

# Studies on electrochemical treatment of wastewater contaminated with organotin compounds

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## Abstract

Different anode materials were tested to evaluate their suitability to eliminate organotin compounds electrochemically from shipyard process waters. The capacity of two types of anode materials was investigated: niobium coated with boron-doped diamond (BDD) and titanium coated with iridium dioxide, (Ti/IrO<sub>2</sub>). The aim of this work was to characterize the performance of the process using both anode materials at different current densities, and also to evaluate the generation of by-products. A further objective of this work was to evaluate if operating at low potentials with BDD anodes (to avoid the generation of elemental oxygen) the consumption of energy for degradation of pollutants could be minimized. The processes were tested on synthetic and real shipyard water containing approximately 25,000 ng L<sup>-1</sup> of tributyltin (TBT) (as Sn) and 5000 ng L<sup>-1</sup> dibutyltin. The range of current densities was between 6 and 70 mA cm<sup>-2</sup>. The results showed that electrochemical treatment is suitable to eliminate organotins down to very low concentrations following a stepwise debutylation mechanism. Both anode materials exhibited a similar performance with energy consumption in the range of 7–10 kWh m<sup>-3</sup> in order to decrease organotins down to 100 ng L<sup>-1</sup> (as Sn). For the water composition tested, BDD did not outperform Ti/IrO<sub>2</sub> as initially expected, most probably because organotins were not only oxidized by •OH, but also by active chlorine compounds generated by the oxidation of chloride present in the wastewater (1.6 g L<sup>-1</sup>, Cl<sup>-</sup>) with both anode materials. It was also found that the residual oxidants remaining in the treated effluent had to be eliminated if the water is to be discharged safely in the aquatic environment. © 2007 Elsevier B.V. All rights reserved.

**Keywords:** Boron-doped diamond; Shipyard wastewater; Electrochemical treatment; Tributyltin

## 1. Introduction

### 1.1. Tributyltin in the aquatic environment

Tributyltin (TBT) is a very toxic compound, which shows endocrine disrupting properties [1]. The main pathways of biocides into the aquatic environment are associated with navigation related activities. Biocides are the main components of antifouling paint formulations to prevent the growth of shells, weed, and algae on submersed surfaces in order to avoid increased friction between water and the ship's hull, and the additional weight of the fouling. Both tend to reduce the manoeuvrability, and increase fuel consumption of ships. Diffuse sources of biocide pollution in water ways is their constant leaching from the ship hull's paint to the water column. The main point source is related to shipyard activities, for example, ship maintenance, coating of hulls, etc. [2].

Shipyards generate biocides containing water primarily from two activities [3]:

- Wash down operations using freshwater to remove salt and slime from the hull. This operation is performed on all dry-docked ships so that hull coatings can be inspected.
- The second source is hydroblasting, which involves the use of high-pressure freshwater (~2000 bar or higher) applied to a ship's hull to remove the paint. The operation may cause the removal of just the surface coating up to removal of all paint coatings (i.e. layers) down to bare metal.

Primer coats applied to bare metal (steel) typically contain high concentrations of zinc due to the improved cutting and welding of high-zinc primer coatings. Copper is also found as a significant component of marine paints due to its biocide properties that inhibit biological fouling on ship hulls [4]. Therefore, paints that contain TBT and may be subject to a specific TBT discharge limit are also likely to need to meet a copper and zinc discharge limit. Consequently, there will be multi-

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ple treatment objectives and removal goals for shipyard wastewater.

Organotin based-paints had been used in all kinds of vessels, from small yachts to large tankers, thus ensuring the global dispersion of TBT throughout the marine environment, in coastal and open sea regions [5]. Already in the early 1960's organotin compounds were tested as efficient biocides in antifouling paint formulations but it was until the mid 1970's that with the development and introduction of tri-*n*-butyltin (TBT) copolymer antifouling paints the use of such products became widespread worldwide [1]. In the mid 1990's organotin compounds were the most toxic substances produced in a large scale with annual productions of 50,000 ton per year. The most customary representatives of the TBT group are tributyltin oxide (TBTO), tributyltin benzoate (TBTB), tributyltin naphthanate (TBTN), and tributyltin acetate (TBTA) [5].

The use of TBT in coating formulations has resulted in economic benefits for the shipping industry because it extends docking intervals to up to five years, reduces the fuel consumption and maintenance need of ships. The constant release of TBT into the environment by ships and at shipyard locations in combination with its toxicity to nontarget organisms, accumulation, and persistence in sediments has resulted in detrimental effects for marine ecosystems at many locations around the world, such as deformities and calcification in oyster species, imposex (masculinisation of females) in marine snails and whelks. After its release into the environment TBT is removed relatively quickly from the water column because of fast adsorption kinetics of TBT on suspended particles and sediments. This results in the accumulation of TBT on sediments because of its much slower degradation rates than in the water column. TBT half-life has been reported by many researchers to be of years [1,6]. On the other hand, the adsorption process has been proven to be reversible, so sediments can act as a TBT source when dredged or when conditions change.

In seawater and under normal conditions, TBT exists as three species (hydroxide, chloride, and carbonate), which remain in equilibrium. At pH values less than 7.0, the predominate forms are  $\text{Bu}_3\text{SnOH}_2^+$  and  $\text{Bu}_3\text{SnCl}$ , at pH 8 they are  $\text{Bu}_3\text{SnCl}$ ,  $\text{Bu}_3\text{SnOH}$ , and  $\text{Bu}_3\text{SnCO}_3^-$ , and at pH values >10  $\text{Bu}_3\text{SnOH}$  and  $\text{Bu}_3\text{SnCO}_3^-$  predominate.

Biological degradation in water and sediment appears to be the most important factor limiting the persistence of TBT in aquatic environment. Dowson et al. [6] found that in aerobic conditions TBT half-life ranges from 360 to 775 days. The experimental results showed little difference between degradation rates in freshwater and estuarine sediments, but increase of half-life occurred in spiked sediments. Under anaerobic conditions, the half-life of TBT could not be determined by the authors, and appeared to be in the order of ten years.

### 1.2. Electrochemical wastewater treatment

Electrochemical treatment, as one of the advanced oxidation processes (AOP), is able to convert organic materials into simple, relatively harmless, and inorganic molecules. In recent years, the electrochemical oxidation process has been shown to

be promising for wastewater treatment, mainly due to its effectiveness and ease in operation [7], and for other applications like remediation of TBT contaminated sediments [8–12]. The electrochemical methods find several applications, such as metal ion removal and recovery, electrodialysis, electrodeionisation, and especially, destruction of toxic and nonbiodegradable organics such as cyanide and EDTA. The main problem associated with such electrochemical treatment is its high cost [13]. The use of new anodic materials like boron-doped diamond (BDD) has allowed achieving high efficiencies in the use of electric energy, and as consequence, to strongly decrease the operating cost of this technology [14]. BDD anode has a high yield of hydroxyl radicals, which can facilitate degradation of organic compounds. It also has high chemical and mechanical stability. The overpotential for water electrolysis of BDD is high so that the side reaction of oxygen evolution can be minimized, thus preventing loss of current efficiency. Nevertheless, it was also found that the side reaction of AOX formation (adsorbable halogenated organics) also occurs in the electrochemical treatment using BDD [12].

In this work, different anode materials are studied to evaluate their suitability to eliminate organotins from shipyard process waters. The aim of this work is to characterize the performance of the process using both anode materials at different current densities, and also to evaluate the generation of by-products. Concerning the environmental effect of AOX, the mechanism of AOX formation, and the possible mechanism of elimination during the electrochemical treatment should be studied. An additional objective of this work was to evaluate if operating at low potentials with BDD anodes leads to minimization of energy demand for the degradation of pollutants.

## 2. Methods and materials

### 2.1. Experimental setup

The performance of two anode materials were studied, and compared with respect to their capacity to destroy organotin compounds in an undivided electrolysis cell in laboratory scale as shown in Fig. 1. The materials tested were:

- Titanium coated with iridium dioxide ( $\text{Ti}/\text{IrO}_2$ ).
- Thin film BDD. This material is known to exhibit the highest over potential for  $\text{O}_2$  evolution (2.8 V versus SHE). This results in a high yield of  $\bullet\text{OH}$  radicals, which in turn make BDD an outstanding material for the degradation of organic compounds.

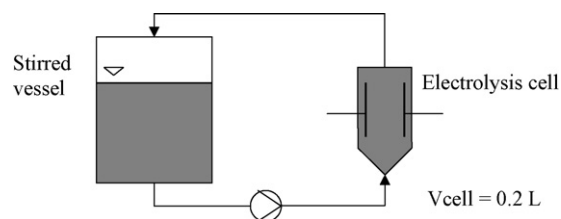


Fig. 1. Experimental setup of the electrochemical treatment.

Table 1  
Composition of artificial wastewater

Inorganic salts	Concentration, g L <sup>-1</sup>	Organic compounds	Concentration
NaCl	0.882	(NH <sub>4</sub> ) <sub>2</sub> hydrogencitrate	0.005 g L <sup>-1</sup>
CaCl <sub>2</sub> ·2H <sub>2</sub> O	0.650	Zn-acetate·2H <sub>2</sub> O	0.0556 g L <sup>-1</sup>
MgCl <sub>2</sub> ·6H <sub>2</sub> O	16.00	Organotins	Concentration, ng L <sup>-1</sup>
MgSO <sub>4</sub> ·7H <sub>2</sub> O	0.228	TBT	50,000
NaHCO <sub>3</sub>	0.440	Dibutyltin	10,000
Fe <sub>2</sub> O <sub>3</sub>	0.018	Monobutyltin	5000
Fe(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O	0.008	Properties	
KCl	0.052	pH	7.9
CuSO <sub>4</sub>	0.015	Redox potential, mV (Ref. SHE)	340
		Conductivity, mS cm <sup>-1</sup>	4.35

The experimental work was divided in two parts:

Part 1 Investigation of the influence of anode materials and current density. In this section, the process was tested on artificial wastewater with the composition detailed in Table 1. The artificial water has the composition of the Weser River (monitoring station of Bremerhaven, Germany, with the chloride contents adjusted to 1.6 g L<sup>-1</sup> Cl<sup>-</sup>). Cu and Zn were adjusted to 6 and 16 mg L<sup>-1</sup>, respectively, while organotins were adjusted to the values shown in Table 1.

Part 2 Treatment of shipyard wastewater. Here, real shipyard wastewater obtained from a shipyard from Northern Germany was used to benchmark the process. The concentration of contaminants and physical properties of the water are presented in Table 2.

In praxis, the concentration range of pollutants in shipyard waters is very wide, and depends on the operation regimes of the shipyards. For example, TBT concentrations of wastewaters have been reported to vary greatly between 5270 and 6,260,000 ng L<sup>-1</sup> [3].

In both experimental parts, a total volume of 1 L wastewater was treated at ambient temperature batchwise in a 0.2 L undivided electrolysis cell made of Plexiglas (Fig. 1). The wastewater was pumped with a peristaltic pump into the inlet located at the bottom of the cell and flowed back into the stirred vessel by gravity. The cathode material was steel (plate of 85 × 100 mm), while the first anode material tested was a bipolar niobium plate (85 × 100 mm) coated with BDD (Nb/BDD with boron content of 2000 mg kg<sup>-1</sup>), which was provided by the Fraunhofer Institute for Surface Engineering and Thin Films. The second anode material was a Ti/IrO<sub>2</sub> (85 × 100 mm) mesh with a surface factor

Table 2  
Composition of real shipyard wastewater

Compound	Concentration	Parameter	Value
TBT, ng L <sup>-1</sup> , Sn	31,331	pH	7.0
DBT, ng L <sup>-1</sup> , Sn	6071	Conductivity, mS cm <sup>-1</sup>	1.248
MBT, ng L <sup>-1</sup> , Sn	162	Redox potential, mV	467
Zn, mg L <sup>-1</sup>	5.25	DOC, mg L <sup>-1</sup>	4.5
Cu, mg L <sup>-1</sup>	0.76	AOX, mg L <sup>-1</sup>	0.035
Fe, mg L <sup>-1</sup>	0.09		

of 2.363 (De Nora Deutschland GmbH). The summary area of the Nb/BDD plate was 170 cm<sup>2</sup>, while that of the Ti/IrO<sub>2</sub> grid was 400 cm<sup>2</sup>. The distance between the cathode plates to the anode was 10 mm.

For all experiment runs, samples were taken from the stirred vessel at different times for analysis of organotins, heavy metals, AOX, and organic matter. All samples were collected in polypropylene 50 mL tubes. During the experiment pH and working voltage were recorded.

The samples for organotins, organic matter, and AOX determination were frozen and sent to an external laboratory. A small amount of reducing agent (sodium sulphite) was added to eliminate residual oxidants in the samples, and prevent the reactions from proceeding. Organotins were analyzed according to DIN 38407-F13: 2001-03, AOX according to EN 1485-H14: 1996-11 DOC according to EN 1485-H14: 1997-08. Samples for heavy metal analyses were stored at 4 °C until being analyzed by means of flame atomic absorption spectrometer FAAS (Perkin–Elmer 1100B) for Cu and Zn, and graphite tube AAS for Fe.

## 2.2. Activated carbon adsorption

Additionally to electrochemical treatment, a granular activated carbon (GAC) filtration step was coupled downstream the electrochemical treatment to eliminate oxidant species, and possible undesired by-products remaining in the wastewater after the electrolysis. The purpose of employing GAC filtration is to couple a “polishing” step after electrochemical treatment. The use of activated carbon adsorption as a “main” process to eliminate organotins from dockyard waters has been studied elsewhere [3]. Although GAC adsorption is able to eliminate a significant load of organotins in water, it is not possible to reach concentrations in the range of tens to hundreds ng L<sup>-1</sup> as some regulations establish [15].

AOX and residual oxidants are eliminated with GAC by two mechanisms:

- Adsorption, to remove chlorinated organics formed during the electrolysis. The van der Waals attraction forces between the large carbon surface (slightly positively charged) and organic molecules are responsible for the binding of those molecules to the carbon surface.

- Reduction of oxidants, for example, free chlorine to chloride. Activated carbon is a mild reducing agent that reacts with strong oxidants when they are adsorbed to the surface.

More exhaustive information about the application of GAC to eliminate chlorinated organics and active chlorine can be found elsewhere [16].

### 3. Results and discussion

#### 3.1. Investigation of the influence of anode materials and current density

Artificial wastewater with the composition shown in Table 1 was treated using both anode materials. The ranges of current densities studied were the following:

- Nb/BDD-Steel: from 8 to 70 mA cm<sup>-2</sup>;
- Ti/IrO<sub>2</sub>-Steel: between 6 and 35.5 mA cm<sup>-2</sup>.

The results for Nb/BDD anode are presented in Fig. 2. The results don't show any large differences in the rate of degradation of organotins (with respect to the charge applied) in the range of current densities investigated. Nonetheless, the degradation of organotin showed that at lower current densities the process consumes less charge than at higher current densities. This behavior implies that at low current densities a more efficient use of the electricity supplied to the cell is achieved, and that increasing the current density results in a decreased current efficiency due to evolution of oxygen and heat generation.

The degradation of TBT with both materials at several current densities is presented in Fig. 3. Here it is possible to observe that there are no large differences between the performances of

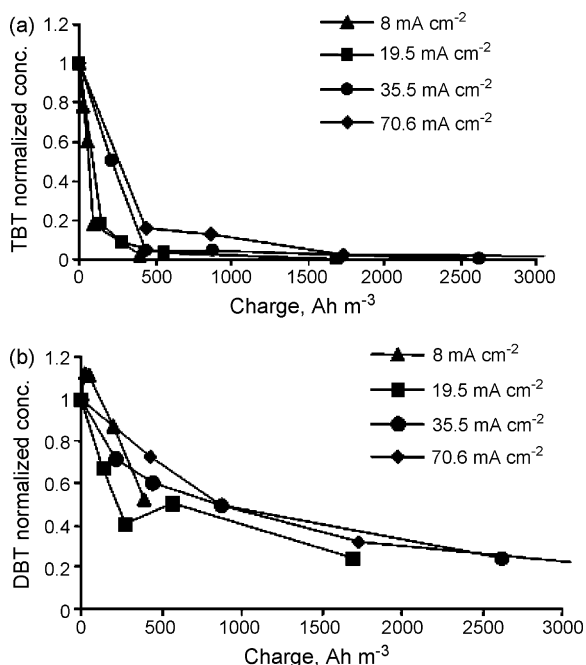


Fig. 2. Degradation of organotins at different current densities using Nb/BDD: a) TBT, b) DBT (concentrations are normalized to the initial concentrations).

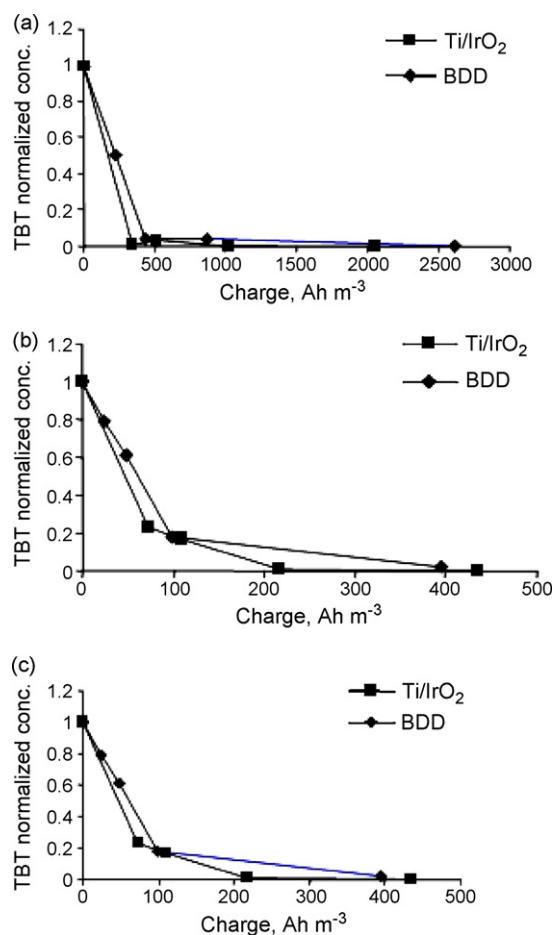


Fig. 3. Electrochemical degradation of organotins with Nb/BDD and Ti/IrO<sub>2</sub> anodes: (a) TBT degradation at 35.5 mA cm<sup>-2</sup>, (b) TBT degradation at 8 mA cm<sup>-2</sup>, (c) DBT degradation at 8 mA cm<sup>-2</sup> using BDD and 6 mA cm<sup>-2</sup> using Ti/IrO<sub>2</sub> (concentrations are normalized to the initial concentration).

both anode materials with model wastewater. This means that the property of high yield of •OH generation reacting with TBT, due to the short life of the radicals occurring mainly in a thin reaction layer at the BDD anodes, does not make a difference in the degradation of organotins. This reaction mechanism is limited by the mass transport phenomena of organotins to the BDD surface. Additionally, the oxidation of organotins is strongly assisted by the presence of Cl<sup>-</sup> in the water, which is oxidized to Cl<sub>2</sub>, hypochlorite, chlorate, and perchlorate due to the electrolysis, and also other oxidants such as O<sub>3</sub>, H<sub>2</sub>O<sub>2</sub>, and peroxides may be present due to the oxidation of the electrolyte support [14,17,18]. Probably these reactions occur at similar rates in both materials, thus BDD does not exhibit a superior performance than Ti/IrO<sub>2</sub>. An ecotoxicological investigation [12] provided further evidence that the oxidation is mediated by oxidants other than the short lived •OH and by oxidants remaining longer stable in solution causing toxic effects to the test organisms.

#### 3.2. Treatment of shipyard wastewater

Wastewater from a shipyard in northern Germany was treated in the laboratory to demonstrate the applicability of the process with real water. The final concentrations of organotins after the

Table 3  
Concentration of pollutants in shipyard wastewater after electrochemical treatment

Parameter	Nb/BDD		Ti/IrO <sub>2</sub>	
	After electrolysis <sup>a</sup>	After GAC	After electrolysis <sup>b</sup>	After GAC
TBT, ng L <sup>-1</sup> , Sn	8.17	<8	16.75	<8
DBT, ng L <sup>-1</sup> , Sn	307.15	<8	422.45	<8
MBT, ng L <sup>-1</sup> , Sn	357.38	58	135.53	75.3
AOX, mg L <sup>-1</sup>	0.85	0.025	1.10	0.049
DOC, mg L <sup>-1</sup>	6.3	2.9	6.6	5.4
Redox potential, mV	907	422	929	590

<sup>a</sup> After applying 960 Ah m<sup>-3</sup>.

<sup>b</sup> After applying 2260 Ah m<sup>-3</sup>.

electrolysis and after GAC treatment are summarised in Table 3, while the normalized concentrations during the electrolysis are presented in Fig. 4.

The experiments showed similar results compared to artificial water experiments. After applying 1000 Ah m<sup>-3</sup> most TBT is eliminated. Nevertheless, AOX is increased from 0.035 to 0.85 mg L<sup>-1</sup> and 1.1 mg L<sup>-1</sup> for BDD and Ti/IrO<sub>2</sub>, respectively, even if the initial chloride concentration of the water was only

0.45 g L<sup>-1</sup> (the artificial water had a concentration of 1.6 g L<sup>-1</sup>). Further investigations are needed to characterize the influence of the initial chloride concentration, current density, and treatment time on AOX levels. Only after GAC adsorption AOX is eliminated from the effluent as shown in Table 3.

#### 4. Conclusions

The experimental work of this study showed that electrochemical treatment is a technology suitable for eliminating organotins in dockyard waters down to very low concentration targets in the range of 100 ng L<sup>-1</sup>. Reaching such low concentrations has proved difficult with “off the shelf” technologies such as activated carbon [3]. The inorganic tin formed as final product of degradation can be considered harmless at such concentrations, and disposed into the environment. The results indicated that the performance of the electrolysis employing Ti/IrO<sub>2</sub> and BDD anodes was in a similar range, contrarily to the initial hypothesis that the high yield of hydroxyl radicals at BDD anodes would result in a much faster degradation rate using BDD. Reasons are due to the presence of active chlorine compounds starting from electrochemical chloride oxidation, and other oxidants, which also lead to the degradation of organotins. Such behavior was confirmed by other experiments conducted to decompose the blue dye Indigo Carmine [12]. These experiments showed when chloride concentration in the water decreased, BDD clearly outperformed Ti/IrO<sub>2</sub>.

The oxidation of chloride to active chlorine compounds does not only degrade organotins but also causes an increase of AOX concentrations in the water with both anode materials tested. This fact, combined with the need to eliminate residual oxidants remaining in the water after electrolysis (e.g. hypochlorite and hypochlorous acid, which cause a high toxicity in the water), made it necessary to couple electrochemical treatment with activated carbon adsorption. Activated carbon proved suitable to eliminate both problem substances (AOX and residual oxidants). In most cases after a contact time of 5 min the treated effluent had AOX and toxicity levels lower than the untreated wastewater. Thus, the use of electrolysis coupled with activated carbon seems to be a suitable combination to eliminate organotins, and the electrolysis by-products.

Further research is needed to characterize the formation and possible degradation of AOX. The influence of chloride concen-

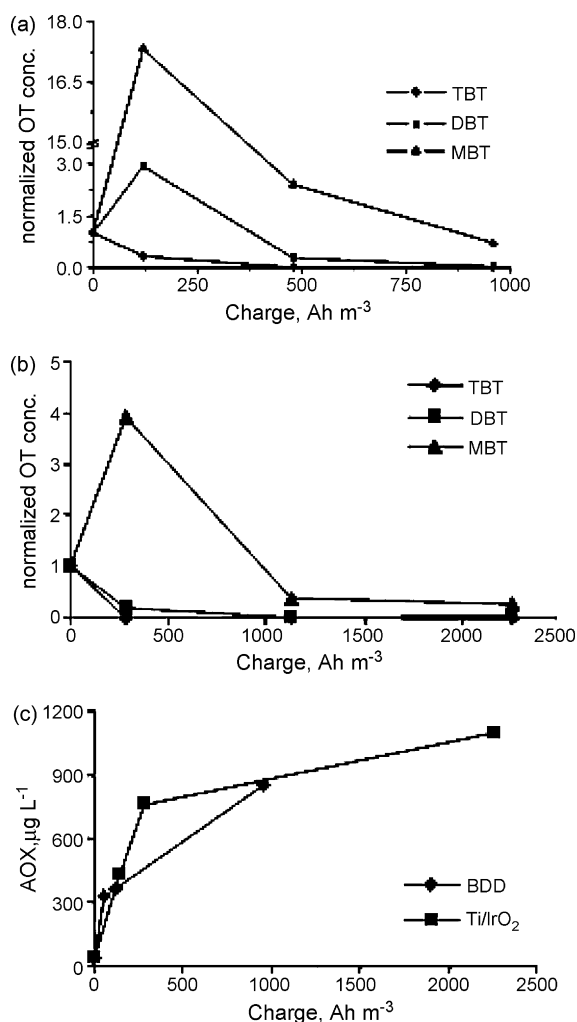


Fig. 4. Degradation of organotins at 19.5 mA cm<sup>-2</sup> using (a) BDD, (b) Ti/IrO<sub>2</sub>, (c) AOX concentrations during treatment.



tration and contents of organic matter in the water, as well as the residence time in the electrochemical reactor with different anode materials should be investigated in more detail in order to assess the technical feasibility of applying this process in a larger scale.

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