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PHOTOCHEMICAL DEGRADATION OF NITROBENZENE BY $S_2O_8^{2-}$ IONS AND UV RADIATION

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Key words: water remediation, homogeneous catalysis, oxidizing reagent, persulfate, advanced oxidation

ABSTRACT

In this work nitrobenzene (NB) degradation was studied through an advanced oxidation process (AOP) by using a photochemical reactor and two kinds of oxidizing reagent: potassium persulfate ($K_2S_2O_8$) and sodium persulfate ($Na_2S_2O_8$), to promote generation of HO^\bullet radicals. It was determined the effect of different parameters on nitrobenzene degradation, such as: wavelength of UV lamps, concentration of oxidizing reagent, type of oxidizing reagent and number of UV lamps in the reactor. By using UV lamps with radiation wavelength of 254 nm, NB degradation reached almost 100 % meanwhile, irradiating at 350 nm resulted in < 35 % degradation. For experiments using UV lamps of 254 nm, the reaction rate increased with the radiant energy, being higher by using four UV lamps than two UV lamps for a reaction time of 60 min. The specific energy consumption for two UV lamps showed the lowest value being of 2626.0 KW.h/Kg of NB degraded.

Palabras clave: remediación de agua, catálisis homogénea, agente oxidante, persulfato, oxidación avanzada

RESUMEN

En este trabajo se estudió la degradación de nitrobenzeno (NB) mediante un proceso de oxidación avanzada empleando un reactor fotoquímico y dos agentes oxidantes: persulfato de potasio ($K_2S_2O_8$) y persulfato de sodio ($Na_2S_2O_8$), con el objetivo de generar radicales HO^\bullet . Se determinó el efecto de diferentes parámetros en la degradación de nitrobenzeno, tales como: longitud de onda de la lámparas UV, concentración del agente oxidante, tipo de agente oxidante y número de lámparas en el reactor. Con la lámpara de 254 nm se alcanzó una remoción de NB cercana al 100 % mientras que con la lámpara de 350 nm se obtuvo un porcentaje de degradación < 35 %. Para el caso de las lámparas de 254 nm se encontró que la tasa de degradación se incrementa con la energía radiante, es decir, aumenta al emplear cuatro lámparas respecto de utilizar dos para un tiempo de reacción de 60 min. Al considerar el consumo energético específico, con el arreglo de dos lámparas UV se tiene el menor valor, siendo de 2626.0 KW/h/kg de NB degradado.

INTRODUCTION

The quality, quantity and supply of water have become a major problem for the world's population. Water is contaminated due to the presence of waste and pollutants that have an undesirable effect on its quality, damaging ecosystems and detrimental for human health. Most organic pollutants are products of anthropogenic origin. These compounds possess a particular combination of physical and chemical properties such that, once released into the environment, remain intact for long periods resisting photolytic, chemical and biological degradation (Buccini 2003, Gerrity *et al.* 2010). In addition, some important episodes of diseases transmitted by contaminated water include the cholera outbreak occurred in Peru in 1991, the cryptosporidiosis occurred in 1993 in the city of Milwaukee (Wisconsin, USA) or the induced gastroenteritis, caused by *Campylobacter* sp., in the year 2000 in Walkerton Ontario, Canada (Richardson 2003, Girones 2006). These cases have served to establish the need for effective treatments for water and how relevant the problem of water pollution to the human health is.

Nitrobenzene (NB) is an aromatic organic compound, which is toxic, easily absorbed through the skin and by inhalation. The most important toxic effect of NB is its ability to cause methemoglobinemia. Some common products contain NB at trace levels and it is widely used in the production of aniline and lubricating oils. Other uses of NB include the manufacturing of paints, medicines, pesticides, synthetic rubber (Zhao *et al.* 2015) and so on.

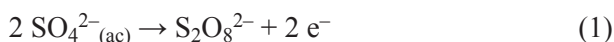
Many research groups have been proposed several treatment methodologies to carry out remediation process in waste water, such as biological, physical, chemical, electrochemical, etc. Some disadvantages of the biological treatment of water contaminated with NB are that its oxidation can produce some more toxic by-products, such as picolinic acid or/and aniline (Nishino and Spain 1993, Lin *et al.* 2013) affecting the adaptation of microorganisms used for degradation of NB and other aromatic compounds (Nishino 1995, Nielsen and Christensen 2004).

Currently, purified water treatment procedures such as coagulation, flocculation, activated charcoal and membrane systems do not take into consideration the composition and properties of organic compounds, dyes, pesticides and heavy metals, which remain in the water after treatment (Navalón 2010, Oller *et al.* 2011). Conventional oxidation processes use oxidizing agents such as ozone (2.08 V vs SHE), hypochlorite (1.57 V vs SHE), permanganate

(1.70 vs SHE), hydrogen peroxide (1.76 V vs SHE) and some combinations of these. In these processes, the organic pollutant is sometimes transformed by the action of the oxidant to harmless products such as CO₂ and H₂O. Oxidizing agents mentioned above may be used to remove both inorganic contaminants and toxic organic compounds, such as pesticides, hydrocarbons, toxins, etc. (Von 2007, Zou *et al.* 2007). Chlorine is an oxidizing agent widely used in water treatment, its main disadvantage is the formation of by-products when reacts with organic compounds.

The peroxydisulfate ion or persulfate (S₂O₈²⁻), have the structure O₃S-O-O-SO₃²⁻, the oxygen atoms are projected toward an approximately tetrahedral structure with respect to each sulfur atom. The oxidation caused by this chemical species is characterized by a slow forward that becomes faster as the temperature increases or a process of catalysts appears. The silver ion or the UV radiation are commonly used to accelerate the kinetic reaction of this oxidizing species (Espinoza 1995, Félix-Navarro *et al.* 2007).

Persulfate/sulfate redox pair (Eq. 1) is known as a strong oxidizing agent (2.07 V vs SHE) and it is useful in aqueous solutions.



The peroxodisulfate ion is able, by itself, to oxidize many pollutants, because of its oxidation potential which is related to its capability to react with organic compounds leading to mineralization.

Under photolytic conditions (UV radiation) or by heating, persulfate ions generate SO₄^{•-} radicals and they can increase the rate of a photocatalytic reaction in presence of semiconductors and even in absence of dissolved oxygen in aqueous media (Shukla *et al.* 2010).

Persulfate/sulfate radical redox pair (Eq. 2) results in a stronger oxidizer (2.6 V vs NHE) than the redox pair in Eq. 1 (Yao *et al.* 2015)



When the sulfate radical generated from the ion peroxydisulfate and UV radiation is combined with oxygen or hydrogen peroxide, a higher proportion of HO[•] radicals are produced and they promote the oxidation and degradation of organic matter in the solution without hazardous by-products (Grätzel *et al.* 1990, González and Braun 1996, Bolton 1999).

Many organic pollutants are highly resistant to conventional chemical and biological treatments. Therefore, it is necessary to find new technologically

viable alternatives for their treatment. Advanced oxidation processes (AOP) are a relatively new concept in treatment for drinking water and wastewater. These processes allow the elimination of organic and inorganic compounds.

A widely accepted definition for the advanced oxidation processes states that they can be considered as physicochemical proceedings capable of producing highly reactive species, and which have a high efficiency in organic matter oxidation, converting it in some cases and under suitable conditions to H_2O , CO_2 and innocuous mineral salts (Augugliaro *et al.* 2006). These processes involve the generation and use of powerful transient species, mainly the hydroxyl radical (HO^\bullet), primarily responsible for the oxidation of compounds or organic matter present in a solution.

Transient species are nonselective oxidizing agents, once generated, attack quickly different chemical compounds. Depending on the nature of the organic species, the hydroxyl radical can take a hydrogen atom from the compound (this occurs in alcohols and alkanes) or can be added to the pollutant structure as in the case of olefins and aromatics.

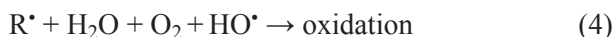
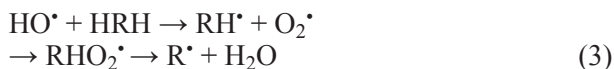
Another definition points to the AOP, as processes in which the oxidative capacity of the species is modified to perform the oxidation-reduction reactions faster or complete (Augugliaro *et al.* 2006).

AOP are important emerging technologies based on physicochemical processes which are able to produce changes in the chemical structure of pollutants (Legrini *et al.* 1993, Helz *et al.* 1994, Huang *et al.* 2002). AOP are defined as processes involving the generation and use of highly reactive transient species. The HO^\bullet radical can be generated by a photochemical via (including sunlight) or by other forms of energy, and has a high efficiency for the oxidation of organic matter (Antonopoulou *et al.* 2014).

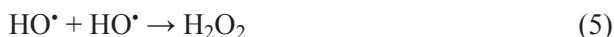
The hydroxyl radicals (HO^\bullet) take part in the reaction under different mechanisms (Legrini *et al.* 1993):

- Hydrogen abstraction related to the interaction of the hydroxyl radicals (HO^\bullet) with organic compounds (HRH), yielding the radical (RH^\bullet) when hy-

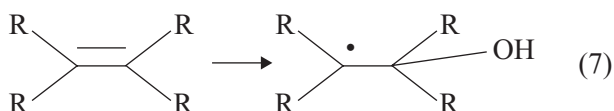
drogen is removed. This radical reacts rapidly with dissolved oxygen to form an organic peroxiradical (RHO_2^\bullet), highly unstable. This leads to thermal oxidation reactions that release the radical R^\bullet (Eq. 3), which reacts with oxygen and HO^\bullet free radicals in solution, oxidizing the organic radical (Eq. 4):



- The radical-radical reaction, produced by high concentration of hydroxyl radicals in solution, yielding to H_2O_2 (Eq. 5). If an excess of H_2O_2 is used, radicals HO^\bullet produce the radical HO_2^\bullet (Eq. 6), which is less reactive and its contribution to the oxidation of organic matter in solution depends on the redox potential of both, the organic species and the kinetic parameters. Their presence can be controlled by adjusting the pH of the solution:



- Electrophilic addition, this consists in the addition of the HO^\bullet radicals into organic molecules with π bonds, releasing the organic radical (Eq. 7):



- Electron transfer reaction, consisting in the reduction of hydroxyl radical to hydroxide ion by the organic substrate (Eq. 8):



NB degradation promoted by hydroxyl radicals includes several reactions and by-products, according to **figure 1** (Li *et al.* 2006).

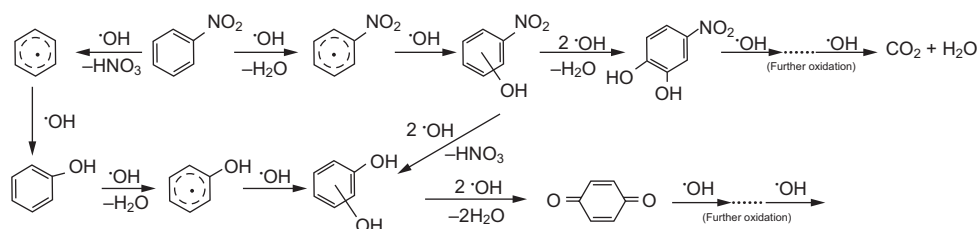
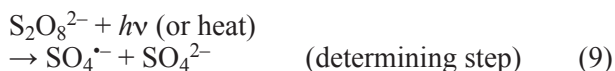


Fig. 1. Degradation pathways of nitrobenzene (NB) by HO^\bullet radicals induced by UV radiation

Some AOP are: ozonation, photocatalysis, the Fenton process, ultrasound, wet oxidation and others. Nearly all of them can be promoted by the action of UV light (Gogate and Pandit 2004). Photochemical processes involve UV radiation, their kinetic reaction can be increased by adding hydrogen peroxide and/or ozone, metal salts or semiconductors. The UV photons are able to initiate a variety of physical, chemical and biological processes. Generally the use of the radiation may lead to the degradation of organic pollutants by two major processes (Legrini *et al.* 1993): excitation of the substrate and its subsequent decomposition, and generation of the oxidizing species, that will react with the substrate.

Light-mediated AOP are not suitable for processing mixtures of high absorptivity or with high content of suspended solids, since the quantum efficiency decreases due to light loss by scattering and/or absorption competition.

Under UV radiation or heat, the decomposition of persulfate ion is its dissociation to sulfate radical (Eq. 9). This dissociation is the rate determining step of the process and is immediately followed by an attack on the reducing agent (Eq. 10):



The rate of decomposition of persulfate in aqueous solutions increases with the concentration of oxidizable species. This is because it can induce the decomposition of persulfate from the produced radicals by disintegrating the oxidizing species, and from the hydroxyl radicals produced by the action of persulfate on water. This can be schematized by a mechanism of radical-chain decomposition (Berlin 1986) (Eq. 11-Eq. 21):



These chemical species are known as good oxidizing agents, however, the hydroxyl radical HO^{\bullet} has been the most studied.

In this study, the degradation of NB in aqueous solutions using a photochemical reactor in the presence of $\text{Na}_2\text{S}_2\text{O}_8$ and $\text{K}_2\text{S}_2\text{O}_8$ as radical precursors was evaluated. Two different persulfate sources were studied to determine their efficiency in the degradation of NB. Additionally, the effect of radiation with different UV lamps, and the number of UV lamps were analyzed on the kinetic degradation of NB.

METHODS AND MATERIALS

Materials and equipment

All reagents were American Chemical Society (ACS) grade. NB (Fermont 99.99 %) was chosen as pollutant model, sodium persulfate (Sigma-Aldrich 99 %) and potassium persulfate (Sigma-Aldrich 99 %) were studied as different sources of oxidizing species. In all experiments, deionized water was used (Barnstead Sybron System). The following equipment was used for experimental development: A spectrophotometer (Hach, DR/4000U) to determine the quantity of NB in the solution, and a recirculating water bath (Fisher Scientific, Isotemp 3016) to maintain constant the temperature of the solution controlled.

For the development of the experiments a photochemical reactor was constructed to study the degradation of NB. The reactor was equipped with UV lamps. Different emission wavelengths of UV lamps were studied, the dimensions of the UV lamps were 45.7 cm of length and 2.5 cm of diameter. The specifications are: white lamp, power 15 W, model F15T8/BL, emission peak at 350 nm (long wave ultraviolet A, UVA) and transparent lamp, power 15 W, model F15T8/TL and emission peak at 254 nm (short wave ultraviolet B, UVC). These types of lamps are commercially available and they were selected to study the generation of HO^{\bullet} radicals by two different energy radiation. Also, an electric pump

brand TAAP CORP, Model 6-700, tension 12 V was used. The photochemical reactor details are shown in the results section.

Experimental

Nitrobenzene determination by ultraviolet-visible spectrophotometry

To determine the concentration of NB during the experiments, a UV-visible spectrophotometer HACH was used. Nitrobenzene presents an absorption band at 267 nm and it can be determined with no further sample treatment. During the experiments, samples of 3 mL were taken at different time intervals and then analyzed in the spectrophotometer (Priya and Madras 2006).

Effect of the concentration and type of oxidizing agent (sodium or potassium persulfate) on the degradation of NB by UV radiation

The experiments to evaluate the effect of the concentration of the oxidizing agent on the degradation of NB, were conducted at constant temperature (25 °C) and with an initial NB concentration of 20 ppm. The sample of the 0 min reaction was taken before adding the oxidizer and was analyzed later. Immediately, after the zero sample was taken, the sodium or potassium persulfate was added. For experiments different concentrations of $\text{Na}_2\text{S}_2\text{O}_8$ or $\text{K}_2\text{S}_2\text{O}_8$ (5, 10, 15 and 20 mM) were used in order to evaluate the degradation of the NB in aqueous solution.

Effect of UV lamps wavelength on the degradation of NB by UV radiation

The aim of these experiments was to determine the effect of radiation energy on the photochemical

oxidation reaction to produce sulfate radicals according to reaction 8, by breaking the persulfate ion. In these experiments, the same conditions were maintained for volume of solution, temperature, reaction time etc. and the varied parameters were the number and type of lamps used in the photochemical reactor by changing the radiation sources. This is intended to get comparative elements between the percentage of NB removal and energy consumption by the photochemical reactor. To observe the effect of the amount of radiation in the photochemical degradation of NB, the number of lamps used in the photochemical reactor was two and four for each type of lamp (254 nm y 350 nm).

The specific energy consumption (SEC) was estimated for each arrangement without considering the type of oxidizing agent, but the degradation percentage. For this calculation the following expression was used:

$$\text{SEC} = L \cdot \text{NP} \cdot t / m$$

L = number of lamps, NP = nominal power of lamps (KW), t = time for maximum percentage of degradation (h) and m = mass of NB degraded (kg), calculated from initial concentration of NB and concentration of NB at “ t ”.

RESULTS AND DISCUSSION

Figure 2 shows a diagram and a photograph of the photochemical reactor. Since the experiments were carried out with back circulating, a storage tank of 6.5 L of capacity was built. The electrical pump has

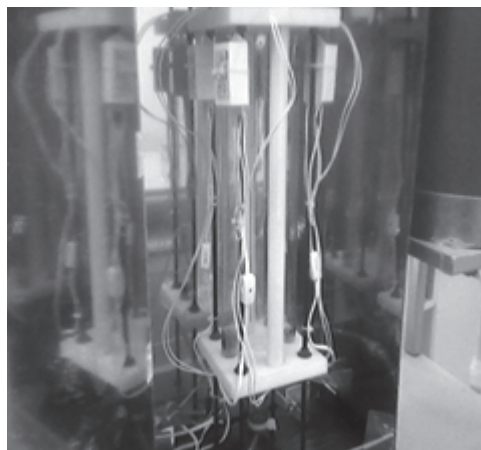
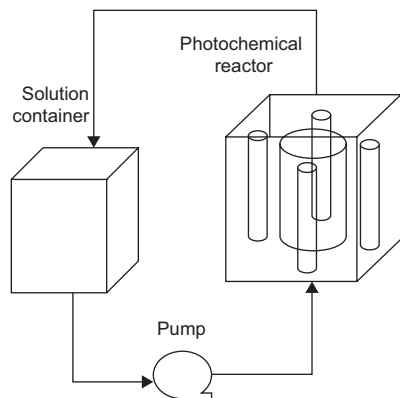


Fig. 2. (a) Scheme of the flow diagram for the photochemical reactor built and (b) photograph of the reactor inside of the container chamber to avoid radiation leakage

a flow capacity of 1 L/min. The center of the reactor was a quartz tube of 3 cm in diameter and 50.8 cm in length, with a volume of 0.323 L. The residence time of the solution inside the quartz tube was 19 seconds. To avoid the risk of exposure to UV radiation emitted by the reactor, this was placed inside a chamber of acrylic coated with a film of UV radiation control.

As it was explained in the experimental section, the experiments considered four factors: the wavelength emission of the UV lamp, the number of UV lamps, the kind of oxidizing agent and the concentration of the oxidizing agent (**Table I**).

TABLE I. PARAMETERS EVALUATED DURING THE EXPERIMENTS OF NITROBENZENE DEGRADATION

Lamp type	Lamps number	Oxidizing reagent	Concentration of oxidizing reagent (mM)
254 nm	2, 4	K ₂ S ₂ O ₈ , Na ₂ S ₂ O ₈	5, 10, 15, 20
350 nm	2, 4	K ₂ S ₂ O ₈ , Na ₂ S ₂ O ₈	5, 10, 15, 20

Effect of the concentration and type of oxidizing agent (sodium or potassium persulfate) on the degradation of NB by UV radiation

The concentrations for both oxidizing agents were 5, 10, 15 and 20 mM for both lamps of different wavelength emission and under the radiation of two and four lamps (**Table II**).

Figure 3 shows the results for the type and concentration effect on the degradation of NB, respectively. Results showed correspond to the experiment with four lamps of 254 nm, under this condition the highest NB degradation was obtained at 60 min of reaction. **Figure 3** also shows that an increasing concentration of the oxidizing agent promotes a higher reaction rate for the first 50 min, but at the end of each experiment (120 min), for both oxidizing agents, the concentration of 5 mM promotes higher degradation of NB. This phenomenon can be explained by two major reasons:

- At a higher concentration of oxidant, it is possible that other side reactions are favored, as the sulfate radical formation from persulfate ion involves reactions where other species are generated (Huang *et al.* 2002), that do not promote the degradation of NB.
- More persulfate ions mean an increment on the ionic strength of the solution, Huang *et al.* (2002) reported an inversely relationship of ionic strength with kinetic rate constant.

A chemical equilibrium finally takes place within the system. Moreover, the residence time of the solution favors this fact, since the radicals' lifetime is very short. Although the radical species formed have not enough time, they can react with persulfate ions,

TABLE II. DEGRADATION OF NITROBENZENE (NB), 20 PPM BY K/NA PERSULFATE UNDER DIFFERENT EXPERIMENTAL CONDITIONS

Lamps arrangement	Lamp wavelength	Oxidizing reagent concentration mM	NB removal (%)	
			K ₂ S ₂ O ₈	Na ₂ S ₂ O ₈
2 Lamps	350 nm	5	30.08	24.91
		10	27.68	25.78
		15	22.84	24.49
		20	24.09	26.74
	254 nm	5	95.40	96.67
		10	92.35	93.66
		15	88.62	87.67
		20	85.20	86.20
4 Lamps	350 nm	5	29.13	30.79
		10	26.69	25.40
		15	26.96	29.37
		20	26.95	26.26
	254 nm	5	96.62	96.84
		10	93.23	92.94
		15	89.47	89.09
		20	85.49	85.53

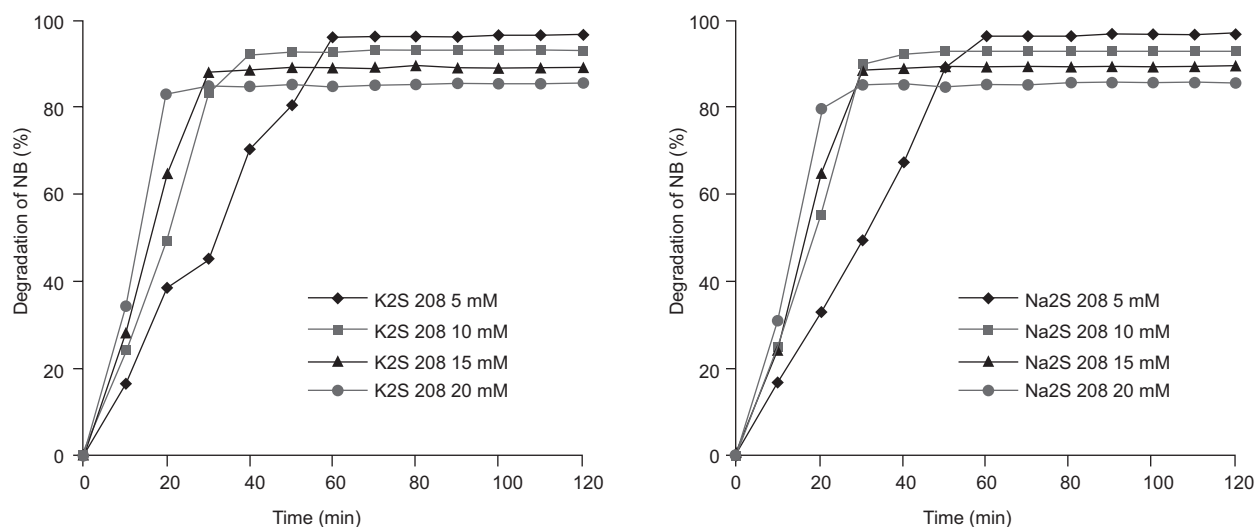


Fig. 3. Degradation of nitrobenzene (NB) at different concentrations of (a) $K_2S_2O_8$ and (b) $Na_2S_2O_8$. Conditions: $[NB]_0 = 20$ ppm, $T = 25$ °C, volumetric flow = 1 L/min and 4 UV lamps of 254 nm

water molecules, etc. The main reaction of the radical species is with NB.

For experiments using four UV lamps (254 nm), the type of oxidizing agent shows no significant effect on NB degradation, since $K_2S_2O_8$ and $Na_2S_2O_8$ reaches similar degradation of NB and the same trend on the concentrations of oxidizing species was observed.

Effect of UV lamps wavelength on the degradation of NB by UV radiation

From the two types of lamps employed, it was observed that transparent lamps (emission wavelength = 254 nm) promoted a higher percentage of NB degradation (**Fig. 4**), reaching up to 96.2 % removal for $K_2S_2O_8$ and up to 96.8 % for $Na_2S_2O_8$. While for white lamps (emission wavelength = 350 nm), the removal percentages were very low with 29.2 % for $K_2S_2O_8$ and 30.8 % for $Na_2S_2O_8$, which indicates that the oxidation process is affected by the type of incident radiant energy in the system, affecting the development of the photochemical reaction rate associated with the capacity to promote formation of sulfate radical from persulfate ions.

Effect of the number of lamps on the degradation of NB

The effect of the amount of radiation in the photochemical reactor is a dependent variable of number of lamps, time of process and type of radiation. For the arrangement of two and four lamps of 254 nm, with $K_2S_2O_8$ (**Fig. 5a**), it was observed that after 100 min of reaction, there was no significant difference in the

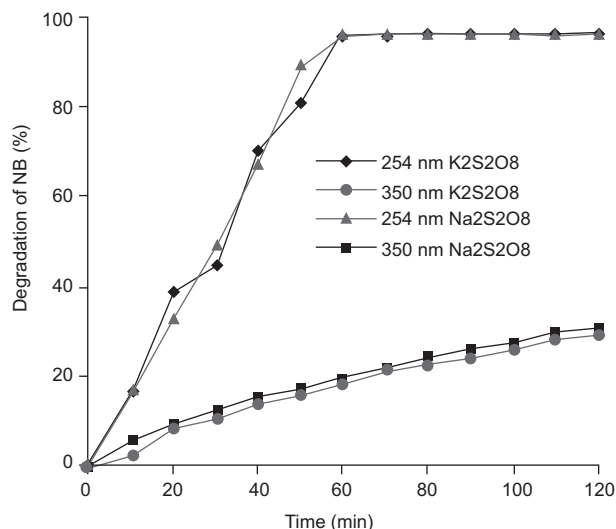


Fig. 4. Effect of type of radiation on the degradation of nitrobenzene. Oxidizing agent concentration = 5.0 mM and 4 UV lamps. Conditions: $[NB]_0 = 20$ ppm, $T = 25$ °C and volumetric flow = 1 L/min

percentage of removal achieved. However, when using four lamps the maximum degradation is achieved at 60 min of reaction, which means that the quantity of radiation increases the reaction rate, but not the final removal of NB.

For the arrangement of two and four lamps of 350 nm, with $K_2S_2O_8$ (**Fig. 5a**), percentages of degradation are similar for the 120 min that each experiment lasted, achieving a minimum increase in the percentage of degradation of NB with two lamps,

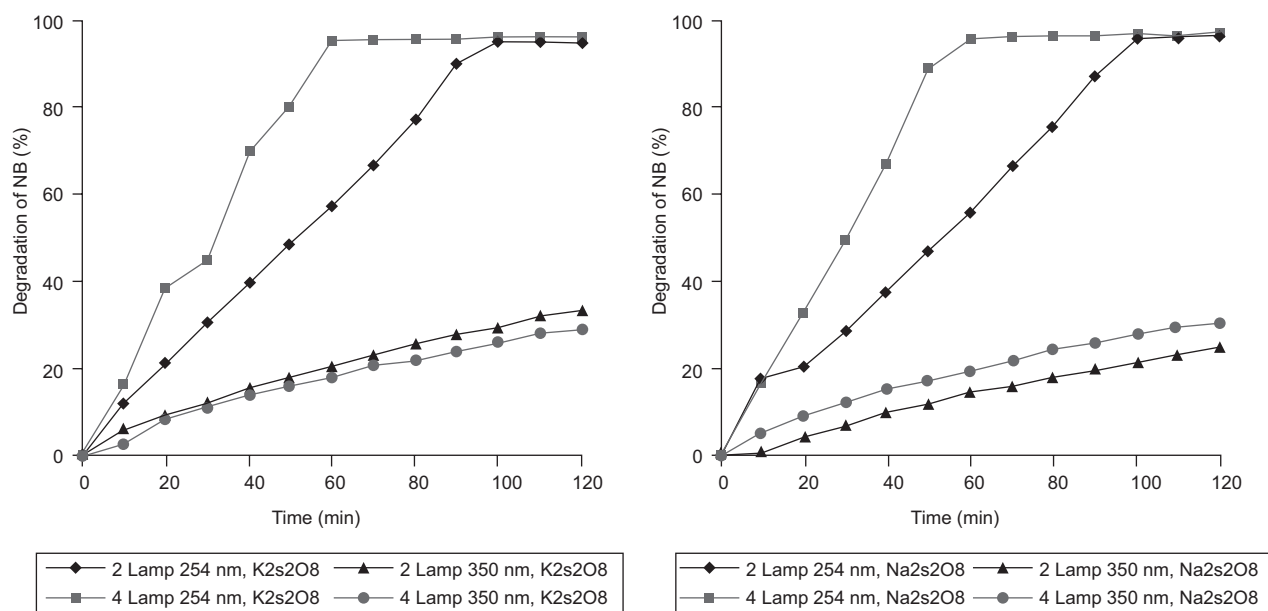


Fig. 5. Degradation of nitrobenzene (NB) at different radiation rate, 2 and 4 UV lamps with (a) $K_2S_2O_8$ and (b) $Na_2S_2O_8$. Conditions: $[K_2S_2O_8] = 5$ mM, $[NB]_0 = 20$ ppm, $T = 25$ °C and volumetric flow = 1 L/min

not very significant, since in several sampling times, concentrations of NB were practically the same.

For arrangement of two and four lamps of 254 nm, with $Na_2S_2O_8$ (**Fig. 5b**), the trend percentage of NB degradation is very similar to that using $K_2S_2O_8$, achieving maximum degradation at 60 min of reaction with four lamps, but after 100 min with both arrangements the same percentage of degradation is produced, so the amount of radiation also increases the reaction rate, but not the final removal of NB.

For the arrangement of two and four lamps of 350 nm, with $Na_2S_2O_8$ (**Fig. 5b**), the degradation percentages with four lamps are slightly higher than with two lamps (average increase of 6 %), but it is still not significant to assess the effect of this arrangement on NB degradation.

The difference in energies between the two wavelengths of UV radiation promotes photolysis of persulfate ion at different rate, so controlling the generation of the oxidizing species $SO_4^{\cdot-}$ responsible for the mechanism of radical-chain decomposition.

The results of specific energy consumption are shown in **table III**. According to them, the lowest energy consumption to degrade 1 kg of NB was reached while using two lamps of 254 nm, with a value of 2626.0 KW.h/kg of NB. On the other hand, the value using 4 lamps was 3121.7 KW.h/kg of NB. So by using two lamps less energy is required, but also more time is needed to reach the maximum degradation than using four lamps.

TABLE III. ESTIMATED SPECIFIC ENERGY CONSUMPTION FOR 2 AND 4 UV LAMPS

UV radiation	Lamps number	SEC KW.h/Kg of NB
254 nm	2	2626.0
	4	3121.7
350 nm	2	8928.6
	4	19280.5

CONCLUSIONS

- The results show that the degradation of NB was achieved in the photochemical reactor provided with UV lamps.

- The wavelength of UV lamps influences strongly the degradation process of NB because UV lamps with emission at 254 nm in both arrangements (two and four lamps) have approximately 3 times higher degradation than the lamps with emission at 350 nm, reaching rates up to 96 %.

- The reaction rate is directly proportional to the increase of radiation, which does not imply that at a greater amount of applied radiation a higher degradation percentage is obtained.

- The nature of the oxidizing agents used shows that there is no significant difference between $Na_2S_2O_8$ and $K_2S_2O_8$, so either can be used.

- With respect to the concentration of the agent added, the removal percentage of NB had the following order $5 < 10 < 15 < 20$ mM, reaching ultimate degradation with 5 mM for a reaction time of 60 min.

- The arrangement using two UV lamps of 254 nm showed the lowest energy consumption (2626.0 KW.h/kg of NB), but this arrangement needs more reaction time (100 min) to reach the highest degradation of NB. The highest energy consumption was for the arrangement of four UV lamps of 350 nm (19280.5 KW.h/kg of NB).

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