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RESEARCH ARTICLE

STUDY OF DEGRADATION KINETICS OF 2,4,6–TRINITROPHENOL (TNP) FROM WATER BY ATMOSPHERIC AIR COLD PLASMA

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ABSTRACT

The degradation of 2,4,6 – trinitrophenol (TNP)in water sample by atmospheric air cold plasma dielectric barrier discharge (DBD) was studied. The DBD reactor consisted of two electrodes separated by an insulating dielectric barrier with the electric discharge voltage varying from 7.0 to 22 kV. The influence of the TNP initial concentration on the degradation initial rate was studied systematically. A series of experiments with varying TNP initial concentrations from 91.02 to 210.17 mg/L was performed to find the initial degradation rate by the air cold plasma dielectric barrier discharge. From these experiments a TNP degradation kinetic equation based on varying the its initial concentration was established being-$R=\frac{0.0252C_{TNP}}{1+0.0076C_{TNP}}$. From here it was interesting shown that if $0.0076C_{TNP} << 1$ the TNP degradation kinetics obeys the pseudo - first - order reaction, depending on the TNP initial concentrations, such as $\ln(C_t/C_0) = -0.0269t + 0.1605$, $\ln(C_t/C_0) = -0.0197t + 0.0792$, $\ln(C_t/C_0) = -0.014t + 0.0623$ corresponding to the TNP initial concentrations 91.02, 153.3 and 210.17 mg/L respectively. Besides, the influence of TNP initial concentration and electric power on TNP degradation efficiency were determined too.

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INTRODUCTION

Based on a fundamental point of view, matter consists of four fundamental states of matter: the solid, liquid, gas and plasma state, depending on the addition of thermal or energy (Yamamoto, 2007). Plasma is an ionized gas, being as a distinct fourth state of matter after solid, liquid, gas and plasma. From the early research, a plasma was easily produced in gas discharge tubes consisting of two parallel metal electrodes, surrounded by an any gas. Under this condition, few neutral gas molecules in the gas tube are ionized, resulting in the occurrence of electron-ion pairs (Conrads, 2000). Under so the electric field free electrons are accelerated toward the anode, producing a small background current, up to 10-6 A. As the voltage is increased, primary electrons are further accelerated in the direction to the anode, gaining kinetic energy by the applied electric field. The primary electrons can collide and react with neutral gas atoms, producing secondary electron-ion pair (Raizer, 1987). Plasma was used dielectric barrier discharges (DBD)in application to the construction of industrial ozonators, then the dielectric barrier discharges used for environmental purposes, in medicinal and industrial applications (Eliasson, 1987). The scheme of a typical planar DBD device is given in figure 1.

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The reactor consists of two parallel (planar) electrodes separated by a dielectric barrier. Specifically, the dielectric barrier can be constructed by covering at least one of the electrodes with a dielectric material (silica, quartz, mica, ceramic materials,). In most cases, quartz glass is used as a dielectric (Wagner, 2003). The characteristics of an atmospheric pressure DBD discharges í similar to classical gas discharges. Owing to the coverage of on one of the electrodes, DBDs are initiated by the application of an AC powered voltage on the high voltage electrode (anode) (Kogelschatz, 2003). Plasma is electrically neutral due to its electron density balanced by that positive ions (Mercedes López, 2019; Yi-Ming Zhao, 2019). Due to plasmas are able to produce very high concentrations of energetic and chemically actives species excited stats like electrons, ions, atoms and free radicals. It was shown that oxygen atoms and $^{\bullet}OH$ radicals effectively generated in atmospheric air discharges play a key role for oxidation process. Therefore, it leads plasma technology to be widely used in practice, especially, for treatment of wastewater containing recalcitrant organic compounds being a clean technology, with no additional chemicals needed (Hoffmann, 2013; Zhitong Chen, 2017). Besides, cold plasma significantly removed odor from wastewater, enhanced dissolved oxygen and reduced the concentration of chemical oxygen demand. However, cold plasma significantly produced the concentrations of nitrite and

nitrate that were undesirable. Other disadvantages of treating with cold plasma were conductivity increase and pH reduction. Generally, nitro aromatic compounds are compounds that contain one or more nitro functional groups ($-NO_2$) making an explosive compound used globally (Rodriguez, 2012). The presence of nitro aromatic compounds in wastewater, because of environmental concern, must be treated before it is discharged (Marvin-Sikkema, 1994; Meng-Wen, 2008; Jian Long Wang, 2012). In addition to traditional treatment methods, advanced oxidation processes (AOPs) to degrade nitro aromatic compounds (TNP) from wastewater have attracted a great attention (Hoffmann, 2013; Zhitong Chen, 2017). Among the AOPs, the atmospheric air cold plasma using reactive species like $^{\bullet}O$, $^{\bullet}H$, $^{\bullet}OH$, H_2O_2, $O_3$to decompose nitro aromatic compounds in wastewater was proven to be the most effective method and the wastewater after treatment by plasma dielectric - barrier discharge (DBD) was nontoxic (Bergendahl, 2004; Manoj Kumar Reddy, 2012; Marotta, 2011; Magureanu, 2008; Hsu-Hui Cheng, 2007; Nguyen Van Hoang, 2008; Alexander Fridman, 2008;) Emile S. Massima Mouelel, 2018; Bruggeman, 2016; Cathey et al., 2008). The decomposition of TNP by DBD is similar to APOs, occurring very complicated through many intermediate stages. So this shows that it is difficult to give an accurate kinetic equation describing the whole process of TNP decomposition. In this paper a series of experiment was performed to attempt try to give a kinetic equation that describes the rate of decomposition depending on the initial concentration of TNP. The reaction rate at the TNP initial concentration is not be depended by the intermediate products of reaction degradation in plasma reactor. Such kinetic equation can be applied in practice for the treatment model at different scales. Among the many nitro aromatic compounds, TNP was an objective selected to investigate the atmospheric air cold plasma - treated degradation kinetics, in detail. Because this intro compound is present in many surface water sources in our country.

MATERIALS AND METHODS

Chemicals: 2, 4, 6 – Trinitrophenol (TNP)and other chemicals of analytical purity grade were obtained from Merck company, no pretreatment. All the solutions were prepared prior to carrying out experiments and kept at room temperature.

Apparatus

- Analytical instruments used in this work, include HPLC Model HP 1100, using diode-array detector (DAD), Agilent (USA), Spectrophotometer UV- Vis Agilent 8453 (USA) and DR/890 Colorimeter, HACH, for COD analysis.
- Air cold plasma DBD reactor is presented in the Fig 2.

The reactor consists of a HV pulse generator connected to electrode system with water thin layer. A pump was used to supply air (2-4 L/min) to the plasma chambers inside and outside the quartz tube to enhance ozone generation and the concentration of ozone dissolved in water. A metering pump was used for wastewater circulation of 1.2 L/min into the reactor to augment the TNP degradation efficiency in the reactor. Dielectric-barrier discharge (DBD) is the electrical discharge between two electrodes separated by an insulating dielectric barrier (Hamouda, 2017; Mahvi, 2005).

The discharge is performed on a system of two coaxial electrodes separated by a thin insulating layer. In our experiment, the plasma DBD reactor consisted of 2 electrodes assembled with a grounded central electrode (21 mm in diameter) of in ox steel and an outer copper electrode (HV) wrapped around the outside of the quartz tube (35 mm in diameter). Two coaxial electrodes are fixed on a teflon insulated base.

The plastic base is drilled to bring water out from the plasma reactor. Insulating glass tubes always keep high voltage to create electrical sparks that spread evenly around the pipe without the breakdown of the pipe. The electrodes were connected to a high voltage source (about> 10kV). The DBD cold plasma was formed between water layer and inner surface of the tube. It is due to the discharge of sparks in the air from the outer surface of the water layer to the inside of the glass tube.

Atmospheric air cold plasma DBD treatment: First, the water sample is supplied into the inner electrode, then the water will flow over the outer surface of the pipe wall downwards and form a 1-2mm thin film (depending on the selected pump velocity). When two electrodes were adjusted to reach a large enough high voltage (about> 10kV), the plasma DBD will be formed in the reactor chamber between two electrodes. Under this condition, air cold plasma DBD was formed, consisting of ozone (O_3), UV and other reactive reagents such as, $^{\bullet}OH$, $^{\bullet}H$, H_2O_2 as reported in (Restiwijaya1 et al., 2019; . Malanichev, 2016; Meng-Wen Chang et al., 2008) as following:

$$O_2 + hv \rightarrow O + O$$
$$O + O_2 \rightarrow O_3$$
$$O_3 + OH^- \rightarrow O_3^{*-} + {}^*OH$$
$$O_3 \rightarrow O^{*-} + O_2$$
$$O^{*-} + H_2O \rightarrow {}^*OH + OH^-$$
$$e^- + H_2O \rightarrow {}^*OH + H^* + e^-$$
$$O + H_2O \rightarrow 2\,{}^*OH$$

These active components are very strong oxidizing agents to degrade pollutant compounds (TNP) in wastewater. The shapes of plasma DBD produced by the atmospheric air cold plasma reactor are presented in Fig 2. I = 3.5 mA); (U= 16kV, I = 10mA); (U= 19 kV, I = 16 mA); (U = 21 kV, I = 22 mA). To enhance the decomposition efficiency of pollutant compounds (TNP), the wastewater sample should be circulated. After a certain amount of time, water samples were withdrawn from reactor for TNP analysis by HPLC. Besides analyzing the decrease of TNP concentration, the factors such as applied voltage, the TNP initial concentrations influencing decomposition efficiency of TNP were also studied in detail to establish the decomposition kinetic equations.

Analytical methods

Determination of TNP concentration: The concentration of TNP in the sample during experiments was determined by HPLC method with the following parameters:

- Detector - Diode Array
- Column of Hypersil C18 (200 x 4 mm)
- The mobile phase consists of Acetonitrile /water = 65/35 (by volume), pH =7.
- Pressure: 280 bar.

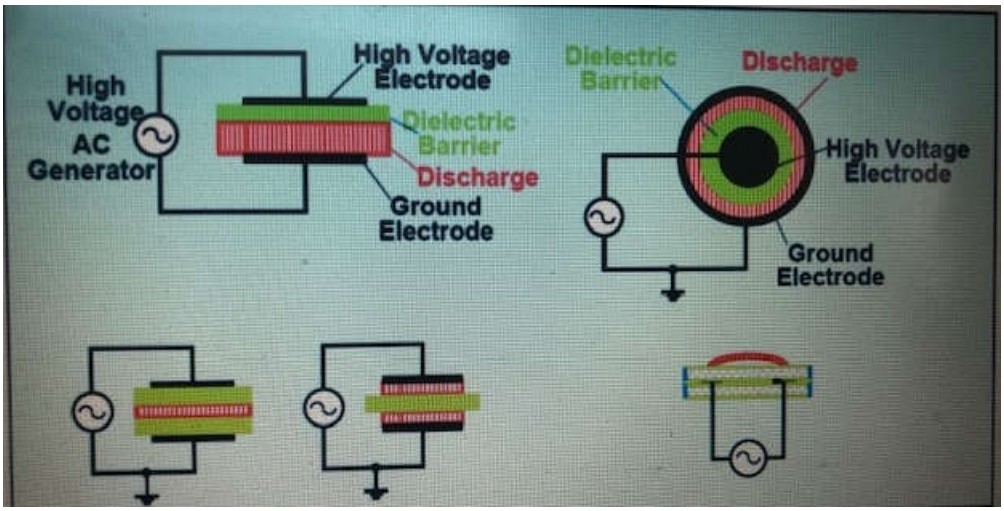

Figure 1. Configuration of planar dielectric barrier discharge reactor

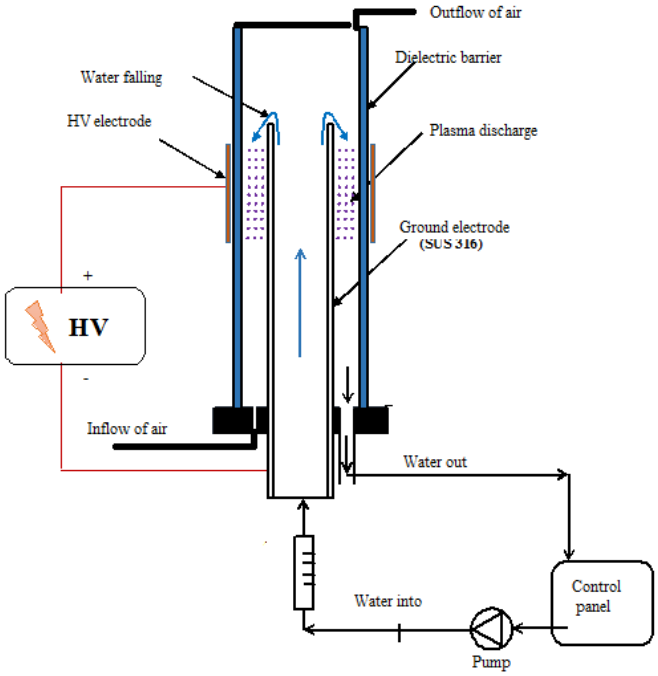

Fig.2. Schematics of batch reactor configuration for plasma treatment

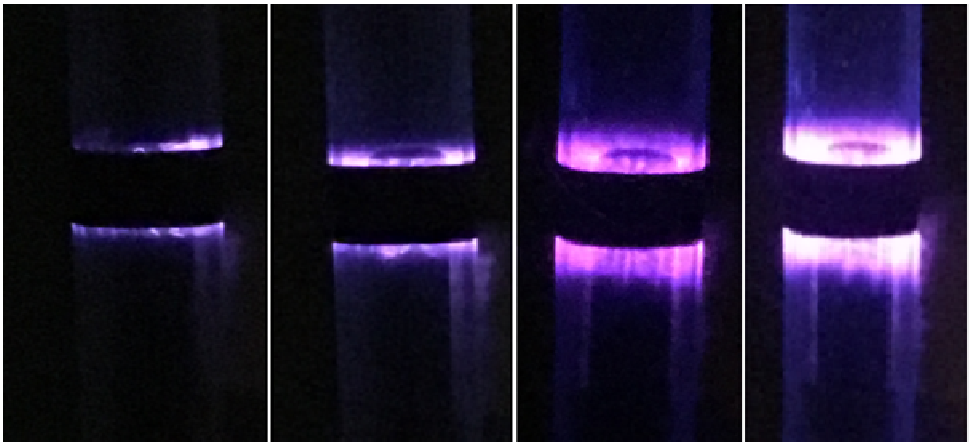

Fig. 3. Shapes of cold plasma DBD under different conditions from left to right are (U = 7 kV, I = 3.5 mA); (U= 16kV, I = 10mA); (U= 19 kV, I = 16 mA); (U = 21 kV, I = 22 mA).

Proceedings

- Take 5 µLof solution from reactor as suggested above to analyze TNP by HPLC determining retention time (tR) and peak height, as well aspeak area for the calculation of percentage (%) of TNP remaining in the samples. The main parameters of HPLC method were established as following:
- Measurement signal:λ= 360 nm
- Flow rate: 0.35 mLper minute
- Sample pump volume: 5.0µl
- Retention time (t_R), corresponding to the peak of TNP is 4.0 minutes
- Measuring unit: mg/L

The TNP concentration present in the samples, corresponding to the peak area (y), was determined by the calibration curve as shown in Fig.3.

Method of determining COD index

The COD content in the study sample was determined by HACH COD DR/890 (US) measuring device according to ISO 6060: 1989: Water quality - determining chemical oxygen demand (COD). The method was carried out according to the test kit provided with the device.

RESULTS AND DISCUSSION

Kinetics of TNP degradation

Establishment of the degradation rate of TNP versus varying initial TNP concentration: The experiments were carried out under conditions, such as, the applied power of 16 mA, the voltage of 19 kV, the circulating wastewater rate of 415 mL/min, sample pH of 3.2.The initial TNP concentrations were selected as following:91.02 mg/L, 135.3 mg/L and 210.17mg/L. The TNP degradation mean rate was determined after 30 minute-treatment by plasma DBD increases with increasing TNP concentration presented in Table 1. The initial TNP decomposition rate increases with increasing TNP concentration. However, the decomposition mean rate is not proportional to the TNP concentration. The TNP degradation mean rate was measured for 30 min reaction indicating the increase of TNP degradation rate at increasing initial TNP concentration presented in Figure 4. In Figure 4, the initial TNP decomposition rate increases with increasing initial TNP concentration, but being not proportional to the TNP concentration. Under these experimental conditions, the TNP initial concentrations less than 210.17 mg/L indicate an effective degradation. Figure 5 shows the relationship between the reciprocal rate (-1/R) versus reciprocal TN Pinitial concentration($1/C_{tnp}$) as a linear function of $-1/R = f(1/_{CTNP})$ of $y = 39.678x + 0.3009$, $R^2 = 0.9957$.

or corresponding to the degradation rate of the empirical equation as following:

$$-R=\frac{0.0252C_{TNP}}{1+0.0076C_{TNP}}$$

This expression is comparable to that in the work (19) when the author studied the degradation kinetics of p - nitrophenol by Fenton reaction.

In the case of $0.0076C_{TNP} << 1$ or $C_{TNP} << 131.579$ mg/L, the TNP degradation rate for the 30 min is proportional to the initial concentration, which corresponds to the experimental data present in Figure 5 from 0 to <135.3 mg/L.

Kinetics of TNP degradation in the air cold plasma DBD reactor: The degradation kinetics of TNP was carried out at three different initial concentrations of TNP under the experimental conditions presented in section3.1.1 above and the reaction time varying from 30 to 120 minutes. The degradation kinetics of TNP in the plasma DBD reactor obeys the pseudo – first - order rate expression (Figures 6, 7, 8)as follows:

$\ln(C_t/C_0) = -0.0269t + 0.1605$, corresponding to initial concentration of 91.02 mg/L, Figure 6.
$\ln(C_t/C_0) = -0.0197t + 0.0792$, corresponding to initial concentration of 153.3 mg/L, Figure 7.
$\ln(C_t/C_0) = -0.014t + 0.0623$, corresponding to initial concentration of 210.17 mg/, Figure 8.
From these equations above, the half time of the TNP degradation of in the plasma reactor would be calculated, as follows:

$t_{1/2\ (91.03\ mg/L)} = 31.7$ min;
$t_{1/2\ (153.3\ mg/L)} = 39.4$ min;
$t_{1/2\ (210.17\ mg/L)} = 53.9$ min;

From the obtained rate equations of the TNP degradation, the degradation reaction in the plasma DBD reactor would be simplified by the equation, obeyed the pseudo - first - order reaction kinetics:

TNP + Reactive reagents → Products

With

$$-\frac{d(TNP)}{dt} = k[TNP][reactive\,reagents]$$

Or $$-\frac{d(TNP)}{dt} = k_{app}[TNP]$$

Here $k_{app} = k$ (reactive reagents)

In this case, k_{app} depends on the ratio of the amount of reactive reagents per TNP initial concentrations. This conclusion is fitted with the obtained experimental data. The kinetic equation of TNP degradation above can be used in practice in predicting the TNP concentration left in the plasma DBD reactor at any treatment time.

Influence of applied power source on TNP degradation

The experiment was performed under the condition, C_{TNP} = 135.3 mg/L, the circulating wastewater rate of 415 mL/min and with varying power source. The experimental data are presented in Table 2.

The degradation efficiency (H%) of TNP is calculated based on the expression:

$$H = \frac{C_{TNP0} - C_{TNPt}}{C_{TNPo}} \times 100$$

The experimental data of H (%) are presented in Figure 9

Table 1. Degradation mean rate of TNP at differential concentrations after 30 minute- treated

Initial TNP conc., mg/L.	0	91.02	135.3	210.17
TNP degrad. rate, mg/L. min	0	1.35	1.71	2.01

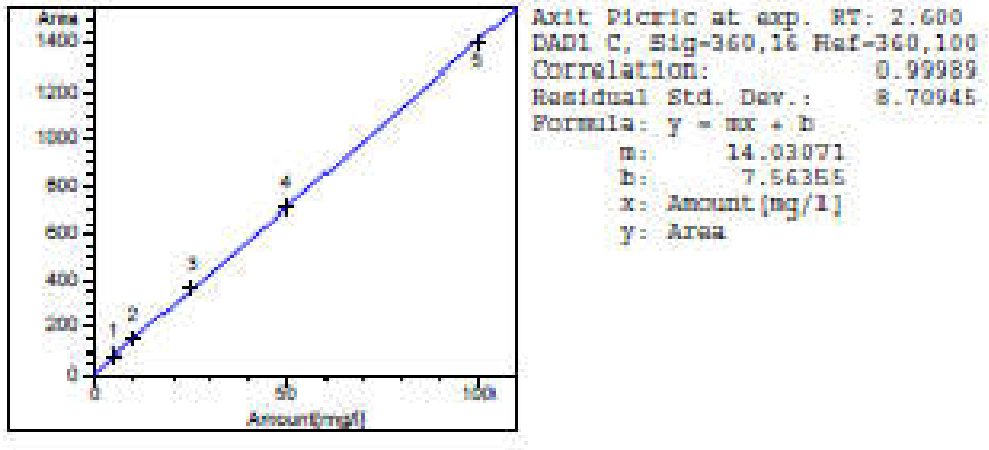

Fig.4. The calibration curve of TNP ($y = 14.03071C_{TNP} + 7.56355$)

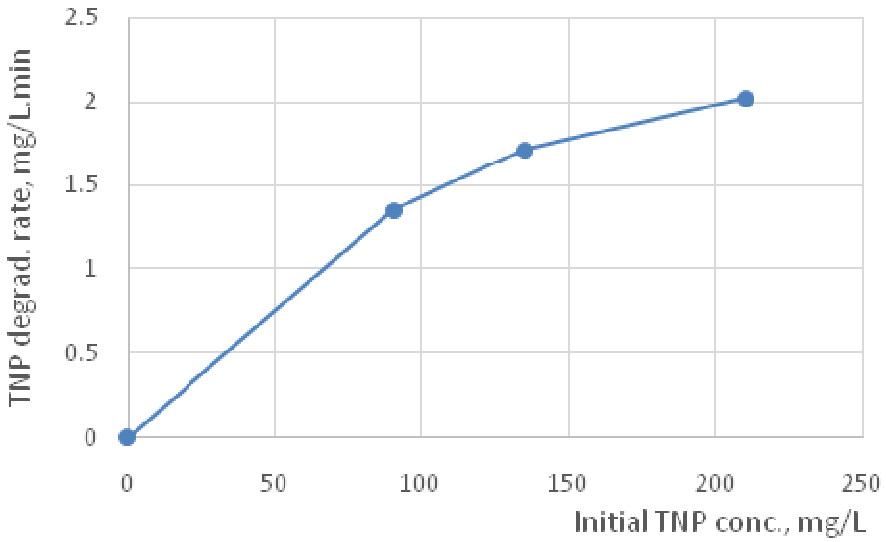

Fig.5. The TNP decomposition rate with increasing initial TNP concentration

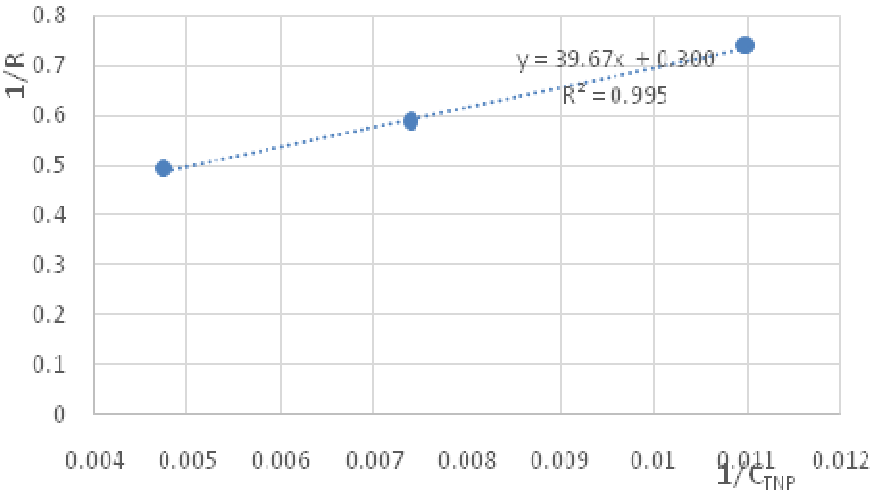

Fig. 6. The dependence of (-1/R) on ($1/C_{TNP}$)

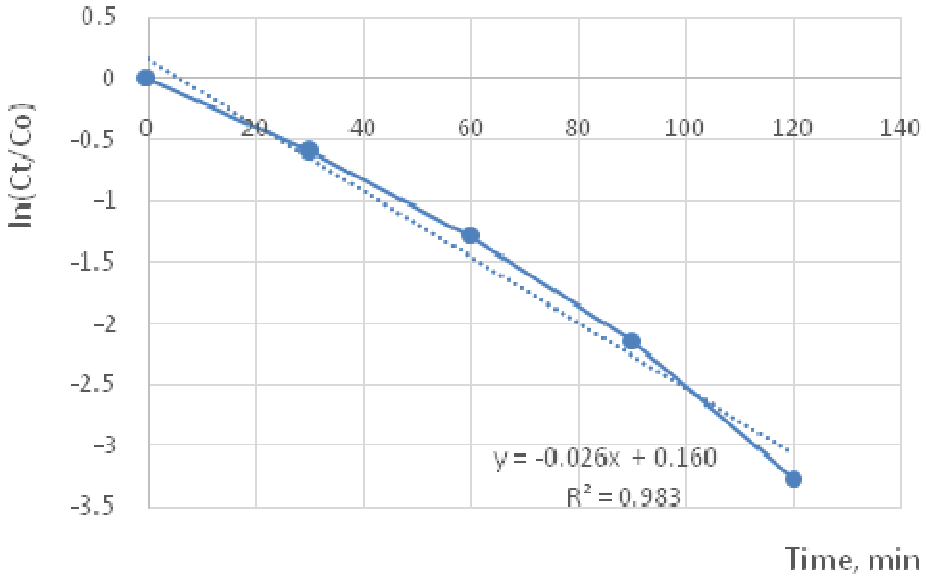

Fig. 7. Degradation kinetics of TNP at TNP concentration of 91.03 mg/L

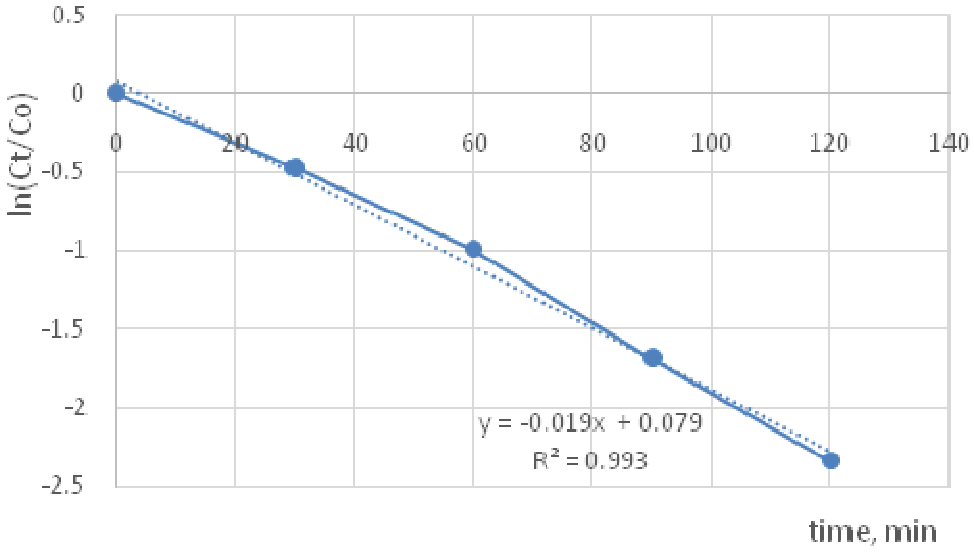

Fig. 8. Degradation kinetics of TNP at TNP concentration of 135.3 mg/L

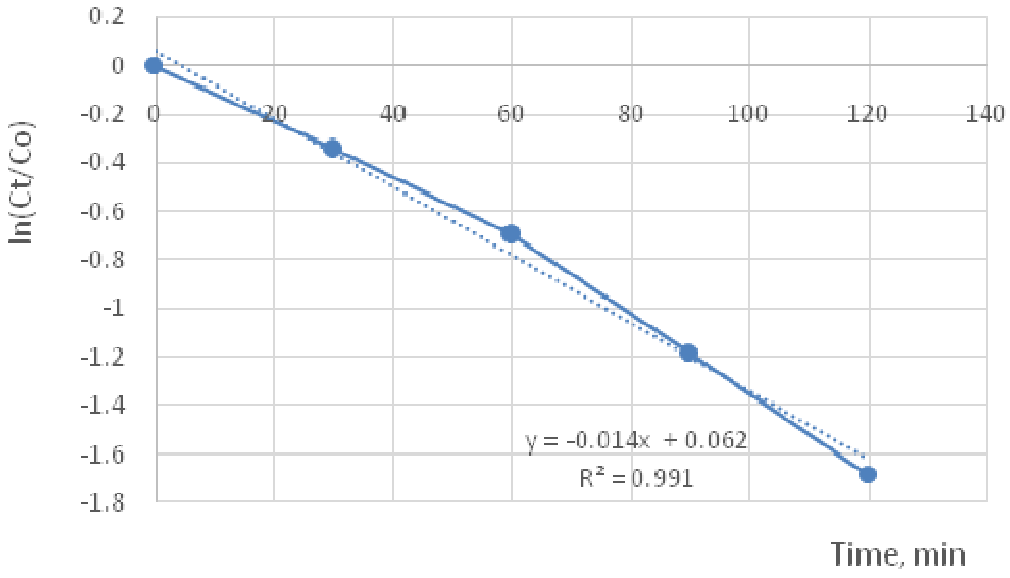

Fig. 9. Degradation kinetics of TNP at TNP concentration of 210.17 mg/L

Table 2. Influence of power source on TNP degradation, C_{TNP} = 135.30 mg/L

Time, min	I=10mA, U=16kV			I=16mA, U=19kV			I=22mA, U=21kV		
	C_t, mg/L	H, %	R,mg/L min	C_t, mg/L	H, %	R,mg/L. min	C_t, mg/L	H, %	R,mg/L. min
0	135.30			135.30			135.30		
30	109.01	19.43	0.88	84.20	37.77	1.70	41.86	69,21	3.11
60	89.20	34.07	0.66	50.19	62.90	1.13	11,65	91,54	1.01
90	68.59	49.31	0,69	25.08	81.47	0.84	2,11	98.59	0.32
120	52.82	60.96	0.53	13.05	90.36	0.40	0,70	99.63	0.05

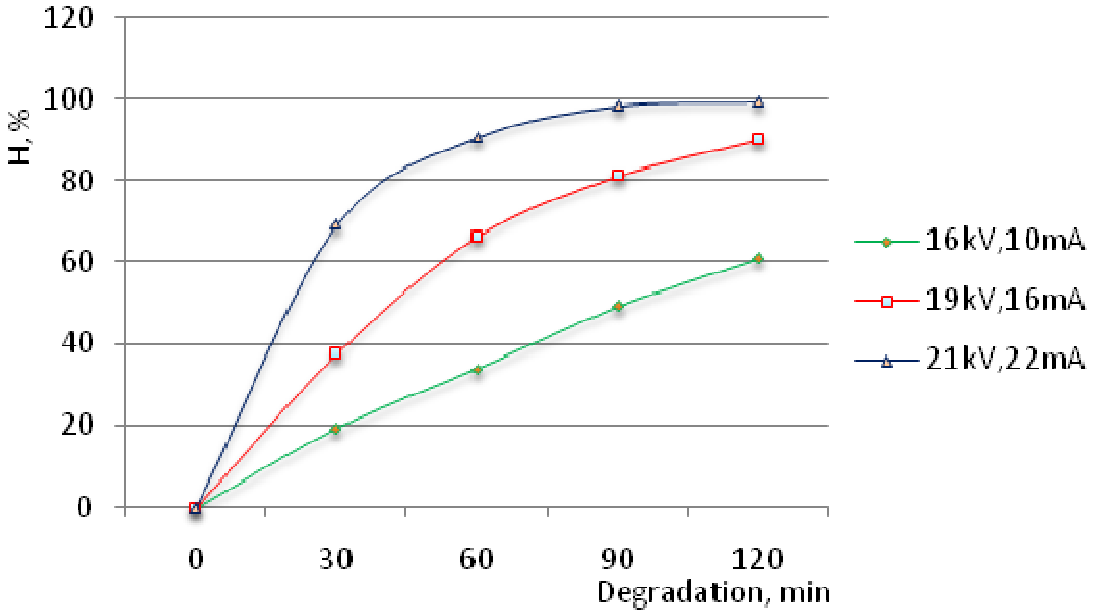

Fig.10. Degradation efficiency (H%) of TNP with varying power sources

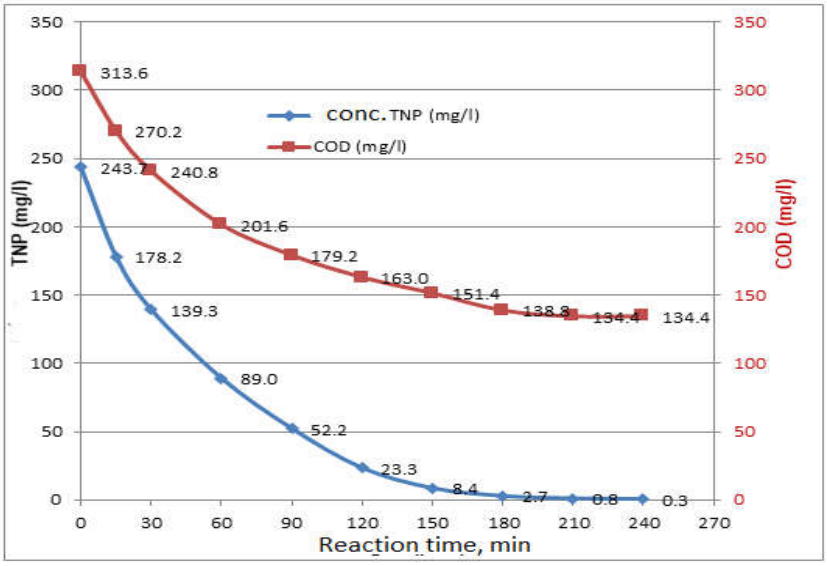

Fig.11. The decrease of TNP concentration and COD values at varying time

The experimental data from Table 2 and Figure 9 indicate that increasing the applied power sources from 16 kV to 21 kV, the TNP degradation rate, degradation efficiency increase. This is due to the fact that at higher voltage,more reactive species would be produced, increasing the degradation process.

Mineralization of TNP: The mineralization of TNP was determined based on the decrease of TNP concentration and COD values in the plasma DBD reactor under the conditions of 20 kV, I = 16 mA. The obtained experimental data are presented in Figure10.

The experimental data (Figure 10) indicate that after 240 minutes of treatment time, the TNP concentration in the sample is almost completely degraded, while 134 mg O2 COD, equivalent to 57% of COD treated,remains. This means the TNP degradation takes place in many steps, with the first step being the is transformation of TNP into other compounds.

Conclusion

An atmospheric air cold plasma dielectric barrier reactor was built used to study the TNP degradation in aqueous.

The plasma reactor consists of varying power source from 16 kV to 21 kV with two coaxial electrodes separated by a teflon dielectric barrier. The TNP degradation by this plasma reactor was carried out under different conditions. The influence of TNP initial concentration from 91.02 to 210.17 mg/L on its degradation efficiency and on the reaction time was established to find the TNP degradation kinetics. The TNP degradation procession under the experimental condition obeys pseudo - first - order reaction rate kinetics. The decrease in the TNP concentration and COD values indicated that the TNP degradation in plasma DBD reactor includes many steps. The first step of TNP degradation may be the TNP transformed into other compounds as intermediate products. The intermediate products or by-products are going to be determined and reported in other article.

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