italian recommendation. The Life Bitmaps project addresses the current problem of TMAH treatment, and in this paper, the main results are described. After a laboratory-scale experiment [16–20], a pilot plant was designed and realized to validate the results obtained on a smaller scale. Finally, the pilot data were used to design an industrial plant for the treatment of TMAH effluent, and a study on the sustainability of the processes from an environmental and economic point of view was discussed. The main goal of the project was to test the performance and prove the feasibility of the E&S industrial wastewater from an integrated point of view (technical, economical, environment, social) while also considering the risk analysis of the processes and plants [21].

2. Materials and Methods

TMAH wastewater treatment was performed in the transportable mobile plant built in the ambit of the Life Bitmaps research project. Besides the TMAH degradation with aerobic bioreactions, the plant can also process chemical precipitation, as well as the other two selected solutions from microelectronic production, BOE and SEZ, which contain mainly fluorides and inorganic and organic acids at high concentrations.

After a laboratory-scale optimization, as reported in our recent paper [19], technical solutions were outlined, carrying out a mass and energy balance for the treatment of the three residual wastes.

The plant was realized in two containers located at the Lfoundry site, the industrial partner of the project (Figure 1).

Figure 1. Mobile pilot plant for the wastewater treatment.

The treatment of TMAH included a neutralization with sulfuric acid to achieve a neutral pH and three bioreactors in series (R101, R102, and R103) with a capacity of 1.1 m^3, which were fed by the activated sludge of the plant located on the industrial site (WWT). The equipment used for this treatment was located in the first container. The biological process operated in continuous (maximum flowrate: 25 L/h). The processes for BOE and SEZ included chemical precipitation by adding calcium hydroxide, a coagulant (aluminum sulfate), and a filtration. The second container contained the equipment for the process of BOE or SEZ (maximum treatable volume per batch: 180 L). This line worked in batch mode. At the end of the processes, all treated wastes were safety sent to industrial plant for the treatment of black waters. The block diagrams of the processes carried out in the Life Bitmaps portable plant are reported in Figure 2.

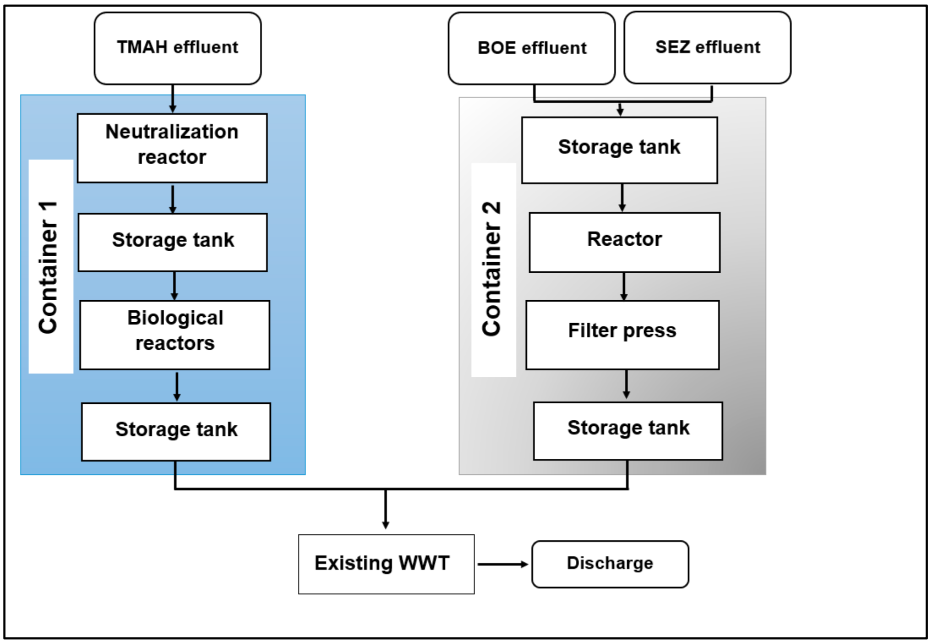

Figure 2. Block scheme of the processes performed in the Life Bitmaps portable plant (WWT = wastewater treatment).

2.1. *Biological Processes for TMAH Degradation at the Pilot Scale*

The experimental test for TMAH degradation began with the inoculation of the biological reactors by activated sludge coming from WWT. TMAH effluent was fed, changing the flowrate in a range equal to 5–20 L/h. The plant worked both in continuous mode and batch mode to stress and study the response of the microorganism and the efficiency of the degradation. The pilot research activity was carried out from June 2018 to December 2018. Over 50 liquid samples were collected, and around 800 chemical analyses were performed to monitor the Chemical Oxygen Demand (COD), pH, Total Suspended Solid (TSS), TMAH and its intermediates concentrations, dimethylamine (DMA), ammonium ions, and nitrates. Chemical oxygen demand (COD), N-NH_4, N-NO_3, TMAH, and dimethylamine (DMA) concentrations were regularly determined according to the APHA Standard Methods. Each sample was centrifuged at 4000 rpm in a 50 mL Falcon tube centrifuge (Rotofix 32A, Hettich, Westphalia, Germany). The supernatant was analyzed to determine the parameters indicated above, whereas the dried settled material (105 °C for 24) provided the TSS, the value proportional to the microbial mass.

TMAH and DMA were measured using the GC-MS technique (DX5000, Dionex, Sunnyvale, CA, USA).

COD, N-NH_4, and N-NO_3 were measured using the UV-Vis method with the spectrophotometer CADAS 200 (Hach Lange, Loveland, CO, USA) and Dr. Lange cuvette kits (LCI 400 for COD, LCK 303 for ammonium ion, and LCK 339/340 for nitrate ion) [22].

2.2. Chemical Processes for BOE and SEZ Effluent Treatment at the Pilot Scale

Chemical treatment for the BOE and SEZ effluents included lime precipitation in the presence of aluminum sulfate, as reported by the authors of [19]. $Ca(OH)_2$ was added at 16% concentrated solutions. Several experiments were performed to determine the optimal dosage of the reagents to remove the pollutants. At an optimal pH value, the system worked under constant stirring. After 2 h (reaction time), the suspensions were filtered. Samples of reactor suspension and filtrate were collected at the end of each test. Solid cake was dried at 105 °C. The liquors were analyzed to measure the fluorides, nitrates, and COD using Dr. Lange's kit, cuvette-test LCK 153 and LCK 114A. Other chemical analyses on the solutions were performed by atomic spectroscopy Agilent Synchronous Vertical Dual View (5100 ICP-OES). The recovered solids were analyzed by XRF spectrophotometer (Spectro XEPOS 2000) and infrared spectroscopy (FTIR, Impact 410 Nicolet spectrophotometer).

3. Results

The processes addressed to treat TMAH, BOE and SEZ waste were performed in the transportable pilot plant from the Life Bitmaps research project. The main results for the pilot scale experience are reported in the following subsections.

3.1. Pilot Scale Process for the Treatment of TMAH Effluent by Biological Degradation

TMAH effluent from the LFoundry industrial plant was collected into reactor N101. Then, sulfuric acid was added to modify the pH level from 12 to 7. Neutralized solution fed the reactor R101, which fed reactor R102, which fed reactor R103. A storage tank collected the solution coming from R103. Then, this suspension was fed WWT. The biological reactions transformed ammonium ions into nitrates, followed by denitrification reactions that produced nitrogen by denitrification [19]. Two series of tests were carried out: (1) the start-up of the plant with several interruptions due to technical problems, and (2) a series of experiments without technical interruptions. The results of the first series showed an abatement of TMAH over 90%. More interesting data were obtained in the second series, as described below. For the first period (until day 60), the flowrate of the TMAH effluent was 5 L/h. After that, there was a period of batch for 2 weeks. Then, the plant worked in a continuous mode, feeding 10 L/h. In the last days (from day 47), the plant was fed 20 L/h. Table 1 reports the TMAH effluent composition (before the treatment).

Table 1. Average composition of TMAH effluent.

Parameter	Value
pH	12
Tetramethylammonium hydroxide	2161–3200 mg/L
Ammonia nitrogen	6.05–8.6 mg/L
Nitrate nitrogen	<0.15 mg/L
Dimethylamine	<0.05 mg/L

The results of the second series are shown in Figures 3 and 4. The parameters TMAH, DMA, ammonium, and nitrates were analyzed because they are important to understand the functionality of the biological system. In fact, N-NH4 concentration is an index that degrades substances containing ammonia (in this case, TMAH).

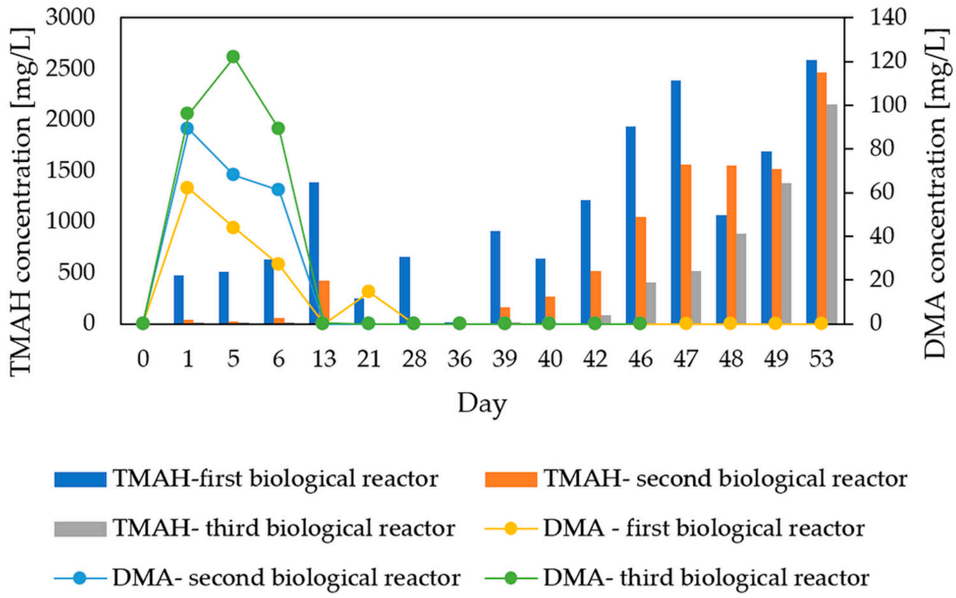

Figure 3. TMAH and DMA concentration (mg/L) trends as a function of the research pilot time.

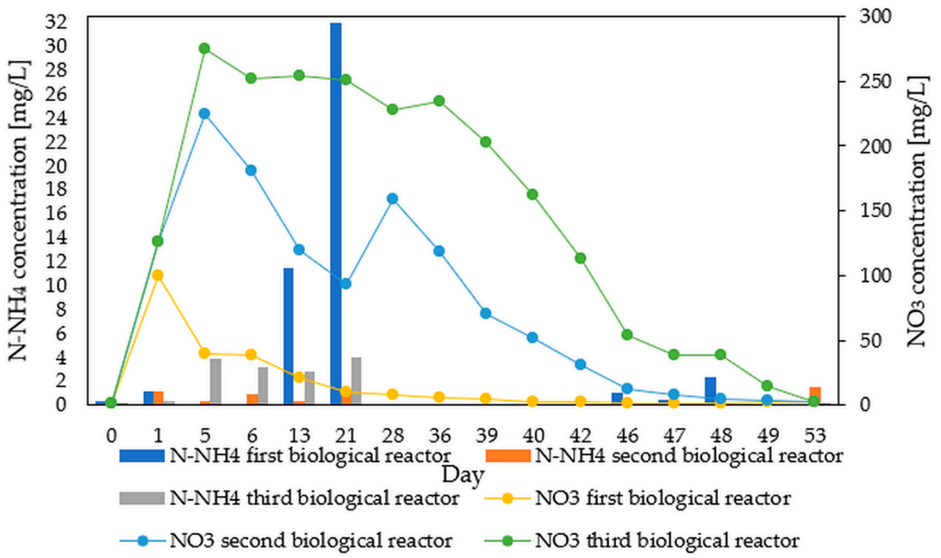

Figure 4. $N\text{-}NH_4$ and NO_3 concentration (mg/L) trends as a function of the research pilot time.

The TMAH concentration in the first biological reactor was variable, reaching close to 0 mg/L at the end of the batch period. When the 10 L/h flowrate was selected, the TMAH concentration started to increase, and this increment was also evident at the flowrate of 20 L/h. After 53 days of processing, the outlet TMAH concentration was close to 2.5 g/L (close to the inlet concentration). This result highlights the low biological degradation of TMAH that occurred when the flowrate was increased. As a direct consequence, the concentrations of ammonium nitrogen and nitrates decreased. The COD values were almost close to 135 mg/L, with a peak near 200 mg/L in the last period when the flowrate reached its maximum. The same situation was also recorded for the second and third biological reactors.

Obviously, the TMAH concentration was close to 0 mg/L in R102 and R103 until the end of batch operation. The $N\text{-}NH_4^+$ concentration was around 1 mg/L, and the NO_3^- concentration was higher with respect to the values in R101. These data indicate that the nitrification process of $N\text{-}NH_4^+$ happens in the last reactor. At the end of the test, the nitrates concentrations decreased, probably due to the changing conditions inside the reactors.

The system performance collapse was likely related to the phenomenon of washout. This situation was also confirmed by the trend of TSS. In fact, the TSS value decreased in the initial phase of adaptation (about 1600 mg/L). The TSS value remained constant until the end of the batch procedure, and then decreased during the subsequent continuous reactors feeding. When the process was stopped, the TSS value was close to 0.2 mg/L.

The experimental data were analyzed to define the TMAH degradation, which was 99%, in optimal conditions (5 L/h) with a process time of 192 h. We also performed an analysis to develop a potential full-scale plant schema.

3.2. Chemical Processes for BOE and SEZ Effluent Treatment at the Pilot Scale

SEZ and BOE come from the washing processes of wafers (thin slices of semiconductor) and are highly polluting effluents. SEZ contains fluorides (18,200 mg/L $\pm$ 1200), nitric acid (145 $\pm$ 15 g/L), and acetic acid (62 $\pm$ 30 g/L). Three experiments were replicated, which involved adding lime to increase the pH from 1.5 to 5. In all tests, a fluoride removal yield greater than 99% was obtained. After treatment, its concentration was about 7.5 mg/L $\pm$ 2.5. The removal yield for other acids was negligible, as these substances are nutrients for bacteria during subsequent biological treatment. The residue solid recovered after the filtration had around 60% humidity and mainly constituted calcium (59.2%), aluminum (3.4%), sodium (2.36%), phosphate (20.66%), and sulfates (7.2%).

The other effluent, BOE, contains fluorides (22.3 $\pm$ 2.3 g/L), phosphates (1.5 $\pm$ 0.6 g/L) ammonium (22.6 $\pm$ 1.5 g/L), and nitrates (12.4 $\pm$ 0.4 g/L). Three pilot experiments were performed for the BOE treatment, which involved adding lime to remove the fluorides and phosphates. The removal results were higher than 99% for both fluorides and phosphates. The final concentration of fluorides was near 9 mg/L, while the final concentration of phosphates was 0 mg/L. The residue solid recovered after filtration had around 50% humidity and mainly constituted calcium (91%), aluminum (2.2%), sodium (1.23%), and phosphate (5%).

Process analyses were carried out using the experimental data with the aim of proposing two process schemas.

Solid wastes characterization displayed that these residues are not dangerous for landfill disposal.

4. Discussion

The process analysis was useful to define the mass and energy balances able to treat the TMAH, BOE, and SEZ effluents for the full-scale plant. The details of the process analysis are not reported in this manuscript. Several process schemes were proposed and tested, fixing a continuous flowrate of 800 L/h for TMAH and assuming a batch mode for BOE and SEZ with a capacity of 435 t/y and 145 t/y, respectively. The mass balances, based on 100 kg of BOE and SEZ, showed that 3 kg of aluminum sulfates was necessary for both wastewater treatments. Lime consumption was 90 kg for BOE and 145 kg for SEZ. The residual solids were 17 kg and 22 kg for BOE and SEZ, respectively. Regarding the economic aspects, the total cost for the TMAH, BOE, and SEZ treatment was EUR 15/m^3, EUR 110/m^3, and EUR 207m^3. The main cost for the TMAH treatment was the energy used for the aeration of the biological reactors. The main cost for the BOE treatment, which was even greater for the SEZ treatment, was the disposal cost.

The environmental study performed by Gabi software (not reported here) showed that electricity utilization represented the principal contribution (larger than 90%) on the whole environmental load (TMAH treatment). In addition, lime consumption for precipitation caused about 70% of the impact, which was correlated to the BOE and SEZ treatment. The highest impact classes were climate change, ionizing radiation, and reserve use.

The process analysis showed that the proposed technologies were more sustainable, both economically and environmentally, when compared with the current disposal processes adopted by Lfoundry.

5. Conclusions

The E&S industry produces a large amount of toxic wastewater, and TMAH is among the most dangerous pollutants. At the moment, discharge limit values are not provided by the EU regulation (i.e., concentrations at discharge). However, the Directive 2010/75/EU and the EU Water Framework Directive 2000/60/ EC (Annex VIII) provide a list of contaminants that affect the water quality as "Substances which have an unfavorable influence on the oxygen balance (and can be measured using parameters such as BOD, COD, etc.)" and "Substances which contribute to eutrophication (in particular, nitrates and phosphates)", with TMAH belonging to both categories. Due the large use of TMAH in many industries in Europe, the problem of TMAH wastewater treatment assumes increasing importance. In fact, many industries synthetize and use TMAH for several industrial applications. The development of advanced treatment of this kind of industrial waste represents a crucial challenge for the European E&S industry that must be improved for the protection of surface and groundwater. Hence, in this study, an innovative wastewater treatment process was proposed and tested at the pilot scale within the Life Bitmaps research project. The pilot plant was able to treat the three effluents produced by microelectronic manufacturing, called TMAH, BOE, and SEZ, in an integrated way. For the first time, biological degradation with three biological reactors has been proposed, and BOE and SEZ, a chemical treatment has been tested. The results show that TMAH can be degraded with a yield of 99%. The same efficiency was also obtained for the removal of pollutants from the BOE and SEZ effluents.

Our economical and sustainability studies demonstrate that the proposed technologies are more sustainable, both economically and environmentally, when compared with the currently adopted disposal processes. The processes addressed in this study are under improvement. Our future work will address an optimization for the full-scale plant for TMAH and for the valorization of the solid residues from BOE and SEZ treatment.

6. Patents

Vegliò, F., Prisciandaro, M., Ferella, F., Innocenzi, V., Di Renzo, A., Saraullo, M., Zueva, S., De Michelis, I., Tortora, F., 2018. Process and plant for the treatment of a wastewater containing tmah, Patent number WO/2020/012240A1.

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Conflicts of Interest: The authors declare no conflict of interest.

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