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Kinetic Study of the Ultrasound Effect on Acid Brown 83 Dye Degradation by Hydrogen Peroxide Oxidation Processes

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Abstract: The effect of ultrasound on the degradation of the dye Acid Brown 83 by seven different degradation methods (blank test using only ultrasound, hydrogen peroxide in a neutral medium, hydrogen peroxide in a sulfuric acid medium and hydrogen peroxide in a sulfuric acid medium in the presence of Fe(II), both without and with ultrasonic irradiation) is studied in this paper. The effectiveness of these methods is compared by analyzing the degradation percentages of the dye and its initial degradation rate. The application of ultrasound leads to a significant increase in the efficiency of any of the degradation method studied. Kinetic study of Acid Brown 83 degradation by the above-mentioned methods is carried out by using four kinetic models (first order, second order, Behnjady and pseudo-first order). The pseudo-first order model is the one that best fits the experimental data in all the used degradation methods. Although when the degradation is performed in the presence of Fe(II), the Behnjady model presents correlation coefficients slightly higher than those of the pseudo-first order, the maximum experimental conversions obtained fit much better in all cases to the pseudo first order model.

Keywords: dye degradation; ultrasound effect; hydrogen peroxide; Fe(II); kinetics



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1. Introduction

The rapid increase in water consumption, a consequence of population growth, urbanization and industrial, agricultural and livestock developments, constitutes one of the most important challenges currently existing worldwide [1]. The continuous and growing contamination of water resources by effluents derived from human activity negatively affects both the quality of ecosystems and the health of the different existing forms of life.

Among the different categories of environmental pollutants, dyes stand out due to their relevance. They are non-biodegradable soluble organic compounds present in large quantities in wastewater from different industrial activities such as textile, tanning, printing, pulp and paper or paint. Currently, there are more than 100,000 commercial colorants with an estimated production of 106 tons per year, with more than 15% of the total dyes produced annually reaching industrial wastewater [2,3].

The presence of dyes in water resources produces a change in the color of the water, reducing, or totally preventing, the penetration of light through it, which leads to a reduction in the rate of photosynthesis and the level of dissolved oxygen. In addition, dyes are toxic, mutagenic and carcinogenic agents [4,5], persisting as environmental pollutants and crossing entire food chains providing biomagnifications [6].

The effects of dyes on human health range from dermatitis and central nervous system disorders [7] to those related to the inactivation of some enzymatic activities, as a consequence of the substitution of enzymatic cofactors [8], its genotoxicity being the greatest potential long-term danger to human health [9].

Azo dyes are the main chemical class of dye used in industrial activities. They are characterized by the presence of one or more azo groups ($-N=N-$), in association with

one or more aromatic rings that usually contain one or more functional groups such as amino ($-\text{NH}_2$), chlorine ($-\text{Cl}$), hydroxyl ($-\text{OH}$), methyl ($-\text{CH}_3$), nitro ($-\text{NO}_2$), sulfonic acid ($-\text{SO}_3\text{H}$) or sulfonic acid sodium salt ($-\text{SO}_3\text{Na}$) [10], making them all very environmentally detrimental due to their high persistence in the aquatic environment.

Therefore, different methods of effluents treatment have been described in an attempt to minimize the problems resulted from this contamination. They include biological methods (algal [11], activated sludge [12], bacterial [13], fungal [14] and enzymes [15]), adsorption [16–18], coagulation/flocculation [19], electrochemical oxidation [20] and membrane processes [21–23]. Combinations of the mentioned methods are often used [24,25].

Advanced oxidation processes (AOP) constitute a new pollutant removal technology, widely used in recent years due to its versatility and wide spectrum of applicability, that have emerged as a group of very efficient methods for water treatment and wastewater [26]. They include all catalytic and non-catalytic processes that take advantage of the high oxidizing capacity of the hydroxyl radical ($\bullet\text{OH}$), a radical that is generated in situ and that reacts rapidly with most organic compounds [27,28]. Hydroxyl radicals are optimal within the group of powerful oxidants because they are the second known species with the highest oxidizing power, after fluorine and they meet a series of requirements such as not generating additional residues, non-toxicity, very short life, non-corrosivity of equipment and generally produced in assemblies that are easy to handle [29].

AOPs are usually classified into homogeneous and heterogeneous processes, distinguishing between those that operate with an external input of energy (radiant energy, ultrasonic energy, electrical energy) and those that do not. Some of the most extensively investigated AOPs are Fenton (homogeneous/heterogeneous), photocatalysis, ozonation and $\text{H}_2\text{O}_2/\text{UV}$ [29].

A relatively new AOP method is sonolysis (or sonication), in which the wastewater is subjected to ultrasound (US) irradiation. Ultrasound produces cavitation, which essentially is nucleation, growth and collapse of tiny gas/vapor bubbles, creating immense energy concentration on extremely small spatial and temporal scale (temperatures and pressures of 5200 K and 250 MPa, respectively, can be reached in a single bubble collapse [30]). The gas and solvent (usually water) molecules present in the bubble at collapse undergo thermal dissociation to generate numerous chemical species, some of them radical species, which are released into the bulk liquid medium, where they can induce oxidation reactions [30].

It is also of interest the knowledge of the kinetics of the degradation of the dyes by means of the different oxidative processes. The studies described in the literature in this regard show that the degradation of dyes can follow the kinetics of first order [31], second order [32], Behnajady model [33] and pseudo first order [34].

In this paper we study the effect of ultrasound on the degradation of Acid Brown 83 (AB83) by different methods. AB83 is an azo dye with a molecular weight of 496.39 g/mol and formula $\text{C}_{18}\text{H}_{13}\text{N}_6\text{NaO}_8\text{S}$ which contains in its molecule, in addition to two azo groups, three benzene rings with a sulfonic acid sodium salt group, an amine group, three hydroxyl groups and a nitro group (Figure 1).

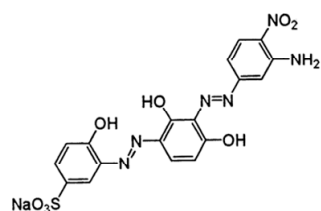


Figure 1. Chemical structure of Acid Brown 83.

The degradations of AB83 under different conditions (hydrogen peroxide in a neutral medium, hydrogen peroxide in a sulfuric acid medium and hydrogen peroxide in a sulfuric acid medium in the presence of Fe^{2+}), both without and with ultrasonic irradiation, were

investigated. The percentages of degradation of AB83 and its initial rates of degradation were determined.

Kinetics of AB 83 degradation, at times ranging from 0 to 60 min, by the above-mentioned methods were analyzed by means of four kinetic models (first order, second order, Behnajady and pseudo-first order) in order to determine the one that best describes the degradation process.

2. Materials and Methods

2.1. Chemicals

ALFA Industries (Guajarat, India) provided Acid Brown 83 azo dye. Hydrogen peroxide (33% *w/v*), sulfuric acid and iron(II) sulfate heptahydrate were purchased from Panreac (Castelar del Vallés, Spain).

2.2. Experimental Procedure

Solutions of 125 mg/L of Acid Brown 83 and 0.5 N of sulfuric acid were used in all tests, except in a blank test, in which a solution of AB83 in pure water and ultrasound was used. In the tests using hydrogen peroxide, a 1/10 AB83/H₂O₂ molar ratio was used, while in the tests using Fe(II) and hydrogen peroxide a 1/10/1 AB83/H₂O₂/Fe(II) molar ratio was used.

The experimental tests using ultrasound were carried out with a Labsonic M equipment (Sartorius SA, Madrid, Spain) with 10 mm diameter titanium probe, sound classification density of 130 W/cm², at 30 KHz and 150 µm. The samples (40 mL) were sonicated in a 50 mL glass reactor, whose jacket was connected to a cooling thermostat to maintain the temperature (25 ± 3 °C). A schematic diagram of the experimental setup is shown in Figure 2.

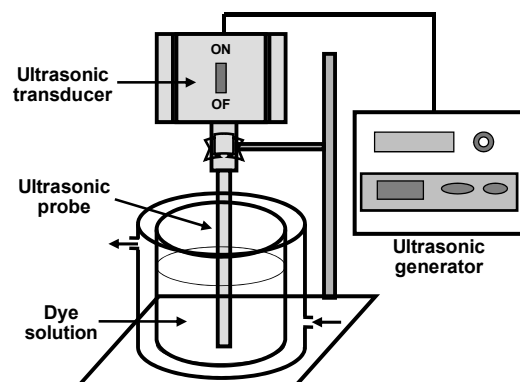


Figure 2. Schematic representation of the ultrasonic experimental setup.

Samples were periodically taken from the reactor and the AB83 concentration was determined by UV/V spectrophotometry, at 448 nm, using an Agilent UV 8453 spectrophotometer (Agilent Technologies, Madrid, Spain). The concentration of AB83 in an unknown sample was determined from the calibration curve (concentration range: 5–100 mg/L; $R^2 = 0.9996$). The results obtained showed a maximum deviation of 3%.

The efficiency of the AB83 degradation by the different oxidation processes was calculated, as degradation percentage (DG), by equation:

$$DG = 100 \cdot X = 100 \cdot \frac{C_0 - C_t}{C_0}, \quad (1)$$

where C_0 and C_t are the initial and time t concentrations of AB83 in the solution (mg/L) and X is the conversion in the degradation process: $(C_0 - C_t)/C_0$.

The kinetics of Acid Brown 83 degradation by the seven processes described above was analyzed by means of four kinetic models by determining both the correlation coefficients

of the experimental data fits to the equations of the models and the characteristic constants of each model.

3. Results and Discussion

3.1. Effect of Ultrasound on the Degradation of Acid Brown 83 by Different Methods

The decrease, with time, of Acid Brown 83 concentration in the solution, for the different degradation processes studied, is shown in Figure 3, while Figure 4 shows the values of the degradation percentages at 60 min and the initial degradation rates (first 6 min) obtained with those degradation processes (US = ultrasonic treatment).

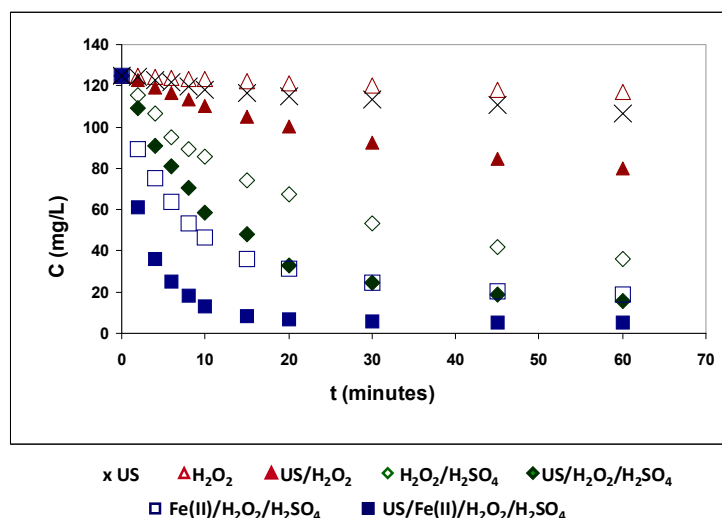


Figure 3. Variation of Acid Brown 83 concentration with time in different degradation methods.

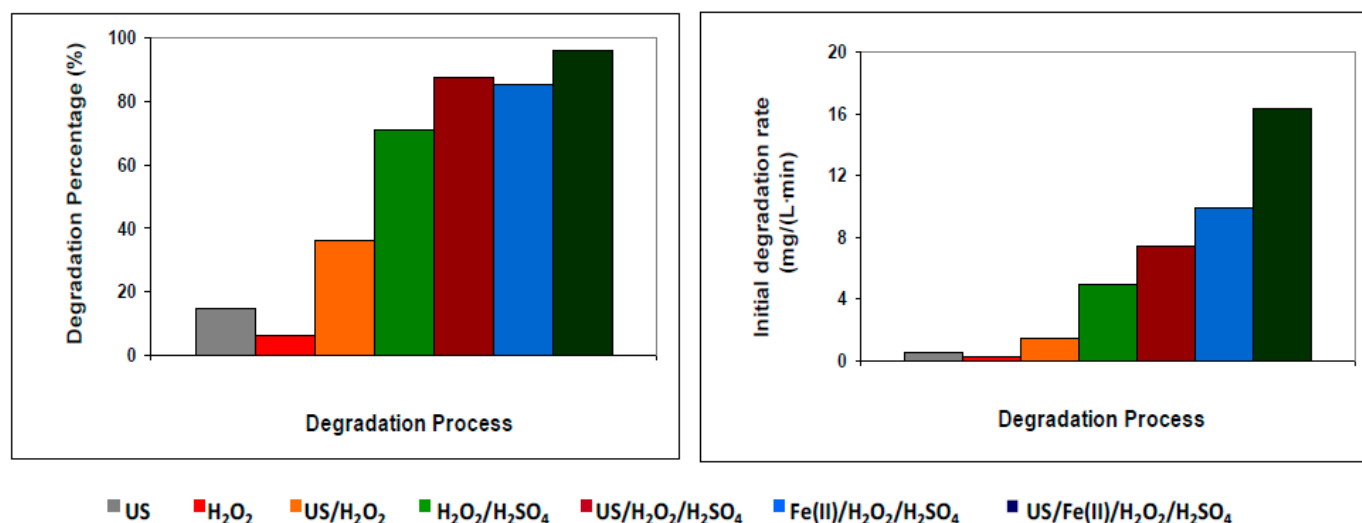
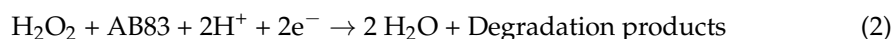


Figure 4. Degradation percentages and initial degradation rates of Acid Brown 83 by different degradation methods.

It can be observed that the effectiveness of the Acid Brown 83 degradation process increases in the form $H_2O_2 < US < H_2O_2/H_2SO_4 < Fe(II)/H_2O_2/H_2SO_4$, leading the application of ultrasound to a significant increase in the efficiency of the degradation in all cases.

Regarding the initial degradation rates, the observed variation is the same, being remarkable the fact that the $US/H_2O_2/H_2SO_4$ process has a higher initial rate than $Fe(II)/H_2O_2/H_2SO_4$, but achieves a somewhat lower degradation percentage.

Hydrogen peroxide in a neutral medium (distilled water) is the degradation method that leads to the lowest results, being the following the reaction that describes its degradation action [35]:



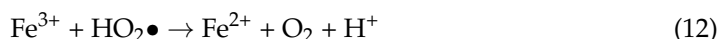
This reaction explains the higher effectivity of hydrogen peroxide in an acid medium (presence of sulfuric acid) due to the increase in its oxidizing power as acidity increases, according to the Nerst equation.

The application of ultrasound to an Acid Brown 83 solution in distilled water also leads to poor degradation effect, but slightly higher than that of hydrogen peroxide in neutral media. When a water solution of AB 83 is sonicated, the hydroxyl radical ($\bullet\text{OH}$) is formed, which reacts with the AB83 causing its degradation.

The reactions involved can be described as follows [36,37]:



In absence of ultrasound, the oxidation process leading to better degradation efficacy is that using $\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}_2\text{SO}_4$. This process is also based on the high oxidant power of hydroxyl radical and can be described by the following reactions [38]:



When an aqueous solution containing hydrogen peroxide is irradiated with ultrasound, the energy released in the cavitation process produces the homolytic breakdown of H_2O_2 , generating two $\text{HO}\bullet$ radicals per molecule of H_2O_2 and other propagation reactions [39]:



If Fe^{2+} is also present in the medium, the reactions Equations (9)–(11) takes also place [40].

In all these, the final degradation reaction of Acid Brown 83 involves, as indicated above, its reaction with the hydroxyl radical (Equation (8)).

The generation of a greater amount of hydroxyl radicals in the case of the ultrasound-treated solutions explains the increase in both the initial degradation rate and the degradation percentage.

However, it is worth highlighting the fact that the Fe(II)/H₂O₂/H₂SO₄ process leads to a lower percentage of degradation than US/H₂O₂/H₂SO₄ process, despite having a higher initial degradation rate. This can be explained by admitting that the reactions associated to the initial degradation stages in the Fe(II)/H₂O₂/H₂SO₄ process, mainly the reaction between Fe(II) and H₂O₂, are faster than those in the US/H₂O₂/H₂SO₄ process, while the reactions associated to later stages of the process are faster in this last process than in the first one. Similar results have been described by different authors [41–43].

3.2. Kinetics Study of the AB83 Degradation Process

The kinetics of Acid Brown 83 degradation by the seven processes described above were analyzed by means of four kinetic models (first order, second order, Behnajady and pseudo-first order) to determine the one that best describes the AB83 degradation process.

The first order model is expressed by the equation:

$$\frac{dC}{dt} = k_1 \cdot C \quad (19)$$

where k_1 (1/min) is the rate constant of the first order degradation process and C is Acid Brown concentration. The integrated form of this expression is linear:

$$\ln(1 - X) = k_1 \cdot t \quad (20)$$

where X is the conversion in the degradation process (see Equation (1)). The value of the rate constant k_1 is calculated from the slope of the plot $\ln(1 - X)$ versus t .

The second order model is described by the equation:

$$\frac{dC}{dt} = k_2 \cdot C^2 \quad (21)$$

where k_2 (L·mg^{−1}·min^{−1}) is the rate constant of the second order degradation process.

The integrated form of the equation is:

$$\frac{X}{1 - X} = C_0 \cdot k_2 \cdot t \quad (22)$$

The value of k_2 is calculated from the slope of the plot of $X/(1 - X)$ versus t .

Behnajady model [33] is described by the equation:

$$\frac{t}{X} = a + b \cdot t \quad (23)$$

where a and b are two characteristic constants of the model related to the initial degradation rate ($1/a$) and the maximum degradation capacity ($1/b$), respectively.

The plot of t/X versus t allows the values of a and b to be determined from the intercept and the slope, respectively.

The pseudo-first order model can be formulated as follows [34]:

$$\frac{dC}{dt} = k_{ps1} \cdot (C - C_{lim}) \quad (24)$$

where k_{ps1} is the rate constant of the pseudo-first order kinetic model and C_{lim} is the minimum AB83 concentration that can be reached in the degradation process.

The integrated form of this equation, expressed in function of the extent of degradation process, can be written in the form:

$$X = X_{max} \cdot (1 - \exp(-k_{ps1} \cdot t)) \quad (25)$$

where X_{max} is the maximum conversion of the degradation process.

Linear regression analyzes based on the first order, second order and Behnjady kinetic models has been performed to obtain the values of the kinetic constants of the first two models (k_1 and k_2) and the constants a and b of the last one. The software Curve Expert 1.4 has been used to fit the experimental data corresponding to pseudo-first order model and to calculate $X_{\max}$ and k_{ps1} .

Figure 5 shows the kinetics plots of the four studied kinetic models for the seven degradation methods employed in this study, while the values of the characteristic constants of those kinetic models and the values of the dimensionless parameter coefficient of determination, R^2 , (statistical measure of how close the data are to the fitted regression line) obtained for each of them are included in Table 1. In addition, the relationship between the maximum experimental conversions obtained at 60 min and the maximum conversions obtained through the Behnjady model ($1/b$) and the pseudo-first order model ($X_{\max}$) is analyzed (Figure 6).

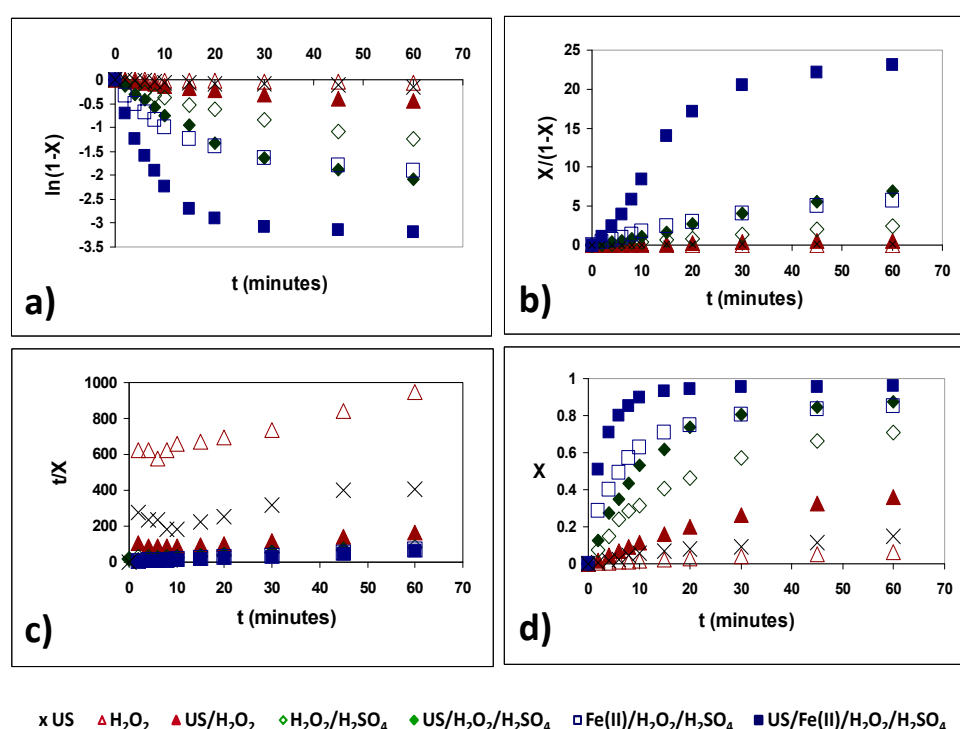


Figure 5. Kinetics plots of the four studied kinetic models (a) first order; (b) second order; (c) Behnjady model; (d) pseudo-first order model.

Table 1. Parameters of the studied kinetic models for the degradation of Acid Brown 83 by seven processes ((1: US; 2: H₂O₂; 3: US/ H₂O₂; 4: H₂O₂/H₂SO₄; 5: US/H₂O₂/H₂SO₄; 6: Fe(II)/H₂O₂/H₂SO₄; 7: US/ H₂O₂/H₂SO₄).

Process	First Order		Second Order		Behnjady Model			Pseudo-First Order		
	R^2	k_1 (min ⁻¹)	R^2	k_2 (L·mg ⁻¹ ·min ⁻¹)	R^2	a (min)	B	R^2	k_{ps1} (min ⁻¹)	$X_{\max}$
1	0.9367	0.0025	0.9462	0.00002	0.6333	163.22	4.6305	0.9991	0.02712	0.18503
2	0.981	0.0011	0.9832	0.00001	0.4960	478.97	8.6474	0.9998	0.01792	0.09640
3	0.9647	0.0077	0.9826	0.00008	0.6225	67.11	1.6769	0.9998	0.03044	0.43215
4	0.9565	0.0208	0.9968	0.00033	0.9218	17.773	1.1386	0.9974	0.05692	0.71953
5	0.9057	0.0353	0.9897	0.00098	0.9684	8.8707	0.9888	0.9989	0.08817	0.87110
6	0.8089	0.0289	0.948	0.00078	0.995	4.2467	1.1092	0.9938	0.15682	0.81581
7	0.6197	0.0438	0.8309	0.00334	0.9994	1.0462	1.0222	0.9976	0.34859	0.94094

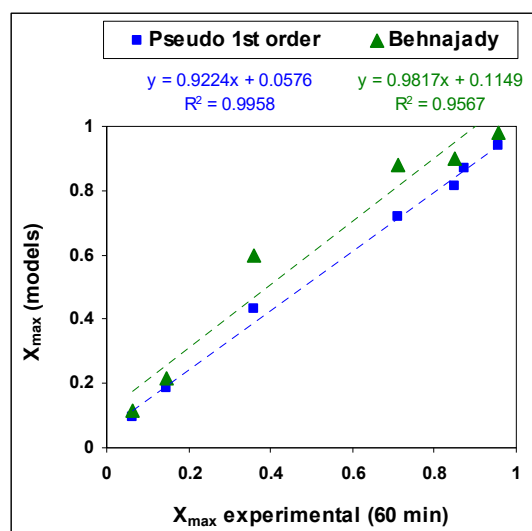


Figure 6. Relationship between experimental maximum conversions (obtained at sixty minutes) and models (pseudo-first order and Behnajady) maximum conversions.

As it is evident from the global analysis of the results shown in Table 1, the degradation of Acid Brown 83 by any of the methods studied is best fitted by the pseudo-first order kinetic model, since the determination coefficients of this model are, in all cases, greater than 0.993.

By contrast, the determination coefficients corresponding to the first order, second order and Behnajady models are, in general, lower than 0.99. Only when the degradation of AB83 is performed in the presence of Fe(II), the Behnajady model presents correlation coefficients (0.995 and 0.9994) slightly higher than those of the pseudo-first order (0.9938 and 0.9976).

Figure 6 shows that the maximum experimental conversions fit much better to those obtained by pseudo-first order model than to those obtained by the Behnajady model, as can be deduced from the higher determination coefficient of the regression line.

Therefore, according to all the results reported in this paper, it can be stated that the kinetic model that best describes the degradation of Acid Brown 83 dye by any of the degradation methods used is the pseudo-first order model.

4. Conclusions

The effect of ultrasound treatment on the degradation of the dye Acid Brown 83 by hydrogen peroxide under different experimental conditions was investigated by determining the percentages of degradation and the initial degradation rates. The obtained results revealed that ultrasound increases the effectiveness of the degradation process in all cases. The best results are obtained for the sample, conducted in sulfuric acid medium, in the presence of Fe(II). Degradation percentage 95% and initial degradation rate $17 \text{ mg} \cdot \text{L}^{-1} \cdot \text{min}^{-1}$ were achieved. Kinetic study of the results reveals that the pseudo-first order model best describes the degradation of Acid Brown 83 by oxidation process provided under described conditions.

Author Contributions: G.L., B.M. and L.M. conceived, designed and performed the experiments. G.L., B.M., L.M., M.I.S. and M.A.G. analyzed the data and contributed to the interpretation of the results. G.L. and B.M. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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

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