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RESEARCH ARTICLE / **ARAŞTIRMA MAKALESİ**

INDIRECT SONICATION OF ACETIC ACID IN AQUEOUS SOLUTIONS

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ABSTRACT

Sonochemical oxidation of organic pollutants has a promising future in the area of wastewater treatment as one of the advanced oxidation techniques. In this study indirect ultrasonic degradation of acetic acid by using rectangular prism reactor was investigated. The effects of parameters such as: power of irradiation, initial concentration of acid, NaCl or oxide (zeolite) addition to solution were investigated on the extent of degradation. An ultrasonic bath was used. The results showed that degradation degree increased with decreasing power and initial concentration of pollutant. Initial degradation rate was lower in the concentration range of 0.37-0.75M for NaCl than that in the absence of salt. Initial degradation rate was the highest at a NaCl concentration of 1.5M in the range studied. However the degradation degree was always lower in the presence of NaCl than that in the absence of salt after a sonication time of one hour although degradation degree increased with increasing salt concentration.

In the present study obtained degradation degree of acetic acid was about 7 % at 84W, 300ppm and 43±3°C after a sonication time of 60 min. Under the same experimental conditions, addition of 1.5M NaCl provided 33 % increase in degradation according to that in the absence of NaCl after a sonication time of 20 min. Addition of 0.3g zeolite caused an increase in the degradation degree of acetic acid from 7 % to 10 % at 84W, 300ppm and 43±°C, providing almost 42 % increase in degradation degree of acetic acid after 60min when compared with degradation degree in the absence of zeolite.

Keywords: Acetic acid, Indirect sonication, Ultrasound, Degradation, Wastewater

SULU ÇÖZELTİLERDEKİ ASETİK ASİTİN İNDİREK SONİKASYONU

ÖZ

Organik kirleticilerin sono kimyasal oksidasyonu, atık su arıtımında gelecek vaat eden ileri oksidasyon tekniklerinden biridir. Bu çalışmada dikdörtgen prizma şeklinde reaktör kullanarak asetik asitin indirek ultrasonik bozunması incelenmiştir. Ultrasonik güç, kirleticinin başlangıç derişimi, NaCl veya oksit (zeolit) ilavesi gibi parametrelerin asetik asit bozunmasına etkileri incelenmiştir. Çalışmada ultrasonik banyo kullanılmıştır. Sonuçlar bozunmanın azalan güç ve başlangıç derişimi ile arttığını göstermiştir. 0.37-0.75M NaCl derişim aralığında başlangıç bozunma hızı tuzun olmadığı durumdan daha düşüktür. Başlangıç bozunma hızının en yüksek değeri çalışılan derişim aralığında 1.5M'lık NaCl derişiminde elde edilmiştir. Bununla beraber 1 saat'lik sonikasyon zamanı sonunda artan tuz derişimi ile degradasyon derecesi artmakla birlikte NaCl varlığındaki bozunma yüzdeleri NaCl ilavesi yapılmamış durumdan daima daha düşük olmuştur.

Çalışmada 84W, 300ppm ve 43±3°C'de 60dk sonra yaklaşık %7 bozunma elde edilmiştir. Aynı deneysel şartlarda 1.5M NaCl ilavesi 20dk'lık bir sonikasyonla bozunmada NaCl'size gore % 33'lük bir artma sağlamıştır. 0.3g zeolit ilavesi asetik asit bozunmasını 60dk'lık bir sonikasyonla, 84W, 300ppm ve 43±°C'de, % 7'den % 10'a yükselterek asetik asit bozunmasında yaklaşık %42'lık artış sağlamıştır.

Anahtar Kelimeler: Asetik asit, İndirekt sonikasyon, Ultrases, Bozunma, Atıksu

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1. INTRODUCTION

Wastewater which produced during domestic, commercial industrial activities usually requires treatment before discharge in order to protect the receiving environment. The main objective of wastewater treatment is to separate undesirable impurities and pollutants from the water.

Wastewater treatment can be achieved by physical, chemical, biological and advanced oxidation methods. Advanced oxidation processes (AOP) are defined as those techniques that involve the generation of highly reactive radical intermediates particularly the hydroxyl radical at ambient temperature. The hydroxyl (OH.) radical is an extremely strong and nonselective oxidant. The OH radical is a short lived species so it must be generated in the actual waste stream rather than supplied as a concentrated solution.

AOP are generated by a variety of methods; photolysis, O_3/UV oxidation, photocatalysis, fenton – photofenton catalytic processes, wet air oxidation and sonolysis.

Sonolysis is developed latest years. Sono-chemistry is the application of ultrasound to chemical reactions and processes. The origin of sonochemical effects in liquids is the phenomenon of acoustic cavitation. Sound is passed through a liquid as a wave consisting of alternating compression and rarefaction cycles. If the rarefaction wave has a sufficiently high negative pressure, it can overcome the intermolecular forces bonding fluid. As a result, the molecules are torn apart from each other and tiny micro bubbles are created. These micro bubbles gradually grow during compression and rarefaction cycles until they reach a critical size. Subsequent compression causes these cavities to collapse almost instantaneously with a large amount of energy and extremely high temperatures on the order of 5000 K and pressures of the order of 1000 atm are attained. Under such extreme conditions, water molecules dissociate into OH. and H. The radical species can either recombine or react with other gaseous substrates within the cavity. There are three potential sites for chemical reactions in ultrasonically irradiated liquids: A volatile substrate would be readily taken into the cavity and its main decomposition reaction pathway may be by pyrolysis within the cavitation bubble. The second region is the interfacial zone between the gas phase and the bulk solution, where large temperature and pressure gradients exist. Therefore, at this site substrate can be degraded by two reaction pathways, either by oxidation with OH. or by thermal reaction. The third possible reaction zone involves the bulk solution, where the decomposition of pollutants might occur by the reaction of ultrasonically produced bubbles of OH. diffusing into the bulk solution. Thus, the reaction zone, or degradation pathways of a particular substrate depends on its chemical nature, for example, its volatility, solubility and chemical structure, etc. Volatile organic compounds may easily undergo direct thermal reactions within the cavitation bubble; however semivolatile or non volatile solutes react at the bubble interfaces or within the bulk solution (Wu et.al, 2001; İnce et.al, 2001; Nam et.al, 2003).

There are several factors affecting the extent of ultrasonic degradation of pollutants, such as; time of irradiation, initial concentration of pollutants, intensity and frequency of irradiation, introduction of gas, temperature, solid catalyst, several salts, H_2O_2, pH, different cavitation equipments and the level of coupling fluid (Ince et.al, 2001; Petrier and Francony, 1997).

The effects of reaction parameters in the ultrasonic degradation of organic pollutants have been widely investigated in literature:

Seymour and Gupta (1997) have reported the en-hancements of reaction rates by the addition of sodium chloride salt. Using 20 kHz ultrasound, large salt induced enhancements were observed: 6-fold for chlorobenzene, 7-fold for p-ethylphenol and 3-fold for phenol oxidation.The addition of salt increased the ionic strength of the aqueous phase which drives the organic pollutants toward the bubble-bulk interface.

Sonochemical degradation of ethylbenzene in aqueous solution at 520 kHz was studied by Visccher et al. (1997). The results indicated that reaction rate increased at low initial concentrations of ethyl benze-ne. The degradation rate increased slightly as a function of time and pyrolysis was an important pathway of ethylbenzene degradation.

According to the study of Naffrechoux et al. (2000) the effects of ultrasound wave in hydrophilic chemical oxidations are mainly due to hydroxyl radical production during the cavitation induced water decomposition. The applications of ultrasound and UV irradiation (sonuv) have studied for the degradation of phenol and for the COD reduction of a municipal wastewater. The degradation of a phenol solution in the sonuv reactor was faster than with techniques such as high pressure UV lamb, 20 kHz or 200 kHz ultrasound, ozone or O_3/ 200 kHz ultrasound.

Nagata et al. (2000) have investigated sono-chemical degradation of chlorophenols in water at 200 kHz and 200 W under argon and air atmosphere. The degradation followed first order kinetics in the initial state. The rate of degradation was faster in argon than in air. In this study, the effect of Fe ions on the degradation of 3-chlorophenol was also investigated. In the presence of Fe(II) the rate increased 2.4 times at 1mM of Fe(II) concentration and 1.5 times at 2mM. The results suggested that there was an optimum Fe (II) concentration for 3-chlorophenol sonolysis with maximal efficiency and that an excessive amount of Fe (II) led to a decrease in the degradation rate due to the scavenging of OH radicals by Fe(II) ions.

Okuno et al. (2000) have studied the sonolytic degradation of hazardous organic compounds in aqueous solution. According to their study, 80-90% of the initial amount of organic compounds was degraded by 30-60 min of sonication time when the initial concentrations were 10-100 μmol/l at 200 kHz and intensity of 6 W/cm^2. They observed that the degradation rate of compound increased with increase in vapor pressure. Compounds with relatively high vapor pressures more rapidly entered the gas phase and more effectively underwent degradation.

Teo et al. (2001) have reported the degradation of p-chlorophenol using three different ultrasonic devices including probe, beaker and cup-horn system. Probe system had the highest initial rate for the ultrasonic treatment of p-chlorophenol and beaker type ultrasonic devices was suitable for the study of sonochemical mechanism. The degradation of p-chlorophenol could be affected in the presence of hydrogen peroxide and external sparking gas.

Sonochemical decomposition of dibenzothiophene in aqueous solution was investigated by Kim et al. (2001). Decomposition of dibenzothiophene followed a first order reaction kinetics. The rate constant was found to increase with increasing ultrasonic energy intensity, temperature and pH and to decrease with increasing initial concentration. The activation energy was 12.6 kJ/mol in the temperature range of 15-50 °C, suggesting a diffusion controlled reaction.

Sivakumar et al. (2002) have reported the sonochemical degradation of p-nitrophenol (p-NP). Reaction has carried out with ultrasound at three operating frequencies, 25, 40 kHz each independently and the combination of two frequencies simultaneously. The influence of various parameters including initial solution concentration, pH, and bulk solution temperature on the degradation of p-NP was studied for three frequencies and kinetic study was performed.

Although the ultrasonic degradation of several organic pollutants have been widely investigated as given above, there is so far no study on sonolytic degradation of acetic acid reported in literature. Acetic acid is an important industrial chemical and it is used in the manufacture of acedic anhydride, cellulose acetate, vinyl acetate, plastics, pharmaceuticals, dyes, insecticides, photographic chemicals, vitamins, antibiotics and cosmetics. It is also used in textile printing, as a preservative in foods and as a solvent for gums and resins. The emission sources of acetic acid are industries that produce acetic acid or use it as an intermediate in the production of other products and consumer products that contain acetic acid such as disinfectants, herbicides, detergents, pharmaceutical preparations. Concentrated acetic acid is corrosive and has to be handled with great care; it can cause skin burns, permanent eye damage and irritation to the mucous membranes. Acetic acid is a very resistant chemical to oxidation because of the difficulty to oxidize the methyl group in α-position of the carboxylic group. That is why in the oxidation of phenol, the route conducting to acetic acid is a dead-end. (http://en.wikipedia.org/wiki/Acetic_acid, 2005, http://www.npi.gov.au/database/substance-info/profiles/2.html, 2005).

The aim of the present study is to investigate the sonolytic degradation of acetic acid in aqueous solution to have information for the effects of ultrasonic power, initial concentration of acetic acid, addition of NaCl or natural zeolite on the extent of degradation of acetic acid.

2. EXPERIMENTAL

An ultrasonic bath (235mm in length, 135mm in width, 100mm in depth with a capacity of 2.5l) was used for sonication of acetic acid solutions. The ultrasonic bath operated at 40 kHz and ultrasound power range of 70-140 W. Operating temperature of the bath is regulated between 20-80 °C. Sonication time is adjusted to a desired time in the range of 1-60 minutes. Ultrasonic bath was filled with 1700ml of water as coupling fluid. Liquid soap (0.17% in weight) was added to water to improve cavitation. Aqueous solution with a known concentration of acetic acid (Merck, extra pure) was prepared and reactor was filled with 200ml of the solution, then reactor was inserted into ultrasonic bath. Reactor was hanged with braces. It was kept 2 cm above the bottom of the ultrasonic bath in all the runs. Ultra pure water was used to prepare aqueous solutions.

A reactor in the shape of rectangular prism was made from optical glass with dimensions: height 90mm, width 70mm, length 135 mm and wall thickness 2.5 mm. Aluminum foil was used to close the reactor.

An experiment took 1 hour and all the experiments repeated at least 4, mostly 7 times. The samples were withdrawn from the reaction mixture periodically. The unreacted amount of acetic acid was determined by titration with 10^{-3} M NaOH solution, using 2 % (in weight) of phenol phtalein as indicator. Dilute NaOH solution was used so that readings of titration were in the range of 10-14 ml with the least count of burette as 0,01 ml.

The percentage of degradation of pollutant was calculated by equation 1.

$$\text{Degradation, \%} = \left(\frac{Co - C}{Co}\right) * 100 \qquad \textbf{(1)}$$

where C_0 initial concentration, C concentration measured at corresponding time.

3. RESULTS AND DISCUSSION

3.1 Effect of Ultrasonic Power

To investigate the power effect on indirect sonication of acetic acid, three different powers, 70, 84 and 98 W were supplied and tested. Figure 1 shows the results. On the degradation degree versus time plots the points represents the average of 4-7 independent runs in the text. Experiments were done with 300 ppm, 200 ml aqueous solutions of acetic acid at 43±3 °C. Due to the dissipation of ultrasonic energy in the liquid, which is related to the structure of pollutant, temperature of the reaction mixture may increase. In this study, it is observed that temperature increases +6 °C during a sonication time of one hour regardless of initial temperature. So, mean temperature with a deviation ±3 °C was used in the study.

According to Figure 1, degradation degree of acetic acid decreases with increasing intensity of irradiation. At power of 70 W, initial degradation rate is the highest. Ultrasound intensity is defined by power/vibration area. When power is increased at the same reactor area, ultrasound intensity increases. The magnitude of the pressure pulse generated during the collapse of a single cavity is found to be inversely proportional to the operating intensity. With an increase in the operating intensity cavitation events become less violent resulting in lower extent of degradation (Gogate et al., 2003).

At higher intensities of irradiation, there exist a large number of a gas bubbles or cavities in the solution which scatter the sound waves to the walls of the vessel or back to the transducer. Thus a lesser level of energy is used although the vessel is exposed to higher intensities. Also it may happen that due to the very high number of cavities per unit volume of the cavitating medium, there is a likely coalescence of the cavities, resulting in the formation of a large cavity which collapses less violently. Similar results have been reported in literature in ultrasonic decomposition of formic acid (Gogate et al., 2003).

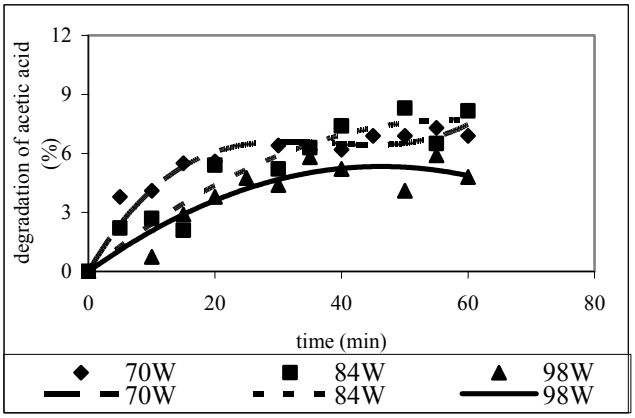

Figure 1 Effect of ultrasonic power on the degradation of acetic acid (temperature=43±3°C, initial concentration=300ppm)

In the present study, ultrasonic device provides indirect sonication, which will inevitably cause energy loss with a limited quantity of energy transmitted into the reaction vessel. So, it should be kept in mind that the powers shown on the figures are supplied ones, not actual powers of ultrasonic energy transmitting into the reaction mixture.

3.2 Effect of Initial Concentration

Figure 2 shows the effect of initial concentration on acetic acid degradation. Degradation rate decreases with increasing initial concentration in the concentration range studied. The highest extent of degradation is obtained with 300ppm of acetic acid at a power of 84W.

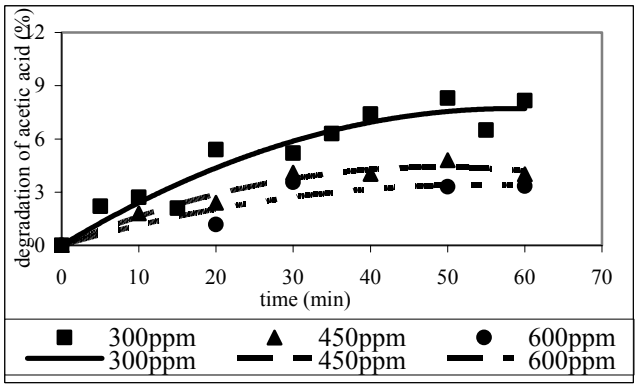

Figure 2 Effect of initial concentration on the degradation of acetic acid (power= 84W, temperature= 43±3°C)

Jiang et al (2002), Drijvers and coworkers (1999) explain the decrease in degradation with increasing initial concentration by the influence of specific heat ratio, γ, (Cp/Cv), of the gas mixture in the cavitation bubbles ,where γ is the ratio of constant pressure and constant volume heat capacities. The proportionality between the concentration of a volatile compound in the bubble and its concentration in the solution will influence the ultrasonic reaction rate, as the temperature of bubble collapse is dependent upon the specific heat ratio(γ) of the gas mixture through equation 2.

$$T_{max} = T_o(\gamma-1)P_{max}/P_{min} \quad \textbf{(2)}$$

where T_{max} is final temperature, P_{max} is liquid pressure of collapse, P_{min} is the minimum pressure in vapour phase and T_o is the ambient temperature of fluid.

A linear relationship between the specific heat ratio(γ) of the gas mixture and the volatile compound concentration (C_i) in the liquid phase can be assumed;

$$\gamma = \gamma_o - K\,C_i \quad \textbf{(3)}$$

K is proportionality constant.

As a result, the specific heat ratio of gas mixture decreases with increasing volatile concentration

resulting in a lower temperature and pressure within the cavitation bubbles and thus a decrease in rate of sonochemical degradation of volatile compound is observed.

Similar results have been reported in literature for the effect of initial concentration on the extent of sonolytic degradation of organic pollutants:

Vischer et al. (1997) studied the degradation of ethylbenzene in aqueous solution at 520 kHz. Sonication was performed at initial concentrations of 1 and 0.5 M ethylbenzene. The reaction rate was higher at low initial concentration. In another study (Wang et al., 2003) the sonochemical degradation in aqueous solution of methyl violet was studied. Degradation of methyl violet was studied for different initial concentrations at 20°C and 20 kHz. It was observed that as initial concentration increased degradation rate decreased. Sivakumar et al. (2001) have studied the degradation of p-nitrophenol. According to their study, rate constant decreases with increasing initial concentration. In another study (Gogate et al., 2004), destruction of phenol using sonochemical reactor was investigated. The percentage of phenol degradation is inversely proportional to the initial concentration at 22.7 kHz and 240 W in the ultrasonic horn.

3.3 Effect of NaCl Addition

The aim of NaCl addition to solution is to push pollutant molecules from the bulk aqueous phase toward the interface. Because degradation takes place in the bubble-bulk interface area. Pollutants present in that region undergo degradation due to exposure to free radicals and high temperature and pressure (Seymour and Gupta, 1997).

Figure 3 shows the effect of NaCl addition on the extent of sonolytic degradation of acetic acid. For addition of NaCl in the range of 0.37- 0.75M to solution, initial degradation rate of acetic acid is lower than that without NaCl in the reactor. At 1.5M NaCl, initial rate is the highest and after 30min, degradation level remains almost constant, it shows that there is no longer bubble occupation for oxidation at the corresponding pollutant concentration. However, the degradation degree is always lower in the presence of NaCl than that in the absence of salt after a sonication time of one hour, although degradation degree increases with increasing salt concentration.

In literature, Seymour and Gupta (1997) investigated the effect of NaCl addition to pollutant solution on the degradation of chlorobenzene, p-ethyl phenol or phenol with direct sonication at 20 kHz. According to their study, degradation rate increases with increasing NaCl concentration. In another study (Gogate et al., 2004), similar results have been obtained for phenol degradation using NaCl.

Addition of NaCl increases the hydrophilicity of the aqueous phase which is an unfavorable environment for the organic pollutants; hence the pollutants are driven towards the organic phase. In addition to this partitioning enhancement, salt decreases the vapor pressure and increases the surface tension. These two factors help in promoting a more violent collapse of the bubble, then the extent of degradation of pollutant increases.

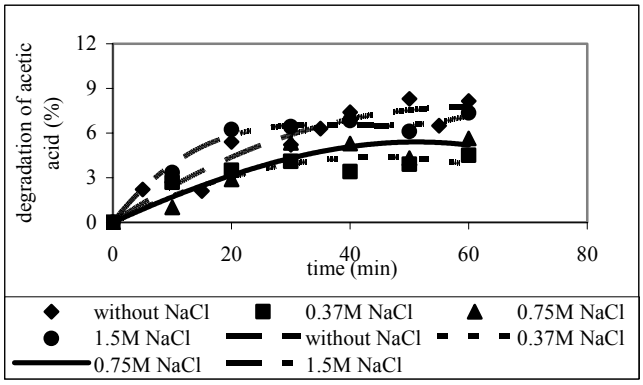

Figure 3 Effect of NaCl addition on degradation of acetic acid (power=84W, temperature=43±3°C, initial concentration=300ppm)

3.4 Effect of Zeolite Addition

The presence of solid particles affects the cavitational activity in two different and opposing ways, firstly it intensifies the process by providing additional nuclei due to the discontinuities in liquid medium and hence the number of cavitation events may increase but at the same time due to the scattering of incident sound waves the net energy dissipation into the system decreases (Gogate et.al, 2004).

In this study, the effect of natural zeolite rich in clinoptilolite from Bigadiç region on sonolytic degradation of acetic acid was investigated.

All the experiments were done with various amounts of zeolite at a temperature of 43±3 °C and at a power of 84 W and 200 ml of 300 ppm aqueous solution of acetic acid.

To investigate the effect of zeolite alone, experiments were done by addition of zeolite to solution without ultrasound. It was observed that no degradation of acetic acid happened in the absence of ultrasound.

Figure 4 shows the effect of natural zeolite amount added to solution. As seen from Figure 4, degradation degree increases with increasing zeolite amount. Initial rate of degradation is the same for all zeolite amounts used.

In literature similar and opposite results have been reported for different oxides. In one study (Gogate et al., 2004) the effect of TiO_2 was investigated for phenol degradation at 22.7 kHz and 240W for

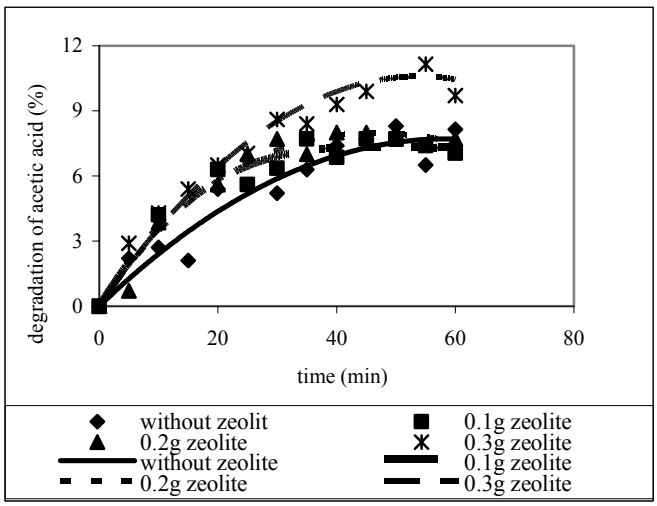

Figure 4 Effect of zeolite addition on the degradation of acetic acid (power=84W, temperature=43±3°C, initial concentration=300ppm)

ultrasonic horn. The presence of TiO_2 particles at 300ppm concentration was found to be detrimental resulting in lower extent of degradation as compared with that observed in the absence of solid particles in spite of the positive contribution of TiO_2 particles in the case of flow cell. The scattering of the incident sound waves appears to be predominant factor as compared with the enhancement in the number of cavitation events for ultrasonic horn. As the concentration of solid particles increases the negative effect of sound scattering becomes dominating resulting in lowering the extent of degradation. Drijvers and coworkers (1999) obtained better H_2O_2 degradation rates in the presence of CuO than those for Al_2O_3, ZnO, Ni_2O_3 .

4. CONCLUSIONS

In this study indirect ultrasonic degradation of acetic acid by using rectangular prism reactor was investigated. Reaction parameters such as: power, initial concentration of pollutant, NaCl or zeolite addition were investigated. An ultrasonic bath was used for sonication. This study showed that degradation degree increases with decreasing power and initial concentration, and with increasing NaCl concentration. Degradation degree increases with increasing zeolite concentration, as well. However, the obtained degradation degree for acetic acid is low, at about 10%. Sonolysis of acetic acid combined with other advanced oxidation processes, such as, ozonation, UV, photolysis, wet air oxidation may give better results than the sonolysis method alone.

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