

## Corona Above Water Reactor for Systematic Study of Aqueous Phenol Degradation

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*A small batch reactor is developed to study the removal of phenol from a thin layer of water by creating pulsed corona discharges above the water. Pulses of up to 40 kV are applied with a duration of ~50 ns and an energy of ~60 mJ. In this CAW (Corona Above Water) reactor an ozone yield of upto 90 glkWh is obtained in ambient air. The phenol degradation is 48 glkWh, using a 1 mM initial concentration in demineralized water. The degradation yield increases to almost 100 glkWh by adding to the water either H<sub>2</sub>O<sub>2</sub> or Fe<sub>2</sub>SO<sub>4</sub> or NaOH. The first two additions are considered to increase to amount of OH radicals. In the case of NaOH addition it is observed that much more ozone dissolves in the water. The addition of the OH scavenger t-butanol shows that in most cases the main oxidation route of phenol in the CAW reactor is direct ozone attack.*

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**KEY WORDS:** Pulsed corona discharge; advanced oxidation technology; phenol degradation.

### 1. INTRODUCTION

Extensive industrial growth is reducing the available water resources. The implemented techniques for industrial wastewater treatment, in order to reuse the water, are expensive and often ineffective. A promising technique is pulsed corona discharge, which is one of the so-called Advanced Oxidation Technologies (AOTs).

Pulsed corona, already used for gas cleaning,<sup>(1)</sup> has also been studied for implementation in waste water treatment. A number of studies have been performed on corona creation in water, both for point-plane and

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plane–plane electrode configurations.<sup>(2–6)</sup> However creation of the corona in the gas above the liquid surface showed an increase in efficiency up to 10 times, compared to generation of a corona within fluid.<sup>(7)</sup> In the framework of the European project *ytriD*,<sup>(8)</sup> the effects of the corona (created above the liquid) on water treatment are being studied.

Pulsed corona is a source for a wide range of reactive species, created both in the gas and the liquid phase. Corona is able to produce ozone, hydrogen peroxide, hydroxyl radicals and others.<sup>(9)</sup> Ozone is considered as one of the most powerful radicals because of its long lifetime and high oxidation potential. Radicals are transferred into the liquid phase, where the reactions with the pollutants take place. The CAW (Corona Above Water) reactor for studying that process was proposed and tested earlier.<sup>(10,11)</sup> Phenol was chosen as a model compound to demonstrate the oxidation process, because it is a good indicator for a lot of industrial waste, its well-known oxidation pathways and the availability of literature on its degradation by other methods.

The main goal of this article is to investigate the influence of electrical, chemical and physical parameters, like electrode configuration and additives, on ozone production and phenol removal. The results will help in the optimization and scale up of the process.

## 2. MATERIALS AND METHODS

### 2.1. The Reactor

For the experiments a batch reactor has been used (Fig. 1). The reactor is 38 cm long and 8 cm wide. The reactor consists of two parts. The upper part, with 1–4 electrodes has an inner height of 1.6 cm. Connectors for a gas flow system are placed, as well as optical lens holders. The lower part, containing liquid, is equipped with inputs for connecting the tubing system of the water flow. There are two bottoms with different heights available. They have 1.2 and 2.5 cm of inner heights, respectively.

The HV electrodes are made from stainless steel and have a diameter of 0.2 mm. To prevent sparking special holders of the wires have been implemented, as can be seen in Fig. 1. The grounded electrode is placed at the bottom side, outside the reactor.

Some of the experiments were performed with a circulating liquid flow using a tubing pump (Cole Palmer Masterflex I/P) setup. The standard flow through the CAW reactor was 0.3 L/min. A gas flow system can be connected to the reactor. This system is capable of flushing different gasses (O<sub>2</sub>, N<sub>2</sub>, Ar<sub>2</sub>).

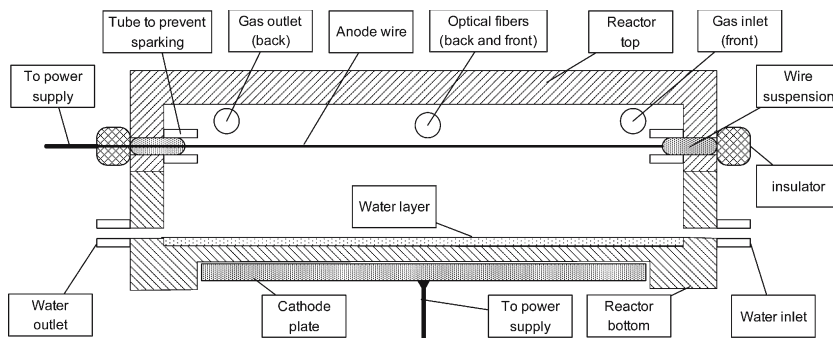


Fig. 1. Corona Above Water reactor layout (not to scale).

## 2.2. Electrical Set-up

The pulsed power supply is a triggered spark gap switched capacitor followed by a transmission line transformer. The polarity of the pulses is positive and the frequency is up to 100 pps.

During experiments the problem with spontaneous triggers of the spark gap occurred. A new design of closed and air-flushed spark gap has been implemented. Also changes in triggering unit have been made, installing a fast thyristor switch (Belkhe). Those improvements give much better recurrence of the energy of the corona pulses. The typical waveforms can be found in Refs. 10 and 11. Compared to Ref. 12, the system operates at lowered voltage and current for better stability; thus the energy value for performed experiments is in the range of 50–60 mJ per pulse. The setup is mostly operated at frequencies from 5–15 Hz with the pulse duration of < 100 ns, with the  $\sim 40$  kV voltage peak and 80 A of current.

## 2.3. Measurements System

The ozone concentration above the liquid is measured using the light absorption technique. The light emitted by the deuterium lamp is absorbed by ozone. Changes in the light intensity, at a wavelength of 256.7 nm, which indicates the presence of ozone, are collected by the OceanOptics (HR-2000) spectrometer. The design of the reactor provides the possibility to measure the ozone concentration directly above the liquid during the corona energization, giving useful information on the ozone uptake.

The phenol concentration is measured using Laser Induced Fluorescence.<sup>(13)</sup> The LIF setup is constructed with a YAG laser (Quantel Brilliant Ultra) with 4 mJ/pulse output at 266 nm and an spectrometer

(Ocean Optics HR2000). The calibration curve gives a very good fit ( $R^2 = 0.98$ ). Comparative tests with HPLC technique show very small differences ( $< 2\%$ ) between these two measuring techniques. In the measurements presented in this paper, a 1 mM/L phenol solution is used as a starting concentration.

### 3. DISCUSSION OF THE RESULTS

To explore the influence of different factors on the efficiency of the waste water treatment process, several experiments have been carried out. These experiments can be divided into three groups, for investigation of the influence of

1. Electrical conditions: energy of the corona, repetition frequency, numbers of the electrodes
2. Physical conditions: layer thickness, velocity of the liquid, reactor dimensions.
3. The chemical conditions: pH value, additives and gas composition

#### 3.1. Effect of Corona Energy and Pulse Repetition

First experiments were performed in a reactor filled with ambient air only. The ozone concentration was measured for different values of charging voltage and frequency. The power supply unit was set to give the voltage at reactor of 36, 40 and 46 kV charging voltage, which corresponds to 35, 54 and 71 mJ ( $\pm 5$ mJ) of energy per pulse, respectively. As is seen in Fig. 2, an increase of the energy per pulse at a fixed repetition frequency, leads to faster build-up of the ozone concentration. The same trend is observed when the repetition frequency was increased from 5 to 15 Hz for

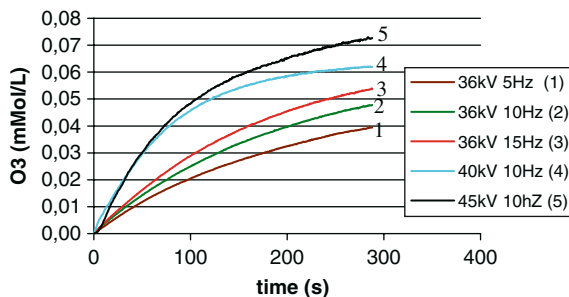


Fig. 2. Ozone concentration in the air-filled reactor depending on voltage and frequency.

**Table I.** Ozone Yields

environ. & setting	g/kWh
Air – 10 Hz, 36 kV	40
Air – 10 Hz, 40 kV	87
Air – 10 Hz, 45 kV	64
Air – 15 Hz, 36 kV	26
Air – 5 Hz, 36 kV	62
Phenol – 10 Hz, 40 kV	45
O <sub>2</sub> – 10 Hz, 40 kV	190

a fixed value of energy per pulse. The increase in energy value, has bigger influence on ozone production than increase in repetition frequency.

Yields of ozone production in g/kWh have been calculated for the above conditions (Table I). The most effective production appears with the charging voltage set to 40 kV and 10 Hz, which corresponds to 54 mJ of energy per pulse dissipated in the discharge.

Those results show that the ozone generation process is strictly connected to corona energy and repetition rate. The high values of energy are desirable only at the beginning of the process. It helped to obtain the maximum concentration of ozone in shorter time. After reaching the maximum concentration of ozone the energy is wasted, because concentration of ozone does not increase, most likely due to self-decomposition.<sup>(12)</sup>

For the next experiments the setting of 40 kV peak voltage and frequency of 10 Hz was used.

### 3.2. Effect of Electrode Length on Ozone Generation and Phenol Removal

For this experiment the reactor was equipped with one, two, three or four electrode wires. All the measurements were taken for the same value of energy per pulse. A significant difference in ozone production, above the 1 mM phenol solution, for the four different electrode lengths, has not been observed. However, as can be seen in Fig. 3, there is a noticeable influence on the phenol removal. Despite longer electrode length the number of created ozone molecules remain the same due to constant value of energy dissipated in the reactor. The uniform corona distribution above the liquid surface is the important factor for the diffusion of reactive species in the air phase and their distribution in the solution.<sup>(14)</sup>

### 3.3. Effect of Different Solutions on Ozone Generation

In this experiment the influence of different liquid solutions on the process was examined. The reactor worked in batch-like configuration. The experiment was done for the reactor filled with: (i) only ambient air

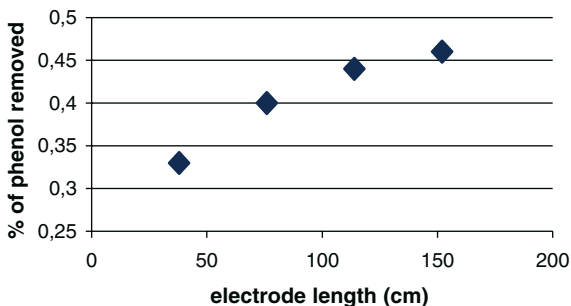


Fig. 3. Effect of electrode length on phenol removal.

(0.85 L), (ii) air plus a 100 mL of deionized water, (iii) as in (ii) but now the phenol solution was added in concentration to obtain 1 mM/L. The distance between the electrode and the liquid surface was  $\sim 15$  mm. A significant difference of the ozone concentration was observed for the reactor with the three different fillings (Fig. 4).

As can be seen in the graph, the initial lines of ozone concentration are similar for all three cases. After around 20 s the curves take different shapes. It is clearly seen that type of filling highly influences the ozone concentration in the reactor. This is expected because in presence of water and phenol solution, the mass transfer process occurs. Ozone molecules diffuse into the liquid where they are scavenged by the water molecules as well as transited in to  $\text{OH}^*$  radical.<sup>(15,16)</sup> In case of phenol solution additional, direct reaction with phenol molecules takes place.<sup>(17)</sup>

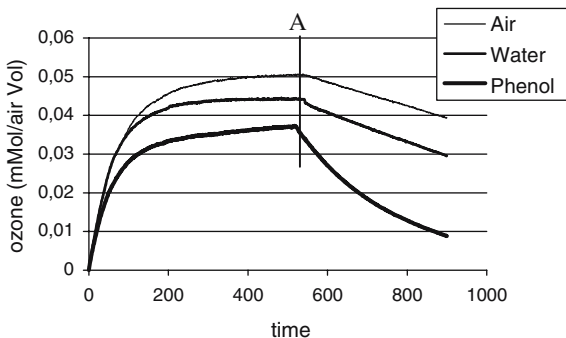


Fig. 4. Ozone concentration in reactor filled with air, deionised water and phenol solution, A – corona off; (38 kV, 10 Hz, 54 mJ/pulse, 100 ml of liquid, 1 mM/L phenol solution).

The total ozone amount after 500 s has been calculated. In the reactor filled with air 27 mM of ozone were produced. In the presence of deionized water 4 mM of total ozone created had been diffused in to the water. The presence of phenol solution decreased that amount, leading to 19 mM of ozone in the air after 500 s.

In the next part of the experiment the corona was turned off after 500 s (line A on the graph) and the decay of ozone has been observed. As is shown in Fig. 4 the curves for ozone concentrations in reactor filled with air and water have similar slopes. As it can be predicted, for reactor filled with air the main mechanism for ozone degradation is its self-decomposition in the volume.<sup>(12)</sup> The layer of liquid has no significant influence on the ozone decomposition. When the maximum level of ozone saturation in water is reached no more ozone molecules are transferred into the liquid phase, thus the decomposition in the volume and at the walls becomes predominant.

In presence of a phenol solution the ozone molecules in the boundary phase<sup>(18)</sup> react with phenol and its by-products allowing more ozone to transfer from the air phase. Therefore a faster decrease of the ozone concentration above the liquid is observed. The decomposition curve has an exponential decay.

### 3.4. Effect of Solution Layer Thickness on Ozone Generation

The reactor was filled with 50, 100, 150 and 200 mL of 1 mM phenol solution, which corresponds to 1.6, 3.3, 4.9 and 6.6 mm of liquid layer thickness, respectively. To keep the air volume fixed, reactor bottoms with different heights were used. The corona was operated with 55 mJ energy per pulse and repetition frequency of 10 Hz. The ozone concentration above the larger volume of liquid increases faster due to lower air volume. After around 300 s. the ozone concentration for all the cases reaches the same value (Fig. 5).

Figure 6 shows the dependence of the phenol removal on the liquid volume after 5 min. corona treatment. Although the relative removal goes down with increasing amount of liquid, the total amount of phenol removed increases. This is agreement with the experiment with exponential decay of phenol as observed by Hoeben.<sup>(7)</sup> Table II shows that the phenol removal yield increases a factor 3 when the layer thickness increases a factor 4, i.e. an almost proportional dependence.

The crucial parameter is the volume to surface ratio.<sup>(14)</sup> It can be clearly observed that this ratio highly influences the ozone uptake as well as the phenol removal. The decrease of ozone concentration at thicker water layers is caused by the presence of the phenol solution. Decreasing

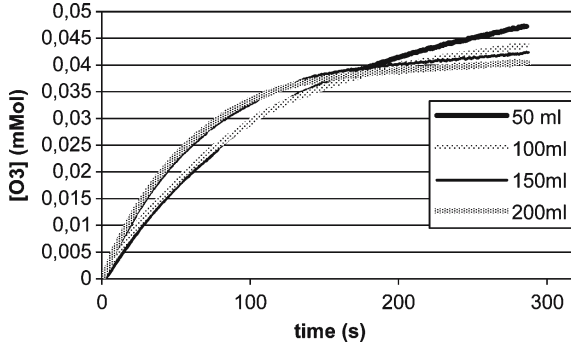


Fig. 5. Effect of liquid layer thickness on ozone concentration (38 kV, 10 Hz, 54 mJ/pulse, deionized water).

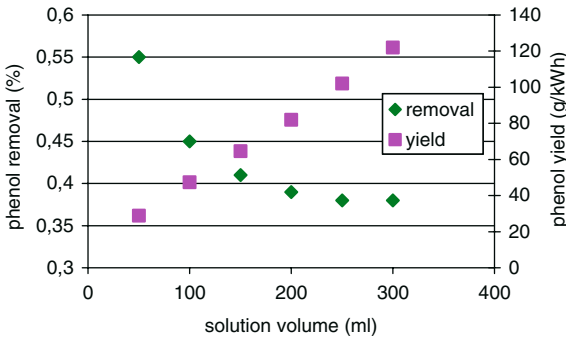


Fig. 6. Effect of solution volume on phenol removal (38 kV, 10 Hz, 54 mJ/pulse, 1 mM/L phenol solution).

the air volume in the reactor leads to an increase of the ozone concentration. With the decreased liquid thickness, ozone can easier penetrate the solution, leading to higher phenol removal. Increasing the liquid volume above the 200 mL neither noticeably affected the ozone uptake, nor the phenol removal. This indicates that 6.6 mm is the maximum depth of the solution where the reactions between ozone and other radicals and phenol take place. In literature<sup>(18)</sup> the figure of 2.5 mm can be found. This difference may be explained from the turbulence caused by the high voltage pulses. In the CAW reactor the water surface shows considerable movement due to these pulses. In Ref. 18 the pulses were less strong resulting in less mixing of the liquid. Another mechanism is the reactivity of the oxidizing species preventing them from deeper penetration in to the phenol solution.



**Table II.** Phenol Removal and Yields (if not Indicated Otherwise: Charging Voltage = 40 kV, Energy pp = 54 mJ, f = 10 Hz, Electrode Length = 152 cm, Layer Thickness = 3.2 mm, 100 ml of 1 mM/L Phenol Solution with pH = 5.5)

Exp. condition	% removal	G (g/kWh)
electrode L. = 38	33	34.7
electrode L. = 76	40	42.1
electrode L. = 114	44	46.2
electrode L. = 152	46	48.3
liquid flow	52	54.6
layer thickness = 1.6 mm	55	28.9
layer thickness = 3.2 mm	45	47.3
layer thickness = 4.8 mm	41	64.6
layer thickness = 6.4 mm	39	81.9
Add butanol – 0.5 mM	44	46.2
Add butanol – 1 mM	40	42.1
Add butanol – 2 mM	40	42.1
Add H <sub>2</sub> O <sub>2</sub> – 0.5 ml	82	86.2
Add H <sub>2</sub> O <sub>2</sub> – 1 ml	91	95.6
Add H <sub>2</sub> O <sub>2</sub> – 2 ml	99	104.1
Add FeSO <sub>4</sub> – 1 mM	69	72.5
Add FeSO <sub>4</sub> – 2 mM	86	90.4
Add FeSO <sub>4</sub> – 3 mM	94	98.8
pH = 2.1	29	30.5
pH = 3	35	36.4
pH = 4	43	45.2
pH = 6	57	60.3
pH = 9.2	65	68.7
pH = 10	91	95.2

### 3.5. Effect of Liquid Flow on Ozone Diffusion and Phenol Removal

The reactor was connected to a pumping system with continuously circulating solution. Water flow speed was set to 0.3 L/min. The liquid layer was ~3 mm. The ozone concentration above the 1 mM phenol solution was measured, both with flow turned on and off. After 5 min samples of phenol solution were collected to measure the removal rate.

The rate of ozone uptake in the continuous-flow reactor is significantly higher than in the batch reactor for the same amount of liquid (Fig. 7). Due to additional mixing of flowing liquid, the ozone was transferred more easily into the liquid bulk. The liquid flow increased the ozone uptake by 23% compared to the non-flow situation. However, at the same

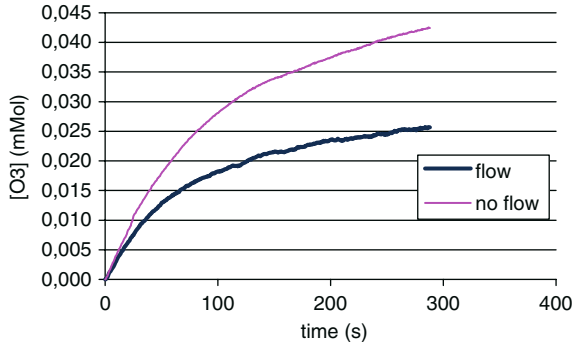


Fig. 7. Effect of liquid flow on ozone concentration (38 kV, 10 Hz, 54 mJ/pulse, 1 mM/L phenol solution).

time, phenol removal increased only 7%, from 45% to 52% (Table II). Obtained results indicate that for destroying one phenol molecule three molecules of ozone are needed, which is in agreement with other works.<sup>(20)</sup>

The effect of liquid flow on ozone mass transfer is also visible, when the corona operates in oxygen atmosphere (Fig. 8). The initial rate of ozone generation in oxygen was five times higher than in an air atmosphere. The ozone concentration reached the maximum level of 0.021 mM after 100 s, which is much faster than in an air atmosphere. Ozone yield reaches the level of 190 g/kWh, which is around two times higher than the highest yield in ambient air (Table I). Obtained results showed that CAW reactor is capable of obtaining yields of commercial ozonators<sup>(16)</sup> 200 g/kWh in oxygen and 90 g/kWh in ambient air.

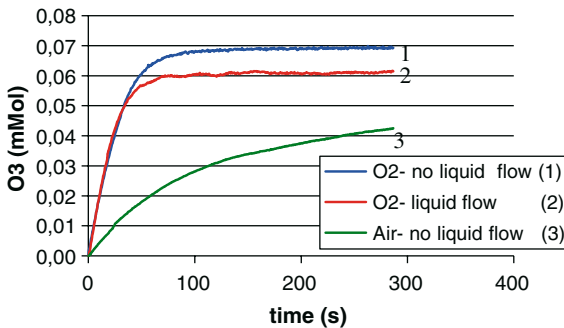


Fig. 8. Ozone concentration in O<sub>2</sub> atmosphere.

In the oxygen atmosphere the phenol removal increased to 61%, giving yield of 64 g/kWh.

### 3.6. Effect of pH on Ozone Production and Phenol Removal

Ozone is known to react with aromatic compounds by two complementary reaction paths. It can react directly with phenol or via the radicals produced by self-decomposition. Increase in pH value promotes the ozone attack on phenol via OH radical reaction.<sup>(15)</sup>

The different amounts of NaOH and HCl have been added to 150 mL of 1 mM/L phenol solution to obtain desired levels of pH. Next, the solutions were treated in batch configuration. The ozone generation as well as phenol removal were measured.

Figure 9 shows that the pH value of the treated solution has a big influence on the ozone uptake. At low pH value the ozone concentration above the liquid is higher than in case of more basic solutions. At pH=10.2 the line takes a different shape. A maximum in the ozone concentration is reached after around 80 s. Then the ozone concentration drops and starts rising again after 180 s. This effect is, to our knowledge, not previously mentioned in the literature. Since high pH promotes self-decomposition of ozone, the ozone concentration in solution decreases, allowing more ozone to diffuse into the liquid phase. This enhanced diffusion process causes the much lower ozone concentration in the air above the water.

Figure 10 shows the dependence of pH solution on phenol removal. With the pH level of 2,29% of the phenol had been removed. With increasing the pH value the removal rate increase. At pH~5, regular phenol solution, 45% of phenol had been removed. Increasing pH value

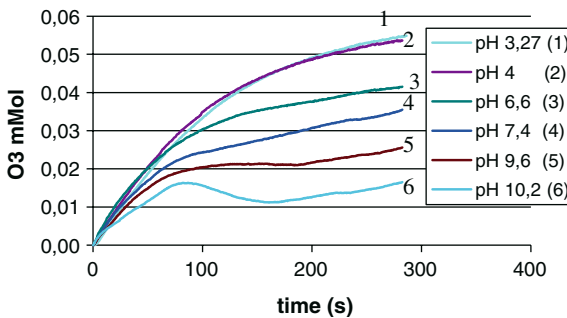


Fig. 9. Effect of solution's initial pH value on ozone concentration.

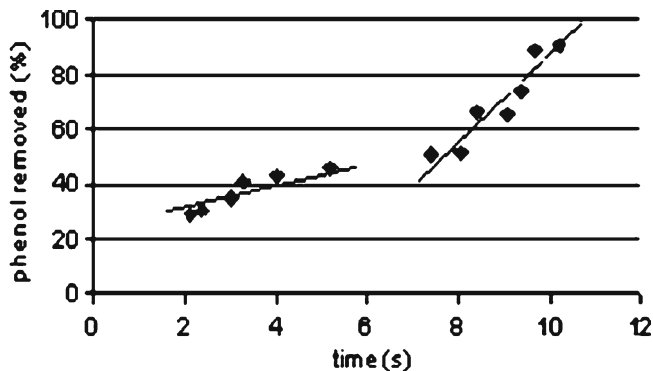


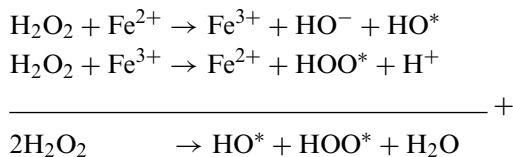
Fig. 10. Effect of pH value on phenol removal.

above 6 causes the removal rate to increase with even higher rate. At pH = 10.2 the phenol removal reached 90%. The yields of phenol removal are shown in Tabel II. The experimental results are in agreement with reaction mechanism reported by other authors.<sup>(21,22)</sup> In acidic condition the direct reaction between ozone and phenol molecules becomes predominant. At high pH values OH\* radicals decompose less likely allowing the phenol molecules to react with them. Thus the phenol decomposition increases. It confirms that in the high pH the free radicals reaction plays a major role in the phenol removal process.

The pH value of the regular phenol solutions drops during the treatment. This is explained by creation of acids, like muconic acid and acetic acid, during the phenol oxidation process.<sup>(7,9)</sup> That effect negatively influences the efficiency of the process with time. As has been shown slight increase of pH above 6 can significantly improve the phenol removal.

### 3.7. Effect of Fe<sup>2+</sup> and H<sub>2</sub>O<sub>2</sub> on Phenol Removal

Several metals have special oxygen transfer properties, which may improve the efficiency of the treatment process. The well-known Fenton's reaction involved iron-catalysed hydrogen peroxide.<sup>(9)</sup> The iron ion has a strong catalytic power to generate highly reactive hydroxyl radicals (OH\*) through the reactions



In the experiments 100 mM/L  $\text{FeSO}_4$  and 30%  $\text{H}_2\text{O}_2$  solutions were used. The influence of different volumes of those solutions on the process was studied. Phenol starting concentration was 1 mM/L and the treatment time was 5 min.

For both additives no significant changes in ozone concentration profile were observed. The differences stayed within measurement error.

The addition of  $\text{H}_2\text{O}_2$  significantly improves the removal rate from 45% for pure phenol solution, up to nearly 99% in case of addition of 2 mL  $\text{H}_2\text{O}_2$  solution. The phenol yield increases to 104 g/kWh.

The same effect was observed with the addition of  $\text{FeSO}_4$  solution. The removal rate increased up to 94% with addition of 3 mL of  $\text{FeSO}_4$  solution. The phenol yield rises up to 99 g/kWh.

The experiment showed the hydrogen peroxide as a powerful reagent, which has the ability to start several radical reactions, leading to phenol decomposition. It can be potentially used in the phenol treatment process.<sup>(21)</sup> On the other hand, the experiments with  $\text{Fe}^{2+}$  ion also showed a big influence on the removal process. Considering the increase of phenol removal rate in presence of  $\text{Fe}^{2+}$  ions, it has been found that the concentration of hydrogen peroxide produced in the liquid during the corona process is sufficient to start the Fenton's reaction even without extra additions.

Combination of these two additives causes the reaction with phenol to start immediately, even without the corona. After corona treatment the phenol concentration was below the detection limit. However, brown, hard to extract sediments appeared in the solution. In all other cases described in this paper such sediments were not observed.

### 3.8. Effect of *t*-Butanol on Ozone Production and Phenol Removal

*t*-Butanol is a well-known radical scavenger. It reacts with a wide range of radicals, especially hydroxyl radical, promoting direct reaction between ozone and phenol.<sup>(16)</sup> In the same time it does not react with ozone. Different concentrations of that additive were mixed with the 1 mM/L phenol solution. In this way, the influence of hydroxyl radicals, on the phenol removal process, was studied.

The addition 0.5–2 mM/L of *t*-butanol decreases the ozone uptake by the treated solution. The concentration of ozone above the liquid was higher, compared to the pure phenol solution. The ratio between ozone in air phase and ozone in the liquid place is considered as constant. Ozone dissolved in the liquid may react directly with phenol or degrade through several reactions, into  $\text{OH}^*$  radical.  $\text{OH}^*$  radical react then with phenol. With *t*-butanol presence, the ozone is less likely converted into  $\text{OH}^*$ .<sup>(17)</sup> More ozone molecules are present in liquid phase thus the diffusion gradient

decrease, increasing the ozone concentration above the liquid. In presence of *t*-butanol the direct ozone-phenol reaction becomes predominant.

The phenol removal rate in presence of rising *t*-butanol concentration decreased (Table II). However, above the 1 mM/L of scavenger concentration, the effect saturates indicates that all of the OH\* radicals have been absorbed. Based on these observations, direct ozone reactions were found to be responsible for around 80% of the phenol removal at pH ~ 5.

#### 4. CONCLUSIONS

The influence of different parameters, on the ozone generation and phenol removal process, has been shown. The CAW reactor proves to be an effective source for ozone production. The ozone yields – both in air and oxygen atmosphere – reaches the range of commercial ozonizers.

The CAW reactor is capable to remove 45% of phenol in 5 min of a 100 ml 1mM batch, this is equivalent to an phenol destruction yield of 48 g/kW h. This removal yield is considerably higher than reported previously.<sup>(6,12)</sup> This is explained by two changes. First, a lower pulsed voltage and repetition rate are chosen leading to a higher ozone yield. Second, the initial phenol concentration is now 1 mM instead of 0.1 mM, making it easier to remove a certain amount because of the exponential decay of phenol in this process. In pure oxygen the removal yield increases to 61%, i.e. 64 g/kWh. The increase in the phenol removal yield is much smaller than the increase of the ozone yield. This again shows that the ozone uptake at the water surface is the rate limiting process.

It is shown that the water layer thickness has a big influence on the phenol removal process. The thinner a solution layer is, the higher the removed fraction becomes. With thick layers the removed fraction is lower but the total amount of phenol removed is higher because more phenol is available. Again this trade-off is expected from the first order reaction kinetics of the process.

It has been shown that several possibilities to increase the phenol destruction yield exist. This can be achieved with the addition of either FeSO<sub>4</sub> or H<sub>2</sub>O<sub>2</sub> or NaOH. Promoting the reaction between OH\* radicals and phenol by addition of FeSO<sub>4</sub> and H<sub>2</sub>O<sub>2</sub> highly influences the phenol removal process. With the addition of Fe<sup>2+</sup> ions the removal increases significantly to 95% with the phenol yield of 99 g/kWh. This shows that the amount of hydrogen peroxide, created by the corona process, is sufficient to start Fenton's reaction without extra addition of H<sub>2</sub>O<sub>2</sub>. The addition of small amounts of the radical scavenger *t*-butanol has made it possible to calculate the relative contribution of the direct ozone-phenol reaction. It has been calculated to be responsible for around 80% of the total phenol

removal at  $\text{pH} \sim 5$ . It has also been shown that the ozone uptake is strongly increased at high pH values due to NaOH addition. This again leads to a two times higher phenol removal compared to the plain phenol solution.

The obtained results will help to improve the chemical model, which has been already proposed.<sup>(11,12)</sup> All this information will assist the design of efficient, large-scale water cleaning reactors.

## ACKNOWLEDGMENTS

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