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Microwave-assisted rapid decomposition of persulfate

C. Costa [a,b], V.H.S. Santos [b], P.H.H. Araujo [a], C. Sayer [a], A.F. Santos [b,c], M. Fortuny [b,c,*]

[a] Departamento de Engenharia Química, Universidade Federal de Santa Catarina, Campus Universitário, Florianópolis, SC 88040-900, Brazil
[b] Laboratório de Sistemas Coloidais e Dispersões, Instituto de Tecnologia e Pesquisa, Av. Murilo Dantas 300, Aracaju, SE 49032-490, Brazil
[c] Programa de Engenharia de Processos, Universidade Tiradentes, Aracaju, SE 49032-490, Brazil

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ABSTRACT

Microwave irradiation has been a promising alternative to conduct several chemical reactions. In this work the microwave effects in potassium persulfate decomposition rate, under controlled conditions of temperature and microwave power, were evaluated. Higher decomposition rate constants were obtained in microwave irradiated reactions in comparison with conventional heated ones. To study the effect of high power microwave irradiation, a pulsed irradiation strategy was developed, in which the samples were repeatedly heated within short intervals of time at high power levels (500 or 1400 W). A great decomposition percentage was achieved in shorter irradiation times, showing the kinetic advantages of microwave-assisted reactions. However, it was found no differences in the reaction yields, even when high power levels were involved, proving that microwave enhancements may arise only from the ability to quickly provide a large amount of energy to the reaction medium.

1. Introduction

Microwave irradiation emerged as an attractive alternative to replace the conventional heating method, which has been recognized to be a slow and inefficient method due to its dependence on convection currents and on thermal conductivity of materials. Microwave heating arises from the ability of some materials to transform electromagnetic energy into heat [1,2]. This nonionizing radiation induces molecular motions – ions migration and dipoles rotation – resulting in friction, collision and, thus, in heat generation [2–5].

The use of microwave irradiation in organic synthesis has received growing interest in the last 20 years [3–21]. This is because microwave heating presents several attractive advantages over conventional heating, such as shorter reaction times, higher yields, cleaner reactions, and many others that have been extensively claimed during last years [3,6,22,23]. Besides these advantages some authors consider that the microwaves can provide specific effects (either thermal or not purely thermal effects) generally connected to the selective absorption of microwave energy by polar molecules [8,24,25].

Changes in the thermodynamic parameters under microwave irradiation were proposed as a cause of the non-thermal microwave effects [9,24]. On the basis of the Arrhenius reaction rate equation (Eq. (1)), it has been speculated that microwave field can induce an increase in the molecular vibrations, due to orientation of polar molecules in the reaction and, therefore, affect the Arrhenius pre-exponential factor A, which describes the frequency of collisions between molecules with the appropriate geometry for a reaction to occur. A decrease in the activation free energy ΔG^{*} promoted by microwave irradiation due to the contribution of enthalpy and entropy to the value of ΔG^{*} $(=\Delta H - T\Delta S)$ was also suggested [9,24]. According to this concept, the entropic term would increase in a microwave driven reaction, because the level of organization is enhanced with the dipolar orientation. Nevertheless, these theories have

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[*] Corresponding author. Address: Laboratório de Sistemas Coloidais e Dispersões, Instituto de Tecnologia e Pesquisa, Av. Murilo Dantas 300, Aracaju, SE 49032-490, Brazil. Tel./fax: +55 79 32182190.
E-mail address: fhmontse@yahoo.es (M. Fortuny).

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not been completely elucidated and the occurrences of non-thermal microwave effects are still doubtful.

$$k = Ae^{\frac{-\Delta G^*}{RT}} \tag{1}$$

Some observed microwave effects have been also connected to intervention of localized microscopic high temperatures (the so-called hot spots) [5–7]. This effect may arise from inhomogeneity of the applied field or from differences in dielectric properties of materials. As a consequence, energy is not homogeneously dissipated and the temperature in certain zones within the reaction medium becomes greater than the bulk temperature [7,10].

In this work we studied the effect of microwave irradiation in potassium persulfate decomposition reactions. Potassium persulfate is a water-soluble initiator frequently used in polymerization processes. Its thermally induced decomposition generates sulfate free radicals, which contribute to microwave heating, through ionic conduction mechanism. Initiator decomposition rates are strongly connected to polymerization reaction rates, and have been even suggested to be the cause of observed enhancements in microwave-assisted polymerization reactions [11–17]. Some publications have also suggested that specific microwave effects can be promoted by the use of high power microwave irradiation, and not by the heating method itself [16,17]. However, these microwave effects are still controversial because they cannot be easily separated in mechanistic studies [6,7]. Recent studies [18,19] focused on the microwave-induced miniemulsion polymerization have shown that the use of pulsed experiments including cycles of rapid heating at high microwave power with subsequent cooling in ice bath provides a means of obtaining polymers with tailored molecular weight distributions. The observed results were assumed to be purely thermal and could be explained by common radical heterophase polymerization kinetics.

In this context, potassium persulfate decompositions were carried out under both microwave and conventional heating, with the same experimental conditions, in order to evaluate the existence of specific microwave effects. The effect of high power microwave irradiation in these reactions was also studied. For this purpose, pulsed reactions were conducted, in which the samples were repeatedly heated and cooled down, receiving a great amount of energy within short intervals of time.

2. Experimental

2.1. Materials

Potassium persulfate initiator (KPS), analytical grade, was used directly as received, without further purification. Hydrated magnesium chloride ($MgCl_2{\cdot}6H_2O$), sodium acetate hydrate ($CH_3COONa{\cdot}3H_2O$), potassium nitrate (KNO_3, 99 wt%) and acetic acid (CH_3COOH, 99 wt%) were used to prepare a buffer solution. Barium chloride ($BaCl_2$, 99 wt%) and all other commercial chemicals were provided by Vetec and used as received. Water was distilled and deionized by filtration through a Millipore cartridge and used in the preparation of aqueous solutions.

2.2. Experimental procedures

2.2.1. Persulfate analyses by turbidimetric method

The concentration of sulfate ions formed by decomposition was determined by the classical turbidimetric method [26]. According to this method, sulfate ion reacts with barium chloride in acidic solution (acetic acid 2.0 v/v%) to form a suspension of barium sulfate crystals of uniform size. The resulting turbidity is determined by a spectrophotometer and compared to a curve prepared from standard sulfate solutions, enabling one to assess the sulfate concentration of the sample. During the KPS decomposition tests, samples collected from the reactor were mixed with a buffer solution composed of $MgCl_2{\cdot}6H_2O$, $CH_3COONa{\cdot}3H_2O$, KNO_3 and CH_3COOH in aqueous phase, followed by the addition of a measuring spoonful of barium chloride crystals, in order to precipitate the sulfate ions ($BaSO_4$ precipitate). The absorbance of the resulting solution was measured employing a Hitachi U-1800 spectrophotometer and used to determine the sulfate concentration. The decomposition reaction was assumed to follow the classical free-radical thermal initiation mechanism in accordance with Eq. (2), where k_d is the rate constant for initiator decomposition. Since the persulfate decomposition in aqueous solutions has long been viewed as a first order reaction [27], the persulfate concentration evolution with time can be described by Eq. (3).

$$S_2O_8^{\;2-} \xrightarrow{k_d} 2SO_4^{\;\bullet-}. \tag{2}$$

$$\ln\frac{[S_2O_8^{\;2-}]_{t=0}}{[S_2O_8^{\;2-}]} = k_d t \tag{3}$$

where $[S_2O_8^{\;2-}]_{t=0}$ and $[S_2O_8^{\;2-}]$ are the molar concentrations of initiator at the beginning of the reaction and at time t, respectively.

2.2.2. Decomposition of KPS under conventional heating

Initiator decomposition tests under conventional heating were carried out in aqueous media using a 1-L jacketed glass tank, with temperature control. Distilled and deionized (DDI) water (600 mL) was added into the reactor, equipped with a mechanical stirrer (150 rpm), a nitrogen inlet, a thermocouple (type J), and a condenser to minimize the loss of reactants, especially water, through evaporation. The medium was then heated to 60, 70, 75 or 80 °C with a thermostated water bath. In order to remove oxygen, the medium was purged with nitrogen at a low flow-rate throughout the entire process. KPS dissolved in DDI water (40 mL) was added and the temperature was kept constant at the desired value. Samples were taken out at regular intervals of time and cooled down by immersing into an ice bath to fully stop the reaction.

2.2.3. Decomposition of KPS under microwave irradiation heating

The microwave reactions were conducted in a Synthos 3000 multimode microwave reactor, from Anton Paar. The microwave source was of 2.45 GHz frequency magnetron powered by a 1400 W variable power generator, which could be operated at different power levels. To prevent nonuniform heating, this instrument is equipped with a rotor system in which eight quartz vials with an 80 mL capacity can be inserted at one time. In addition, samples could be stirred through a magnetic stirrer. The temperature of the sample was monitored by a gas bulb thermosensor inserted in the liquid phase of one reference vial. Additionally, an infrared sensor placed on the rotor bottom measured the surface temperature of the vials. The microwave reactor was also equipped with a pressure transmitter which provides pressure information of all vials. The control system supplied with the instrument enabled us to perform heating ramps with well defined sample temperature and applied power conditions programmed prior to the test.

2.2.3.1. Microwave heating – constant temperature. To perform the KPS decomposition under constant temperature, the vials were filled with aliquots (20 mL) of a previously prepared KPS solution, and purged with nitrogen during 30 min. Thereafter, the vials were closed with screw caps and were submitted to microwave irradiation during specific reaction times. The vials were promptly heated up to the reaction temperature and, after being kept at the desired temperature (60, 70 or 80 °C) for a certain time interval, the samples were taken out and quenched in an ice bath to stop reaction. The sample temperature just after the microwave heating time was determined by immersing a thermocouple into the sample directly and immediately after the prescribed reaction time. This thermocouple was similar to that one used during conventional tests. This was done in addition to the measurements obtained by the temperature sensor immersed in the reaction medium, to ensure a reliable temperature monitoring. The formed sulfate ions were determined by turbid measurements and used as input to compute the decomposition rate constant from Eq. (3). This experimental procedure was repeated for different reaction times.

2.2.3.2. Microwave heating – pulsed irradiation at constant power. Pulsed microwave reactions were carried out to investigate the effect of high power microwave irradiation in KPS decomposition. After purging the 20 mL KPS solutions with nitrogen during 30 min, the samples were repeatedly heated from room temperature to 60–90 °C through microwave irradiation at constant power, within short intervals of time (between 24 and 80 s depending on the applied power). The samples were cooled down in an ice bath for 4 min between microwave irradiation pulses. Experiments were carried out applying 1–4 irradiation pulses. After each procedure, samples were collected and the formed sulfate ions were determined by turbid measurements. The microwave power was adjusted to be kept constant at 500 or 1400 W during sample heating.

3. Results and discussion

3.1. Decomposition of KPS under conventional and microwave heating – constant temperature

The decomposition rate constants (k_d) of KPS were determined for both microwave and conventional conditions at four different temperatures (60, 70, 75 and 80 °C). The obtained values are shown in Table 1. Compared with k_d under conventional heating, k_d values under microwave heating were about 3–4 times higher, indicating that microwave irradiation improved KPS decomposition. These results agree with those already reported [15–17], which showed an increase of 2.4–4.8 times in the decomposition rate under microwave irradiation compared with conventional heating. Table 2 presents some values obtained from literature.

Table 1
Decomposition rate constant (k_d) of KPS obtained experimentally, for microwave (MW) and conventional heating (CH).

T (°C)	k_d (s^{-1})		k_d MW/k_d CH
	Microwave	Conventional	
60	2.97×10^{-5}	1.00×10^{-5}	2.97
70	1.03×10^{-4}	2.83×10^{-5}	3.64
75	1.86×10^{-4}	4.25×10^{-5}	4.38
80	3.31×10^{-4}	1.02×10^{-4}	3.24

According to Table 1, a decomposition rate enhancement was observed for all microwave irradiated reactions. Considering that careful bulk temperature control was guaranteed, we found that specific microwave effects, either thermal or non-thermal effects, can explain the observed microwave-enhanced decomposition rates. As mentioned before, the acceleration of chemical reactions under the action of microwaves have been linked to non-purely thermal effects produced by microwaves, which could be explained on the basis of modifications in each of the terms of the Arrhenius reaction rate equation (Eq. (1)) [9,24]. According to this theory, the k_d value could be influenced by three factors: the pre-exponential factor, A, the activation free energy, ΔG^*, and the reaction temperature, T.

In order to investigate the origin of microwave effects in the KPS decomposition reaction, we determined the parameters of Arrhenius equation for both heating conditions (shown in Table 3).

The pre-exponential factor obtained for microwave heated reactions (in Table 3) was higher than that found for conventional heating conditions. This could be due to an increase of molecular mobility induced by microwaves. Researches in organic chemistry [28] have previously explained the observed accelerations in reaction rates based in Arrhenius pre-exponential factor increases. Some other authors have described, however, that there were no differences in these reactions under microwave and conventional heating, postulating therefore that the pointed non-thermal effect is not existent [4,7].

Table 2
Decomposition rate constant (k_d) of KPS reported in literature, for microwave (MW) and conventional heating (CH).

T (°C)	k_d (s^{-1})		k_d MW/k_d CH	References
	Microwave	Conventional		
60	2.37×10^{-5}	8.03×10^{-6}	2.95	Zhu [17]
68.5	8.05×10^{-5}	3.35×10^{-5}	2.40	Zhu [16]
110	1.01×10^{-2}	2.11×10^{-3}	4.79	Li [15]

Table 3
Pre-exponential factor (*A*) and activation free energy (ΔG^*) of Arrhenius reaction rate equation.

	A (s^{-1})	ΔG^* (kJ/mol)
Microwave	9.13×10^{13}	118
Conventional	1.11×10^{12}	109

The activation free energy (ΔG^*) of the reaction performed with microwave irradiation (in Table 3) was slightly higher than that obtained with conventional heating, showing that this parameter is not significantly influenced by microwave energy. This contrasts with Bao and Zhang [13] results, who found a decrease in the apparent activation free energy in KPS decomposition reaction. A reduction on the activation free energy in microwave-assisted chemical reactions was also suggested by Berlan et al. [29], Lewis et al. [30], Shibata et al. [31], Jovanovic and Adnadjevic [32]. Some of these results were contradicted by Raner et al. [33] who determined and evaluated activation parameters for organic reactions heated dielectrically and conventionally, and found no divergences on kinetic parameters.

Subsequently, there is a wide range of published studies [10,34–40] that reevaluated some microwave-assisted reactions and found no differences depending on the heating mode. It was indicated that most rate enhancement effects were observed during microwave heating because there was inadequate temperature monitoring and control or by the assignment of one temperature to a sample which in fact had a high temperature gradient.

The development of thermal gradients within the sample was suggested to be the cause of several observed microwave effects reported in literature [29,30,38]. Existence of hot spots seems to be the more probable cause of the reaction rate enhancement observed in our work under microwave irradiation. From this point of view, we could state that decomposition temperatures differ from those experimentally measured for the bulk, leading to inadequate comparisons between microwave-assisted and conventional reactions. As only the bulk temperature is measured (because microscopic temperature cannot be assessed), there is no experimental evidence to ensure this possibility.

We may also consider that the solution conductivity is expected to increase due to the presence of salts (potassium persulfate) and radicals (sulfate) formed throughout the initiator decomposition. This may contribute to the ionic conduction mechanism, leading to rapid local temperature enhancements which could explain deficiencies in measuring the real temperature.

Another hypothesis that has been already mentioned to explain the microwave effects in initiator decompositions is that the rapidly alternating electric field may align the radicals generated by decomposition, reducing the amount of geminate recombination and thus enhancing the initiator efficiency [11]. This effect can play a role in microwave-assisted KPS decomposition and should be tested carrying out reactions with other kinds of initiator, where geminate recombination is of reduced importance.

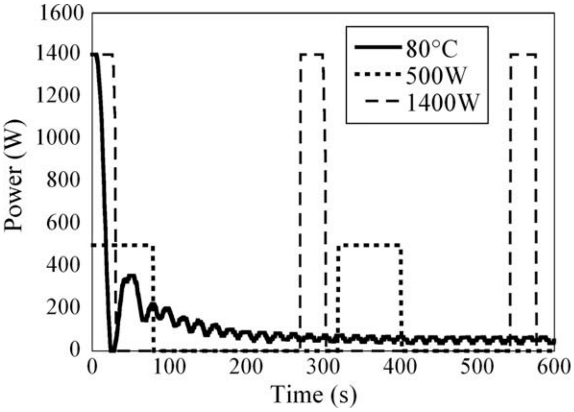

Fig. 1. Power profile for microwave heated reactions: constant temperature (80 °C) and pulsed irradiation at constant power (500 and 1400 W).

3.2. Decomposition of KPS under microwave heating – pulsed irradiation at constant power

Within short intervals of time samples were heated, at constant irradiation power, up to temperatures at which rapid KPS decomposition occurs, immediately followed by a temperature quench in an ice bath, where the samples were left for 4 min. The heating times varied from 24 to 80 s depending on the final temperature and the supplied power, and these cycles (heating + cooling) were repeated four times. Power and temperature profiles for pulsed irradiation reactions at constant power are shown in Figs. 1 and 2, along with profiles for reactions performed at constant temperature.

Fig. 1 shows that power is alternately on and off in pulsed reactions, while in reactions at constant temperature it is continuously on, remaining at low levels for almost the entire experiment (except in the first seconds, when power has its maximum value in order to heat the sample up to the desired temperature). This procedure with cycles of heating and cooling allows one to supply a

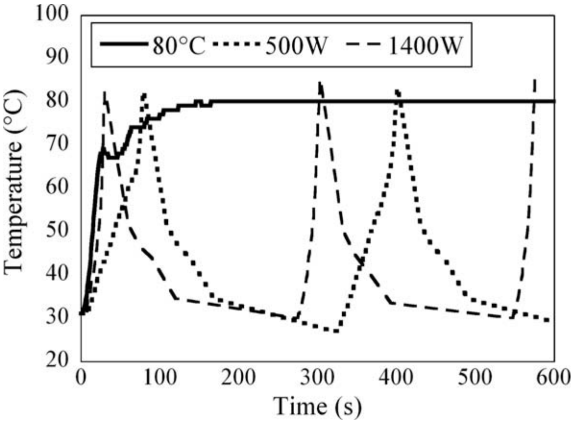

Fig. 2. Temperature profile for microwave heated reactions: constant temperature (80 °C) and pulsed irradiation at constant power (500 and 1400 W).

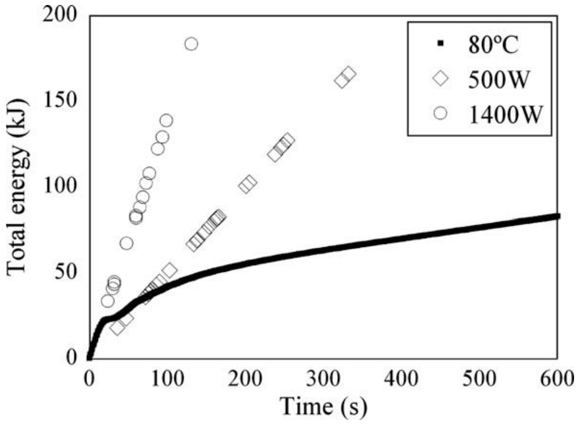

Fig. 3. Total energy applied with irradiation time for microwave heating at constant temperature (80 °C) and at constant power (500 and 1400 W).

greater amount of energy to the reaction media compared to those tests conducted at constant temperature. In addition, the temperature of the samples is continuously increased and decreased (in Fig. 2), preventing samples overheating. As a result, we can reduce dramatically the irradiation time, without achieving higher temperatures. This can also be seen in Fig. 3, which shows the total energy applied by microwaves source as a function of irradiation time, for constant power at 500 W or 1400 W, and for constant temperature at 80 °C. The total energy was calculated by the numerical integration of the power profiles shown in Fig. 1. According to results shown in Fig. 3, an energy level of 100 kJ may require about 70 s at constant 1400 W, or about 200 s at constant 500 W, or even more than 600 s at constant temperature.

Heating pulses at constant 500 or 1400 W were conducted until the desired temperatures were achieved. According to the temperature profiles obtained in these tests, three main final temperature levels (T_f) may be distinguished: 83 ± 3, 76 ± 3 and 64 ± 4 °C. The obtained decomposition percentages (computed from Eq. (4)) for each temperature level and power condition are presented in Fig. 4. It should be noted that for each experimental condition, the increase on the total energy was accomplished by incrementing the number of cycles.

$$\text{Decomposition}\ (\%) = \frac{[S_2O_8{}^{2-}]_{t=0} - [S_2O_8{}^{2-}]}{[S_2O_8{}^{2-}]_{t=0}} \times 100 \qquad (4)$$

As one can observe from Fig. 4, the persulfate decomposition is strongly affected by the final temperature level, in such a way that at lower temperatures, the increase of applied energy has smaller effects on persulfate decomposition than at higher temperatures, which means that even during the pulsed scheme k_d should follow some sort of Arrhenius relation. Moreover, results shown in Fig. 4 indicate that the decomposition was not affected by power level. Despite the significantly difference in power levels, we found no change in the reaction yields. It seems that, besides the final temperature of the solution, what is really significant for the decomposition extent is the amount of energy effectively applied into the medium. Studies about microwave irradiation with simultaneous cooling [34,35] have also obtained similar results using different power levels, concluding that the microwave power applied to the reaction is apparently irrelevant. In this way, the only effect obtained using higher power irradiation was the decrease in the needed time to supply a certain amount of energy to the mixture. The reduction of time is evident in reactions under different power levels (see Table 4). To achieve 83 °C, it was necessary about 80 s at 500 W, but only about 32 s at 1400 W. At lower temperature, the difference is smaller, but also significant (46 and 24 s, for 500 and 1400 W, respectively). Thus we can save a significant time reaction applying high power irradiation, without changing the provided amount of energy.

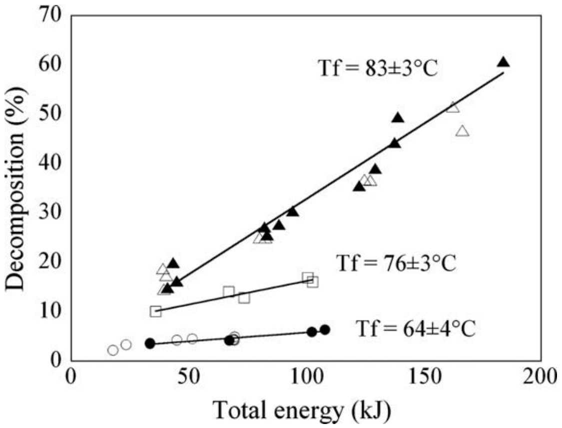

Fig. 4. KPS decomposition as a function of the total energy applied. Conditions: (△) 83 ± 3 °C at 500 W (▲) 83 ± 3 °C at 1400 W (□) 76 ± 3 °C at 500 W (○) 64 ± 4 °C at 500 W (●) 64 ± 4 °C at 1400 W (the lines represent the best fit for each T_f condition).

4. Conclusions

The use of microwave irradiation enhanced the potassium persulfate decomposition rate constant about 3–4 times, in comparison with values obtained under conventional heating. The kinetic parameters were determined for both heating conditions, and were found no significant differences between them, indicating the inexistence of non-thermal microwave effects in these reactions. The observed microwave effects were ascribed to the development of thermal gradients within the sample (the hot spots), arisen from inhomogeneity of the applied field or from ionic conduction induced by the dissolved sulfate ions.

Table 4
Average irradiation time applied to achieve 83 ± 3, 76 ± 3 or 64 ± 4 °C, under constant power (500 or 1400 W).

Temperature (°C)	Power (W)	Average irradiation time (s)	Power (W)	Average irradiation time (s)
83 ± 3	500	80	1400	32
76 ± 3	500	75		
64 ± 4	500	46	1400	24

Studies on the effect of high power microwave irradiation upon the potassium persulfate decomposition showed that the decomposition was not affected by power level, in agreement with the statement that only thermal effects are present in these reactions.

Despite these findings, the use of high power microwave irradiation promotes a drastic decrease in the reaction time, by applying a great amount of energy to the reaction medium in short intervals of time. Rapid reactions can be performed using cycles of heating and cooling, improving decomposition reactions, and, consequently, polymerization processes.

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