The reactor design as a critical input in the electrochemical production of peroxoacetic acid

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Abstract

In the present work, the influence of the most critical inputs affecting the reactor design for the simultaneous electrochemical production of peracetic acid and hydrogen peroxide are studied. Based on our previous findings, we studied the role of the ratio between the volume of the solution (related to the chemical equilibrium between PAA and hydrogen peroxide) and the intensity (related to the rate and efficiency of the electrochemical processes). Moreover, we tested the performance of a jet-reactor, for the production of PAA. According to the results, it can be stated that the production of PAA is robust and stable when working at volume to intensity ratios equal or higher than 8.12 L A^{-1} . Moreover, it was found that a current densities around 27 A m^{-2} are suitable for both reactor designs. Finally, it was observed that the production rate of PAA is higher for the GDE-reactor due to the higher value of the volume to intensity ratio, the higher relative cathode vs. anode area and the reactor configuration (anode-cathode). This complex scenario demonstrates the key role of the reactor design and clarifies the future research lines that have to be further developed to work on an efficient electrochemical production of peracetic acid.

Highlights

- PAA production is stable with volume/intensity ratios of, at least, 8.12 L A^{-1}
- The optimal PAA production is obtained at 27 A m^{-2} for both prototypes
- The GDE-device gives a higher PAA and H_2O_2 production rate than the jet reactor
- The dissimilar configuration, volume and electrode area explains the different rates

Keywords: peroxoacetic acid; reactor optimization; hydrogen peroxide; electrochemical production; BDD

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1. Introduction

The production of peroxoacetic acid (also peracetic acid, peroxyacetic acid, or PAA) is reaching an increasing industrial interest due to its properties related to disinfection [1-3], bleaching [4, 5] and oxidant agent [6-8]. The in-situ production of a chemical reagent is always favorable from the viewpoint of reducing the CO_2 footprint related to its transportation [9]. However, in the case of PAA is even more important, because of the difficulties found in the handling and transport of peracetic acid [10, 11].

The base reaction for the production of peroxoacetic acid is the equilibrium between acetic acid and hydrogen peroxide (Equation 1), which is shifted to the production of PAA in acidic media [12].

$$CH_3COOH + H_2O_2 \leftrightarrows CH_3COOOH + H_2O \quad (1)$$

Thus, the formation of peroxoacetic acid is directly linked to the production of hydrogen peroxide, being necessary to assure a minimum concentration of the latter to promote the

formation of PAA. Because of that, a PAA formulation consists of a ternary mixture of the raw acetic acid and the two electrogenerated hydrogen peroxide and PAA molecules. Based on this fact, our research group recently proposed two advances in the design of electrochemical reactors that can be used in the electrochemical production of peracetic acid. First, we presented a prototype in which we performed the production of PAA from acetic acid aqueous solutions combining the oxidation of acetic acid by BDD anodes and the cathodic production of hydrogen peroxide using a carbon-based gas diffusion electrode (GDE) [13, 14]. With this design, we observed a clear synergistic effect of combining both the anodic and the cathodic processes in the production of PAA, giving as a result the highest concentration reported so far in the literature for the electrochemical production of PAA. Moreover, in those works, we demonstrated that the flow direction (from cathode to anode or vice versa) played an important role in the efficiency of the process and we hypothesized that it should be necessary a minimum ratio of reaction volume *vs* electrode area to permit the hydrogen peroxide reacts with acetic acid, lowering the consumption of this key reagent in electrochemical parasitic reactions.

Simultaneously, we developed a novel design for the electrochemical production of hydrogen peroxide based on two innovations: 1) replacement of the compressor used in a typical GDE design by a jet device, that supersaturates the solution in oxygen, and 2) the use of a flow-through cathode [15-17]. This device has been tested for the production of hydrogen peroxide and showed a promising behavior, reporting some of the highest production yields and efficiencies previously reported for the electrochemical production of this compound.

Based on these findings, in the present work we move forward in the design of efficient electrochemical devices for the simultaneous production of PAA and hydrogen peroxide 1) by studying the role of the ratio between volume of solution (chemical reaction zone)

and the intensity (electrochemical reaction zone) for the GDE-based reactor and 2) by exploring the applicability of the jet reactor for the combined production of PAA and hydrogen peroxide. With the work proposed and discussed here, we aim to confirm the hypotheses reached in previous works and to stablish design rules for the efficient simultaneous production of hydrogen peroxide and PAA.

2. Material and methods.

2.1. Experimental setup.

The production of peroxoacetic acid (PAA) is carried out by the combination of the oxidation of the acetic acid at the anode and the production of hydrogen peroxide at the cathode. The acetic acid (10% aqueous) is used as raw material and it was stored in the amber glass tank, which was continually recirculated through the electrochemical cells by a centrifugal pump. This process was carried out in bench-scale and galvanostatic conditions in the range from 12.7 to 191.0 A m^{-2}, always referred to the anode area. The temperature was kept constant at 25ºC by a heat exchanger. A modified carbon felt (CF) material was selected as cathode. Its modification consisted in the immersion of the CF into a solution of distilled water (30 mL), carbon black (0.3 g Vulcan ® XC72R), PTFE (0.3 g 60% Teflon ® emulsion solution from ElectroChem, Inc.) and n-butanol (3%) for 60 min in an ultrasonic bath [15]. A boron doped diamond (BDD) provided by NeoCoat, Switzerland was used (boron content: 500 -700 ppm; sp^3/sp^2 ratio: 220 ± 5%; thickness: 2.7μm ± 10%).

Two different electrochemical cells were used and compared in the present work. The first cell (referred in this manuscript as GDE reactor or GDE device), described in detail in previous works [13, 14], consists of an electrochemical reactor with three chambers: anode, cathode and gas chamber. It is used a Carbon Felt (CF) as cathode with a

rectangular area of 33.75 cm^2, which separates the gas phase from the cathodic chamber. BDD was used as anode with a rectangular area of 18.24 cm^2. The anodic and cathodic compartments were separated by cationic membrane (NAFION N-117). The air required to produce hydrogen peroxide in the cathodic compartment was provided by a vacuum pump that acts as an air compressor.

The second cell (referred in the manuscript as jet reactor or jet device) is schematized in Figure 1 and consists of an electrochemical reactor where the cathodic compartment is flow-through. As anode, a BDD (circular area was 78.54 cm^2) was used. A modified carbon felt (with a rectangular area 100.00 cm^2) was placed as cathode. The oxygen required to produce hydrogen peroxide in the cathode was provided by a jet device that allows the intake of air by the well-known venturi effect. A fix volume of 0.6 L of acetic acid (10% aqueous) was pumped to the cell across the jet device. Inside the cell, the solution first crosses the cathode, where it was produced the hydrogen peroxide, and then gets to the anodic surface, exiting the cell through a perpendicular outlet.

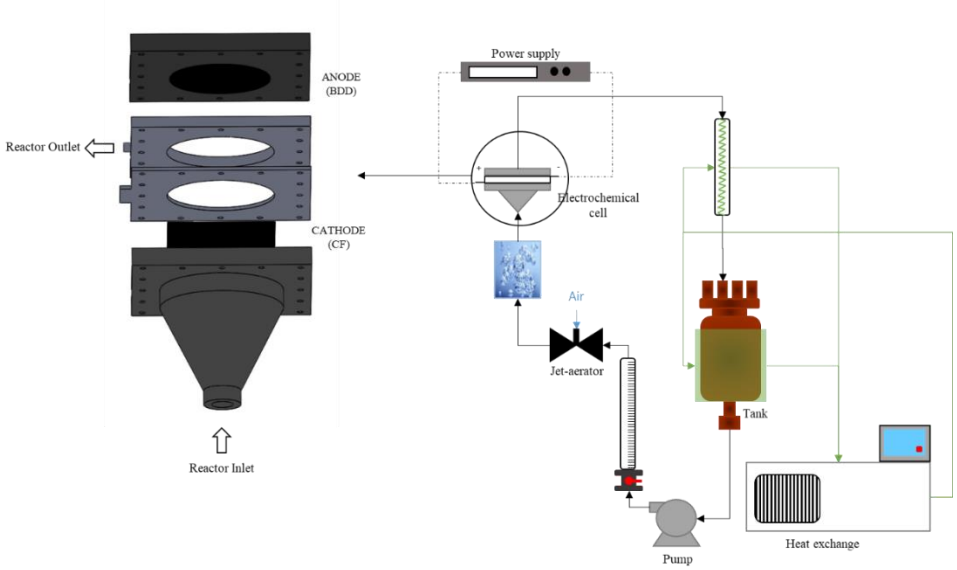

Figure 1. Schematic plot of the jet-based electrochemical reactor for the simultaneous production of hydrogen peroxide and peracetic acid.

2.2. Analytical techniques.

The determination of peroxoacetic acid is complex due to the equilibrium reached between this compound, acetic acid and hydrogen peroxide. As it was explained in a previous work [14], it was decided to calculate the concentration of PAA as the difference between the amount of total oxidants and that of hydrogen peroxide. The hydrogen peroxide was determined by potassium titanium (IV) using the oxalate method. The absorbance was determined at 410 nm by and Agilent 300 Cary series UV- VIS spectrophotometer. The total oxidants were determined by titration with $Na_2S_2O_3$. The procedure consisted of adding 5 mL of 20% (v/v) H_2SO_4 solution to 10 mL of sample with an excess of solid KI. This iodide was oxidized to form brownish iodine, which was titrated with 0.002 N $Na_2S_2O_3$ until a colorless solution was obtained. Sigma-Aldrich supplied all reagents used and the titanium solution was supplied by Fluka.

3. Results and discussion.

3.1. Role of ratio volume vs. electrode area

In previous works [14], we pointed out that it is necessary to balance chemical and electrochemical processes for a successful PAA production, in order to permit the hydrogen peroxide reacts efficiently with acetic acid and stabilize the PAA formulation. This balance in the reactions must have a reflection on the ratio between the volume of solution (chemical reaction zone) and the intensity (electrochemical reaction zone), which in turn can be modified either with the current density (operation parameter) or with the electrode area (sizing parameter). It was demonstrated that the concentration of hydrogen peroxide (and thus the concentration of peracetic acid) is increased when the fluid enters the cell through the anodic chamber and it exists through the cathodic chamber. It was

explained in the base of the concurrence of parasitic reactions. If the fluid comes out the cell through the cathodic chamber, where the hydrogen peroxide is being produced, its concentration is the highest at the outlet of the cell, being available to react with acetic acid. On the contrary, if the cathodic chamber is placed first, the fluid circulates latter through the anode, where hydrogen peroxide can be oxidized, diminishing the overall efficiency of the process.

Based on the previous explanation, it was hypothesized that a minimum reaction volume (for a given electrode area) would be necessary to allow hydrogen peroxide react with acetic acid. This minimum reaction volume may depend on the relation between the rates of hydrogen peroxide production and destruction pathways and on the kinetics of Equation (1), that is, the reaction between hydrogen peroxide and acetic acid.

Thus, the first attempt made in this work was to evaluate the influence of the reactor design on the efficiency in the production of peracetic acid and hydrogen peroxide consisted of varying the ratio of reaction volume *vs* electrode area. To do this, the GDE reactor described in the materials section was tested with three increasing reaction volumes (400 mL, 600 mL and 800 mL) trying to explore ratios bulk volume/electrode area over the evaluated in that previous work, in order to improve what it is considered the bottleneck of this production: the chemical interaction of the ternary system acetic acid / hydrogen peroxide / PAA in the PAA formulation

Figure 2 represents the amount (mmol) of peracetic acid (Figure 2.a) and hydrogen peroxide (Figure 2.b) produced with respect to the electrolysis time. Although the specific electric charge and concentration are generally used in this type of plots to compare results, these two parameters should not be used in this case to compare the production rate, because they both depend on the reaction volume, and this parameter is varied between the different tests. In all cases, the current density was kept constant to 27 A m^{-}

^{2}, because this value corresponds to the optimum current density obtained in a previous work with a reaction volume of 400 mL [14]. As these tests are the base for evaluating the influence of the volume to area ratio, they were performed in triplicate, being the standard deviation represented as error bars.

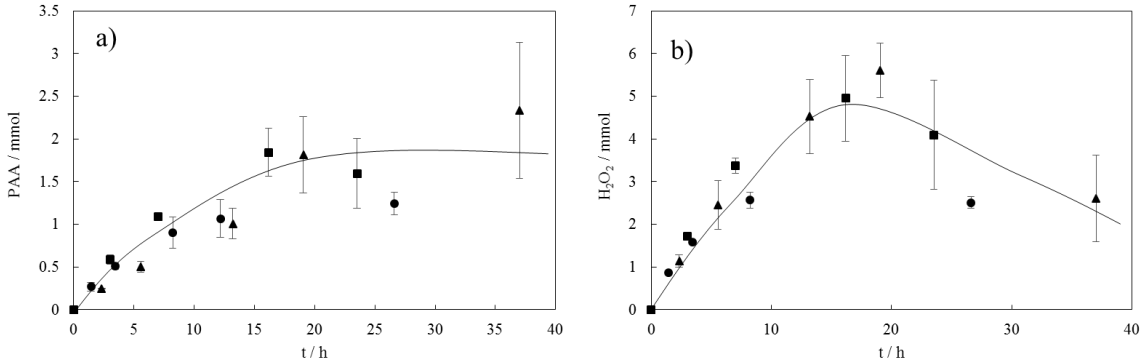

Figure 2. Amount of peroxoacetic acid (Figure 2. a) and hydrogen peroxide (Figure 2.b) generated versus time for different reaction volumes: 400 mL(●); 600 mL(▲); 800 mL(■). Constant current density: 27 A m^{-2}

As it can be observed, in all cases the concentration of peracetic acid increases with time until reaching a plateau. Hence, PAA is formed successfully using this electrochemical cell. Likewise, hydrogen peroxide is formed but at longer times. In addition, it is observed a decrease in the amount produced instead of a plateau. This initially unexpected behavior indicates the complexity of the processes happening in the cell.

Thus, the peracetic acid can be produced through several mechanisms. First, it can be formed by the reaction between acetic acid and hydrogen peroxide (reaction 1), being the latter produced by reduction of dissolved oxygen at the cathode (reaction 2) [13, 18]. Moreover, the production of peracetic acid can also be performed via oxidation mechanisms, due to the reaction between acetic acid and oxygen radicals (equation 3) or with hydroxyl radicals (equations 4 and 5), both of them produced on the BDD surface [19]. Reaction 1 can take place in the bulk while the others are promoted in the nearness of the electrodes, due to the very short lifetime of radicals.$H^+ + ½\ O_2 + 2\ e^- \leftrightarrows H_2O_2$ (2)

$CH_3COOH + O^{\cdot} \rightarrow CH_3COOOH$ (3)

$CH_3\text{-}COOH + OH^{\cdot} \rightarrow (CH_3\text{-}COO)^{\cdot} + H_2O$ (4)

$(CH_3\text{-}COO)^{\cdot} + OH^{\cdot} \rightarrow CH_3\text{-}COOOH$ (5)

Regarding hydrogen peroxide, it increased within the first part of the tests due to the reduction of oxygen (equation 2) but it further decreased due to the consumption of hydrogen peroxide to form PAA (equation 1) and, more importantly, due to the concurrence of parasitic reactions, *f. i.* the reduction (equation 6) and/or oxidation (equation 7) of hydrogen peroxide, which becomes more important as the concentration of hydrogen peroxide produced is higher. This decrease in the concentration of hydrogen peroxide is a serious drawback as the final PAA formulation should keep a good ratio hydrogen peroxide / PAA in order to keep stable the concentration of the PAA reagent contained in the formulation.

$H_2O_2 + 2\ e^- + 2\ H^+ \rightarrow 2\ H_2O$ (6)

$H_2O_2 \rightarrow H^+ + ½\ O_2 + e^-$ (7)

For both PAA and hydrogen peroxide, the formation rate slightly increased when passing from 400 mL to 600 and 800 mL of total volume, although this increase seems to be within the standard deviation of the three replicates performed, which means that this increase cannot be considered significant from an exigent statistical point of view. Thus, if the production of PAA is compared at a value of time at which its production is almost stabilized (25 hours for all cases), there is an increase of 0.58 mmol (from 1.24 to 1.82 mmol) when increasing from 400 mL to 600 mL and of 0.60 mml (from 1.24 to 1.84 mmol) when passing from 600 mL to 800 mL. If this is compared to the averaged standard deviation (0.286 mmol), it can be concluded that these increases are not significant with a confidence of 90 % (t·s value of 0.84 mmol) and it is necessary to decrease the confidence value to 83% to consider significant the change in PAA production from 400 mL to 800 mL (the increase in the production being equal to the value of t·s). This

observation means that, with ratios volume vs. intensity 8.12 L A^{-1} or its equivalent ratio volume vs. anode area of 219.3 L m^{-2}, the performance of the system is robust and the desired PAA-product obtained is stable.

Another way to change the balance between the chemical and electrochemical reactions, is to change the intensity applied to the cell. This can be done either by changing the area at a constant current density or, in an easier way, by modifying the current density keeping the same electrode area. In this work, we used the second choice. Figure 3 shows the production of PAA at two higher current densities, 52 and 179 A m^{-2}, selected in order to make the electrochemical contribution more important than in the test carried out at 26 A m^{-2}. As seen, again, results are overlapped for the different volumes tested for each current density and this clearly indicates that even with a ratio as low as 1.23 L A^{-1}, the chemical reaction required is not limited and a stable PAA formulation can be obtained.

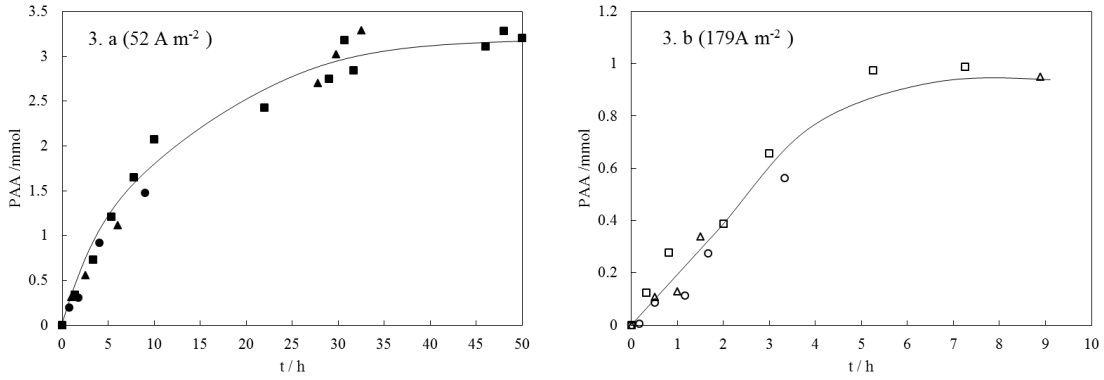

Figure 3. Amount of peracetic acid generated versus time for different reaction volumes and two current densities. 52 A m^{-2} (Fig 3.a, full symbols); 179 A m^{-2} (Figure 3.b, empty symbols). 400 mL (●,○); 600 mL(▲, △); 800 mL(■, □).

On the contrary, in comparing the results obtained for the different current densities, it can be seen that the higher the current density, the lower the efficiency of the process. The rate of PAA production at 52 A m^{-2} is similar to that obtained at 27 A m^{-2}, what means that the efficiency is much lower because the amount of charge passed for the same

time is almost doubled (double current density). In the case of 179 A m^{-2}, both the rate and efficiency are clearly diminished. At this point, it is important to have in mind that the two resulting molecules from these cells (those that made the mixture PAA/hydrogen peroxides expected as PAA formulation) are not final products but intermediates of larger reaction sets. In fact, PAA is an intermediate in the further anodic oxidation of acetic acid to carbon dioxide, while the hydrogen peroxide is an intermediate in the further cathodic reduction of oxygen to water. Hence, a higher current density affects negatively because although the overall rate of the process will be increased, electric charge will be used for side reactions and not for the desired reactions expected in the production and stabilization of the PAA product.

This means that for an efficient and optimal production of the PAA formulation current densities below 26 A/m^2 and volume/intensity ratios higher than 8.12 L A^{-1} has to be proposed.

3.2. Role of the reactor configuration

A key aspect to be considered for the success of an electrochemical synthesis process is the design of the reactor. In this research field, our research group recently developed a flow through jet-reactor (described in the experimental section) that efficiently performs the production of hydrogen peroxide [15-17]. This reactor design is based on the use of a flow-through carbon-based cathode and on the application of a jet aerator as an oxygen supplier instead of using a compressor. Based on this idea, a similar concept was developed for the production of peracetic acid in the search for a higher efficiency in the production of these valuable products. It is important to point that this concept was tested successfully to produce hydrogen peroxide, demonstrating an improved performance as compared to conventional GDL-cell.

Figure 4 shows the amount of peracetic acid (Figure 4.a) and hydrogen peroxide (Figure 4.b) vs. the applied electric charge for increasing current densities ranging from 12.7 A m^{-2} to 191.0 A m^{-2}.

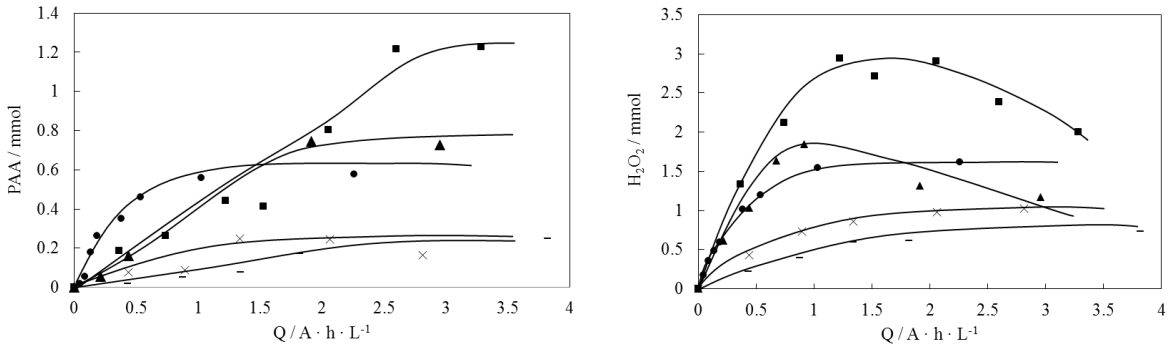

Figure 4. Amount of peracetic acid (4.a) and hydrogen peroxide (4.b) generated versus applied electric charge for the jet-based reactor at increasing current densities. ● 12.7 A m^{-2}; ■ 26.7 A m^{-2}; ▲ 63.7 A m^{-2}; - 127.3 A m^{-2}; x 191.0 A m^{-2}

In general terms, the production of peracetic acid follows a similar trend to that described for the GDE reactor. This means an increase in the production of PAA in the first stage of the tests, reaching a plateau from a given charge threshold. As it was previously described for the reactor based on GDE, the maximum amount of PAA is produced at an intermediate value of current density (26.7 A m^{-2} in this case). At higher values of current density, the amount of PAA accumulated in the reactor is lower due to the concurrence of parasitic reactions, including the oxidation/reaction of hydrogen peroxide and peracetic acid, as previously commented in Section 3.1.

To compare the performance of the two prototypes developed in this work, Figure 5 presents the amount of peroxoacetic acid (5.a) and hydrogen peroxide (5.b) for the optimal value of current density (26 A m^{-2}) and three values of applied electric charge.

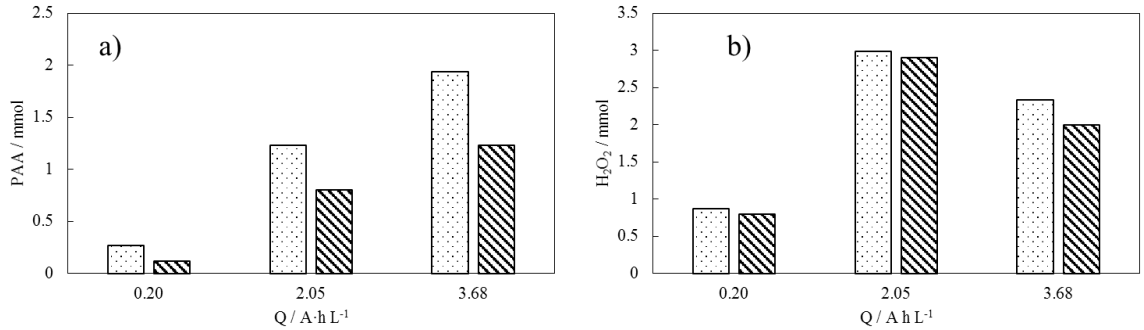

Figure 5. Amount of peracetic acid (5.a) and hydrogen peroxide (5.b) generated versus applied electric charge for GDE-reactor (dotted bars) and jet-reactor (stripped bars). j = 26.7 A m^{-2}

As it can be observed in Figure 5, the rate of peroxoacetic acid and hydrogen peroxide production by the GDE-reactor is higher than that registered for the jet-reactor. Surprisingly, the differences observed between the two designs are more accused in the production of peracetic acid than in the formation of hydrogen peroxide. This dissimilar behaviour can be explained attending to two key aspects of the design of both prototypes. The first one is the flow pattern of both reactors. In our previous work [14], it was demonstrated that when the fluid pass first through the anode chamber and then through the cathodic side of the reactor (flow anode-cathode), the rate of PAA production was higher than with a cathode-anode configuration. In the case of the jet-reactor presented in this work, it is difficult to work with an anode-cathode configuration because the inlet of the reactor in the anode side is perpendicular and a high tangential velocity is required to assure an adequate flow of air through the jet device. Thus, the flow of the fluid is cathode-anode, which may explain the lower efficiency in the formation of peracetic acid than that found in the GDE prototype.

Secondly, it is also important to compare the ratio volume/anode area of both prototypes. In both cases, the volume was the minimum required to assure the operation of the devices and to permit the sampling of the required number of samples. Taking this into account,

in the GDE reactor, the volume/area ratio was 0.219 L cm^{-2} meanwhile in the jet design the ratio is 0.0764 L cm^{-2}. As it has been previously commented and was postulated in our previous works, a lower volume may cause that the hydrogen peroxide produced is not as efficiently used in the formation of PAA (Equation 1). Finally, the ratio between anode and cathode area of both prototypes is not equal due to the dissimilar geometry of both devices. The jet reactor presents an anode area 4.3 times greater than the GDE device, meanwhile the external cathode area is 2.9 times higher. This may promote preferentially the anodic reactions, including the decomposition of hydrogen peroxide and the oxidation of acetic acid to molecules different from PAA. As the formation of PAA is directly linked to the production of hydrogen peroxide and this production is expected to be mainly a cathodic process (especially taking into account the high ratio sp^3/sp^2 of the BDD layers used (255) [20]), a lower overall efficiency in the production of both oxidants can be explained for the jet device.

These results confirm the huge significance of the reactor design of the efficient production of PAA and indicates that further work must be done in the reactor design in order to improve the efficiency in this production, which is especially dependent on the reactor design, because of the many factors that influence on that complex process. Thus, when trying to use a more efficient reactor to produce hydrogen peroxide, results were worse because this cell design did not promote the special characteristics required for the production of peracetic acid.

Conclusions

From the present work the following conclusions can be obtained:

- The GDE-reactor exhibits a robust and stable PAA formulation when working at ratios volume vs. intensity equal or higher than 8.12 L A^{-1}

- The optimal production of the PAA formulation is performed at a current density close to 27 A m^{-2} in both prototypes tested.
- The production of PAA and hydrogen peroxide performs better in a GDE-device (with anode-cathode configuration) than in a jet-reactor, despite the jet-aerator can achieve higher concentrations of hydrogen peroxide in other supporting electrolytes. This encourages the necessity for research in the efficient design of cells tailored for the PAA production.
- The lower value of the volume/anode ratio, the configuration anode-cathode and the higher ratio anode/cathode area explains the lower efficiency of the jet-based reactor.

Acknowledgements

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