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pH affects the spontaneous formation of H₂O₂ at the air–water interfaces

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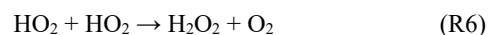
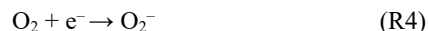
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ABSTRACT: Recent studies have shown that the air-water interface of aqueous microdroplets is a source of OH radicals and hydrogen peroxide in the atmosphere. Several parameters such as droplet size, salt and organic content have been suggested to play key roles in the formation of these oxidants. In this study, we focus on the effect of acidity on this spontaneous interfacial hydrogen peroxide formation of salt-containing droplets. Na₂SO₄, NaCl and NaBr bulk solutions, at the range of pH 4 to 9.5, were nebulized, using ultra high purity N₂/O₂ (80% / 20%), and H₂O₂ was measured in the collected droplets. All the experiments were performed in T = 292 ± 1 K and humidity levels of 90 ± 2 %. For Na₂SO₄ and NaCl, the H₂O₂ concentration was increased by ~40% under alkaline conditions, suggesting that OH⁻ enriched environments promote its production. When CO₂ was added in the ultra-pure air, H₂O₂ was observed to be lower at higher pH. This suggests that dissolved CO₂ can initiate reactions with OH radicals and electrons, impacting the interfacial H₂O₂ production. H₂O₂ formation in NaBr droplets did not display any dependence on the pH or the bath gas, showing that secondary reactions occur at the interface in the presence of Br⁻, which acts as an efficient interfacial source of electrons.

The air-water interface of small droplets exhibits high reactivity that can lead to the acceleration of some reactions and promotes others which are thermodynamically unfavored in the bulk.¹⁻⁴ This unique interfacial property has been attributed to the dynamical solvation behavior of solutes there that lead to large fluctuations of the external electrostatic potential,⁵⁻⁷ as well as the presence of an intrinsic local electric field.⁸

Near the air-water interface of aqueous droplets, water’s autoionization into hydronium cations (H⁺) and hydroxide anions (OH⁻) can lead to the spontaneous formation of OH radicals, solvated electrons and H atoms (R1,R2), due to these electrostatic forces.⁹⁻¹² Several studies have shown that hydrogen peroxide (H₂O₂) is formed, most probably via the mechanistic scheme described by reactions R3-R7. Two OH radicals can recombine to form H₂O₂ (R3).^{10,11,13-16} Solvated electrons in the presence of O₂ can form HO₂ radicals (R4,R5) that can lead to H₂O₂ production either via self-recombination (R6) and/or reaction with H atoms (R7), adding a second pathways to its formation.^{12,16}



Interfacial H₂O₂ production has been studied as a function of droplet size by Lee *et al.*; small droplets favour its formation due to their higher surface-to-volume ratio.¹³ Mehrgardi *et al.*, and Mofidfar *et al.*, showed that low nebulization flow rates enhance the H₂O₂ formation, as it results in higher droplets’ lifetimes in the reactor, which leads to water evaporation.^{15,17} Mofidfar *et al.*, also found that peroxide formation decreases at high humidity levels i.e., 95 % due to the water droplets dilution.¹⁷ Recently, we showed that halides such as Br⁻ and I⁻ enhance or suppress, respectively, H₂O₂ formation near the interfacial region, while ions like SO₄²⁻ and Cl⁻ do not affect the overall process.¹⁶

There is still limited knowledge on how droplets’ pH can affect spontaneous H₂O₂ formation. Bulk acidity of aerosols varies from 0 to 10 depending the origin, size, chemical composition and aging processes.^{18,19} In contrast, interfacial pH is matter of intense debate and certainly requires further investigation.²⁰⁻²² Recent simulations have showed that the interface might be very slightly more acidic than the bulk, but not super acid nor

alkaline.²³ Hence, we investigated the effect of (bulk) pH on interfacial H₂O₂ production occurring in Na₂SO₄, NaCl and NaBr containing droplets.

Aqueous nano-droplets were generated by nebulizing salt-containing solutions and introduced into a flow-tube reactor. The droplets were collected (after a reaction time of 10 seconds) as a bulk liquid phase, and the H₂O₂ produced in them was measured off-line, using a H₂O₂ analyzer (detailed experimental description in SI:Text S1-S2, Figures S1-S3). Bulk pH values were set between 4 and 9.5 due to limitations induced by the H₂O₂ monitor, as at lower or higher pH values, the catalase and other reagents used in the detection are reported to be destroyed.^{24,25} This was confirmed via control experiments (SI:Text S3, Figure S4).

Initially, experiments were performed using Na₂SO₄ and NaCl solutions in humidified 80:20 mix of high purity (UHP) N₂/O₂ bath gas. Figure 1 shows the pH measurements of the bulk and the collected droplets. The values were identical for all salts studied here. For both salts, the pH of the collected droplets was measured to be the same as for the bulk, within experimental uncertainty. The results of the H₂O₂ measurements produced in Na₂SO₄ and NaCl droplets are presented in Figure 2 (see Table S1). The error bars show the 2 σ uncertainty and include the estimated systematic uncertainties.

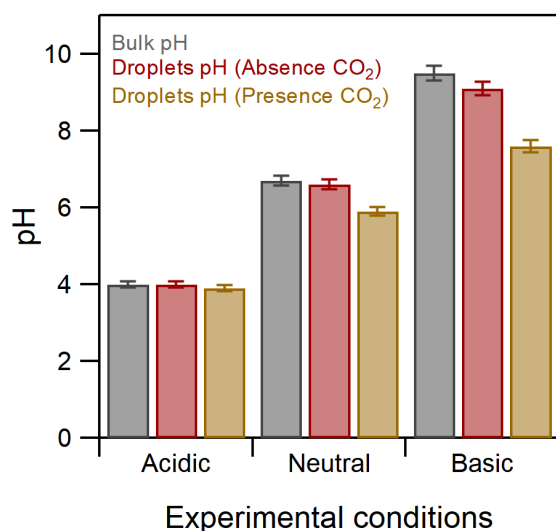


Figure 1. pH measurements of bulk solutions and collected droplets in 80:20 N₂/O₂ and compressed air.

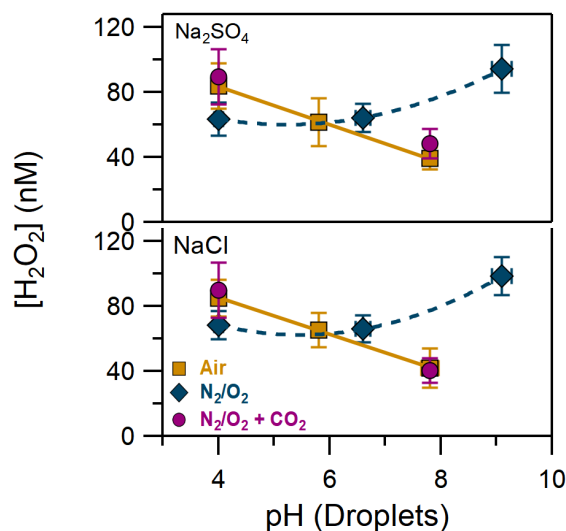


Figure 2. H₂O₂ produced in Na₂SO₄ and NaCl droplets (pH=4-9.5), in N₂/O₂, compressed air and N₂/O₂ with additional flow of CO₂ (see legend).

For both salts, in alkaline conditions H₂O₂ production was increased by 40% compared to acidic conditions, indicating that the OH⁻ enriched environment promotes oxidant formation. As the reaction that initiates the chemistry is proposed to be driven by the hydroxide anion (R1), a higher concentration of OH⁻ would lead to higher production of OH radicals and electrons and therefore H₂O₂. However, considering that the OH⁻ concentration was increased over 6 orders of magnitude in our measurements, the production of H₂O₂ did not display a strong dependence on hydroxide concentration, suggesting that it is not proportional to [OH⁻]. Recently, it has been suggested by de la Puente *et al.*, that interfaces are enriched with hydronium cations, while hydroxide ions are mainly underneath the interface²³. Tse *et al.*, also showed that hydroxide ion is repelled from the air-water interface.²⁶ Given that, the fraction of interfacial OH⁻ may be not the same as that in the bulk, in accord with our experimental observations.

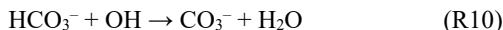
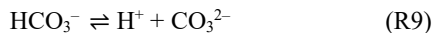
To investigate these interfacial reactions under atmospherically relevant conditions, the same experiments were also performed using compressed air, which contains ambient amounts of CO₂. Altering the bath gas gave significant differences compared to our observations with a pure N₂/O₂ mix. In the presence of CO₂, H₂O₂ production is decreased under alkaline conditions (i.e., the opposite trend to that seen in the absence of carbon dioxide, Figure 2). In addition, using compressed air, the pH of the collected droplets generated from bulk solutions of pH 6.7 and 9.5 was decreased by 12% and 20%, respectively, while for acidic conditions, it remained stable within $\pm 2\%$ (Figure 1). Since the major difference between the two different experimental conditions is the presence of CO₂ in the compressed air, we attribute the decrease of the pH seen in Figure 1 to the acidification of the droplets by dissolved carbon dioxide. To demonstrate that CO₂ is responsible for the trend observed in Figure 2, additional experiments were performed using a mixture of UHP N₂/O₂ and UHP CO₂ (Figure 2). CO₂ concentration was set at 400 ppm, simulating atmospheric levels. The addition of CO₂ in the N₂/O₂ mixture led to identical results to those obtained

using compressed air, verifying that the presence of CO₂ alters the chemistry.

CO₂ dissolution is favoured in alkaline conditions as the effective Henry's Law coefficient is higher by four orders of magnitude compared to acidic environment (SI: Text S4, Figure S5). In complete agreement with our observations, Cohen *et al.*, reported a decrease of pH of alkaline levitated droplets due to CO₂ uptake.²⁷ Li *et al.*,²⁸ have also reported that spraying alkaline solutions of phenolphthalein in air environment gave rise to decolouration of the droplets due to CO₂ dissolution.

The results displayed in Figure 2 indicate that under neutral conditions, H₂O₂ production was not affected by a change of the bath gas, while in acidic conditions we observed a small increase in the presence of CO₂. However, in alkaline droplets, the concentration of H₂O₂ became significant lower with added CO₂, implying that in such conditions, the enhancement of OH⁻ leads to the suppression of H₂O₂. Among the three different pH values we studied, H₂O₂ displayed the greatest sensitivity to the bath gas (with or without CO₂) only at high pH, where the dissolution of CO₂ is favoured. However, the decrease of H₂O₂ cannot be attributed to a decrease of pH due to dissolved CO₂, as the collected droplets are still alkaline (Figure 1) and the observed H₂O₂ is lower than that obtained under neutral and acidic conditions.

The relationship between pH and dissolved CO₂ is governed by the coupled equilibria (R8) and (R9). Under alkaline conditions, the main carbonaceous species that are present are HCO₃⁻ and CO₃²⁻ in a ratio of 0.8, as it is shown in Figure S6. HCO₃⁻ can act as an OH radical scavenger via reaction R10 ($k_{10} = 8.5 \times 10^6 \text{ M}^{-1} \text{ s}^{-1}$).²⁹ Although this process is slower than OH recombination by 3 orders of magnitude, based on bulk kinetics parameters, HCO₃⁻ concentration is expected to be at least 11 orders higher than that of the produced OH radicals, at pH 9.5 (SI: Text S5). Therefore, R10 can be considered as a competitive reaction, minimizing the contribution of OH recombination to the oxidant production.



In addition, CO₂, prior to its hydrolysis (R8 slow process, $k_8 \approx 4 \times 10^{-2} \text{ s}^{-1}$)³⁰, may react rapidly with the solvated electrons, via the competitive reaction R11 ($k_{11} = 7.7 \times 10^9 \text{ M}^{-1} \text{ s}^{-1}$).³¹ Solvated electrons play a key role in the H₂O₂ formation mechanism as they react with molecular oxygen to form O₂⁻. O₂⁻ reaction with hydronium cations leads to HO₂ formation, which via R6 and R7 produces H₂O₂. Therefore, in the presence of carbon dioxide, fewer electrons are available to react with the dissolved molecular oxygen, resulting in less H₂O₂. Both chemical reactions lead to a decrease in H₂O₂ formation, in excellent agreement with our results, showing that the reactions initiated by dissolved CO₂ participate in interfacial H₂O₂ production.

To explore further the role of CO₂, we performed experiments using NaCl solutions of bulk pH 4 and 9.5, varying the concentration of CO₂ from 0 to 800 ppm. Figure 3 presents the H₂O₂ production as a function of CO₂ concentration (see Table S2). In acidic conditions, H₂O₂ produced at the interface of NaCl droplets is seen to be independent of the CO₂ content, suggesting that the amount of the dissolved CO₂ is not sufficient to

affect its production. However, in alkaline conditions, H₂O₂ displayed a systematic decrease as CO₂ increases, which highlights the importance of the CO₂ chemistry and the involvement of reactions R8 to R11.

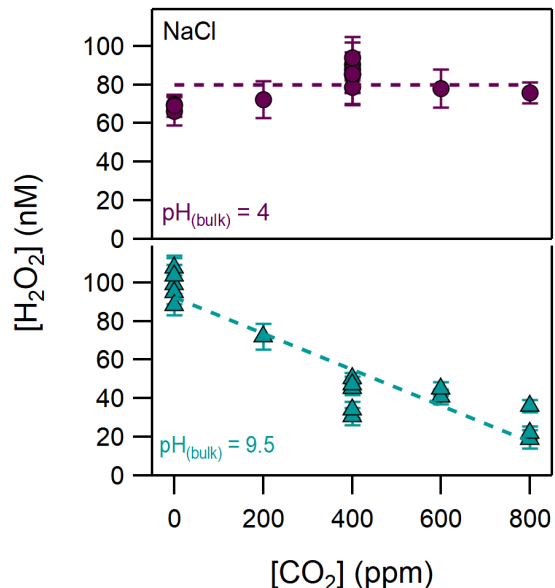


Figure 3. H₂O₂ produced in NaCl droplets as a function of the CO₂ concentration, at bulk pH of 4 and 9.5.

Finally, Figure 4 displays results of experiments were performed with NaBr droplets in both UHP N₂/O₂ and compressed air environments. Unlike the other salts, in the absence of CO₂, H₂O₂ remained stable, suggesting that the changes in the concentration of hydroxide anion have a negligible effect on its production. It was recently proposed that bromide anion (Br⁻), may undergo charge separation (Br⁻ ⇌ Br + e⁻) in the presence of the interfacial electric field,^{8,32} producing Br radicals and electrons (R12).¹⁶

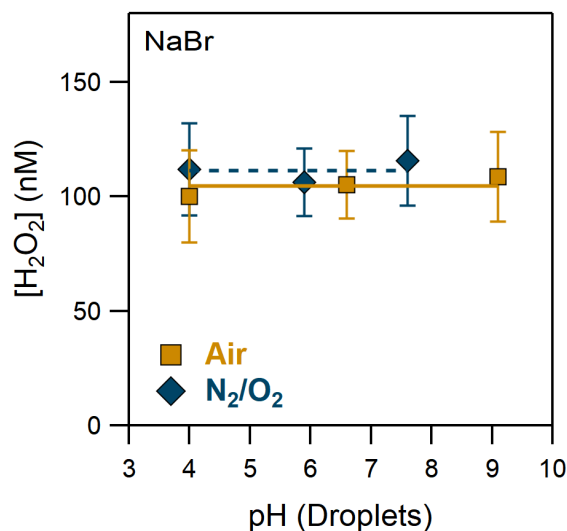


Figure 4. H₂O₂, produced in NaBr droplets (pH=4-9.5), in UHP N₂/O₂ and compressed air (see legend).

We suggest that in the presence of bromide anion the mechanism of H₂O₂ formation changes. The enhancement of solvated electrons near the interfacial region of NaBr droplets would promote H₂O₂ production via the reactions R4-R7, while interfacial OH⁻ (R1) and OH recombination (R3) contribute less to its formation. In addition, no dependence on the bath gas was observed in the range of our measurements. In the presence of CO₂, electrons can react with both carbon dioxide and oxygen. However, R11 does not seem to have an effect in peroxide production, due to the higher electron concentration. The presence of HCO₃⁻ eliminates further the OH recombination, and thus in both bath gases H₂O₂ is mainly formed via HO₂ initiated reactions (R6-R7).

Overall, these results suggest that aerosol pH can affect H₂O₂ production near the interfacial region. Sea salt aerosols' pH can be acidic or alkaline, depending on their ageing processes and their size. For example ammonia enriched environments and high relative humidity will result in almost alkaline droplets, while the uptake of gases such as HNO₃ and H₂SO₄ in sea aerosols will significantly decrease their pH.^{18,19} Therefore, the contribution of interfacial OH/H₂O₂ production occurring in marine droplets to the total tropospheric budget will depend on their environment and their composition. The H₂O₂ production mechanism seems to be more complex than thought initially. The results given here emphasize the need for further studies to elucidate the concentration of different species at droplets interface and bulk, as well as the inclusion of all the reactions that are involved to this interfacial process in atmospheric simulation models.

ASSOCIATED CONTENT

Supporting Information

Text S1 and Figure S1: Description of the experimental procedure, conditions and set-up, Figure S2: Size distribution measurements, Text S2 and Figure S3: H₂O₂ production as a function of nebulization flow, Text S3 and Figure S4: Bulk H₂O₂ measurements as a function of pH, Text S4 and Figure S5: CO₂ Henry's law constant as a function of pH, Figure S6: Speciation diagram of CO₂, HCO₃⁻ and CO₃²⁻ as a function of pH. Text S5: Calculations for OH radicals and HCO₃⁻ concentrations. Table S1: Experimental conditions and results for the H₂O₂ production measured in the collected droplets of Na₂SO₄, NaCl and NaBr. Table S2: H₂O₂ production measured in the collected droplets of NaCl, at pH 4 and 9.5 at different levels of CO₂.

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Notes

The authors declare no competing financial interests.

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