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Plasmachemical decolourisation of Bromothymol Blue by gliding electric discharge at atmospheric pressure

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Abstract

The colour of aqueous solutions of 3,3′-dibromothymolsulfonephtalein (Bromothymol Blue) fades when they are exposed to a gliding electric discharge, due to the nature of impinging plasma species at the liquid surface. For treatment times >10 min, alkaline BTB solutions turned from blue to yellow due to acidic effects and also fading resulting from both decolourisation and degradation. The addition of NaN_3 reduced the decolourisation rate from 99.1% without NaN_3 to 68.2% and lowered the pH to 6.7. In contrast, H_2O_2 increased the extent of decolourisation from 60.6% to 94.0% after 5 min treatment. Additionally, an increase in decolourisation rate of the plasma treated solutions' post-discharge was observed for the first time.

Keywords: 3,3′-Dibromothymolsulfonephtalein; Gliding discharge; Non-thermal plasma; Post-discharge

1. Introduction

Two main approaches are used to remove organic pollutants in waste waters namely, bio- and physicochemical-degradation. However, these processes often are inadequate with respect to the more drastic environmental regulations recently imposed. For instance, biodegradation of dyes is not efficient enough due to the presence of aromatic nuclei in colorants [1]. Among the available technologies, advanced oxidation processes (AOPs) are of special interest, as exemplified by UV induced, photochemical effects combined with H_2O_2/O_3 [2–7]. Electron-beam irradiation, TiO_2 photocatalysis and sonochemistry also rely upon the application of energy to aqueous solutions in order to produce highly active species, especially OH radicals [8]. In terms of new degradation techniques, electric discharge can be considered as a green source of reactive species that are able to degrade pollutant molecules; gliding discharge [9–13] is one of the most promising and efficient representatives of such technology.

This paper concerns the bleaching of Bromothymol Blue (3,3′-dibromothymolsulfonephtalein, or BTB) in aqueous solution by means of gliding electric discharge. Subsequent post-discharge phenomena were observed and the effects of chemical additives on dye degradation were also considered.

1.1. The gliding discharge (''glidarc'')

First proposed by Lesueur et al. [14] and by Czernichowski [15], the gliding arc (or ''glidarc'') was examined as a convenient source of quenched, non-thermal plasma in several laboratories, mainly for pollution abatement of gases [16–18].

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A gliding arc is an electric discharge in a high-intensity electric field formed between two or more diverging electrodes [14–18]. The discharge leads to the formation of positive and negative ions, photons, electrons and other chemically active species such as molecules and radicals. In gliding arc reactors, the gas flows between the electrodes at very high rates in order to maintain the quasi-thermal characteristics of the plasma. The gas flow, which is directed along the axis of the electrodes, gently moves the arc feet to the tip of the electrodes, so that the arc length increases and its temperature decreases, as does its energy when the arc is short-circuited by a new one. The resulting plasma is actually a quenched plasma, similar to non-thermal plasma, and operates at atmospheric pressure and at a (macroscopic) temperature which does not exceed 60 °C. For the present study, a circulating water flow controlled the temperature of the liquid target at 25 °C.

Aqueous solutions can be exposed to such plasma, so that chemical reactions may occur at the liquid/gas interface and the liquid target is thus subjected to the chemical properties of the impinging activated species present in the discharge. Many parameters such as the composition of the solution, the nature and flow rate of the working gas, the electrode material and shape, as well as the power supply voltage play important roles in the degradation of an organic pollutant and on the variation of the pH, conductivity and electrical potential of the solution.

The species present in the plasma phase are governed by the composition of the feed gas, the nature of the target solute (provided they are partly volatile) and by the discharge parameters. For example, a discharge burning in a mixture of N_2 and O_2 yields nitrogen oxides such as NO which are able to react at the liquid surface with suitable organic molecules.

1.2. Chemical properties of a gliding discharge

Recently, the humid air gliding discharge technique has been applied to the treatment of condensed matter; remarkable chemical properties can be evidenced in the condensed matter [17]: strong acidic and oxidising properties have been observed and quantified in aqueous solution [19–21]. These properties are directly related to the composition of the plasma gas. Molecular emission spectroscopy [21,22] showed the presence of NO and HO radicals [23] and molecular ions N_2^+ in the case of humid air plasma, in addition to other expected heavy (O_2 and N_2) and light (electrons, photons) species. Other species such as NO^+ were evidenced through their chemical action on selected solutes, such as urea and thio-urea [24].

The nature of the activated species depends on that of the feed gas, as mentioned: in the case of discharge in humid air, the activated species derive from N_2, O_2 and H_2O (Fig. 1) and, therefore, hydrogen peroxide, ozone or nitrogen oxides NO_x are expected [25], although the occurrence of water vapour is not favourable to the formation of ozone. Fig. 1 shows that the oxidising properties of the discharge are mainly due to OH radicals, while the NO radicals are considered as parent molecules for acid derivatives. However, the high standard potentials of the HNO_2/NO and NO_3^-/HNO_2 systems (i.e., 1.00 and 1.04 V vs. NHE, respectively) explain the oxidising character of the nitrate ion. The OH radical is one of the most powerful oxidising agents [i.e., E^0 (OH/H_2O) = 2.85 V vs. NHE] and is responsible for oxidation reactions, both by its own properties and derivatives and/or parent molecules H_2O_2 (E^0 (H_2O_2/H_2O) = 1.68 V/NHE):

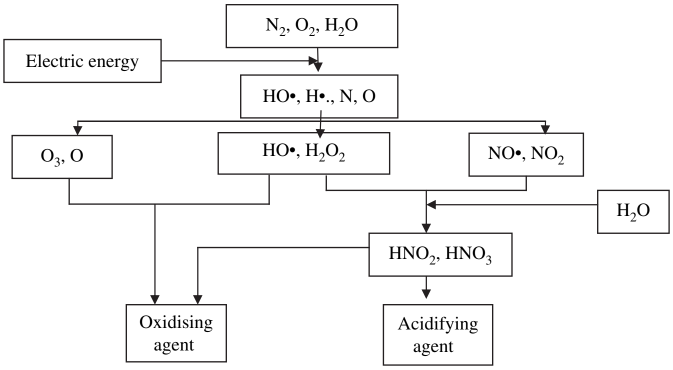

Fig. 1. Main molecular species in humid air plasma.

$$H_2O_2 \rightleftarrows 2OH \qquad (1)$$

Benstaali et al. [21] showed that OH radical was present in much higher density than NO radical in a plasma plume; several other species with high oxidative reduction potential are also formed in the discharge and confer a marked oxidising character to the plasma.

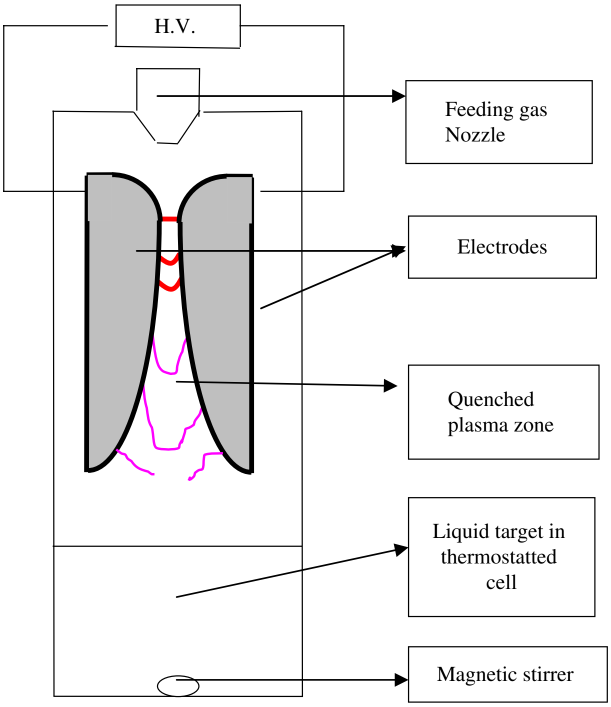

Fig. 2. Sketch of a gliding arc device.

2. Materials and methods

The experimental apparatus is shown in Fig. 2; it has been described previously [14,17,18] and largely used for pollutant abatement of gases [18]; the reactor was developed for the treatment of liquid effluents [26,27]. Compressed air passes through a distilled water-filled bubbling flask, becoming water-saturated before being injected in the reactor along the axis of two diverging electrodes at a controlled flow rate ($8.33\ \mathrm{L\,min^{-1}}$). The two electrodes were connected to an Aupem-Sefli HV transformer (9 kV; 50 Hz in open conditions; maximum intensity delivered 100 mA). When the circuit is connected, an electric arc forms between the electrodes at the minimum gap. The arc is pushed away by the feeding gas flow, sweeps along the increasing electrode gap and breaks in a large plasma plume as mentioned in Section 1.1. A new arc then appears and develops according to the same procedure. The arc is actually a thermal plasma; however, its length increases and the volume of the ionised channel also increases as the arc ‘‘glides’’ along the electrodes. The gas temperature then reduces so that the medium becomes a non-thermal plasma subsequent to the arc breaking. The resulting plasma that reacts at the liquid surface is a quenched plasma which has no noticeable thermal effect on solutions for short exposures.

The physics of the gliding arc have been detailed elsewhere [16,20,22]. The plasma plume ‘licks’ the liquid surface and allows chemical reactions to take place at the interface. The contact surface was increased by means of magnetic stirring of the target solution. The working parameters were optimised and the gas flow rate fixed at $Q = 500\ \mathrm{L\,h^{-1}}$. The dye solution (500 mL) was circulated through the reactor and exposed to the humid air plasma.

The dye (3,3′-dibromothymolsulfonephtalein, $C_{19}H_{10}O_5Br_2S$) is a complicated, three aromatic ring molecule. It was purchased from RAL and used without further purification. BTB is a commonly used acid–base indicator ($pK_a \approx 7$), with a transition from blue (basic medium, absorption peak at 617 nm) to yellow (acid medium, absorption peak at 433 nm). The initial alkaline pH of the dye solution (to ensure solubilisation) was adjusted using 1 M aq NaOH solution.

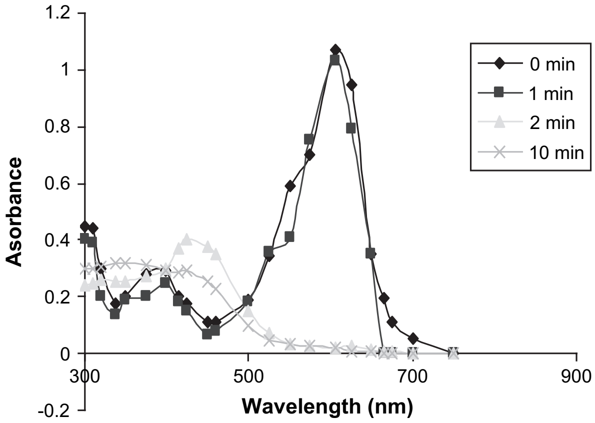

Fig. 3. Variation of absorbance spectra of basic BTB solutions with the exposure time t^* to the discharge (t^*(min) = 0–1–2–10).

A Jenway UV/vis spectrophotometer was used to measure dye absorbance at the λ_{max} of the acid form (433 nm) of the indicator in 1 cm cells (Fig. 3). A Techcomp UV/vis spectrophotometer was used to record the absorption spectra of acidic solutions as they were exposed to the discharge.

The extent of decolourisation (decolourisation factor, F_D) was calculated according to Eq. (2):

$$F_D = \frac{\mathrm{Absorbance}_{(\mathrm{initial})} - \mathrm{Absorbance}_{(\mathrm{treated\ for}\ t\ \mathrm{min})}}{\mathrm{Absorbance}_{(\mathrm{initial})}} \tag{2}$$

The conductivity was measured with an ATC Conductivity Conmet 1 and the pH and the electrical potential of the solutions were determined by means of a Microprocessor Bench pH/mV/°C Meter.

3. Results and discussion

As the multiplicity of dyes prevents the selection of a universally applicable chemical way to decolourise such compounds, we decided to employ a new technique, gliding discharge, to the decolourisation of several types of dyes dissolved in water. In this study, we focussed on BTB as a representative of one type of dyes.

3.1. Plasma bleaching of BTB without the addition of chemicals

Fig. 3 shows the evolution of the absorption spectrum of BTB solutions with the plasma treatment time t. The spectrum of the alkaline, untreated solution displayed two absorption bands at wavelengths slightly different from those cited in the literature, which may result from the solvent used: a weak peak around 385 nm (molecular form of BTB) and a large band around 605 nm (basic form) were observed. The 605 nm-peak intensity gradually decreased with increasing treatment time and completely disappeared for $t^* > 10$ min. The evolution of the spectra matches the colour change of the solution which first passed from blue to yellow via an intermediate green colour; this feature accounts for the expected acidification of the liquid solution. For longer treatment times, the pH tended to steady at 3.5 and the colour faded. The pH decrease (Fig. 4) was close to 7 units for a 10 min treatment.

At high pH, the proton concentration obeyed a mono-logarithmic variation with treatment time, which is similar to the titration plot of a strong base with a strong acid and which is characterised by an equivalent point t_{eq} [20,29]. The pH fall resulted from the formation of H_3O^+. The matching anions were identified to be NO_2^- and NO_3^- which resulted from the occurrence of NO in the gas phase. NO readily reacts in air to yield NO_2 which gives rise to the acidic species, HNO_2 and HNO_3 at the liquid/gas interface. The conductivity of the solution consequently increased [12] with treatment time t ($\chi = 22.645\,t^* + 101.85$).

The bleaching properties of the plasma treatment are thus directly related to its acidic nature. Another feature must be concurrently considered, namely the oxidative degradation of

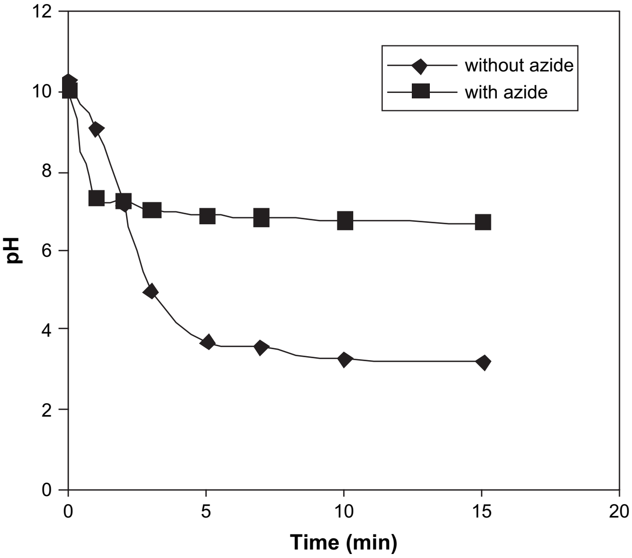

Fig. 4. pH evolution of the plasma treated BTB solutions against the treatment time t^* min (with and without added azide).

the dye, which is also related to acidity. This is confirmed by the formal E'^0 vs. pH plot which shows the acidity dependence of the solution potential [29]. These properties are mainly due to the occurrence of the OH radical which has a standard potential of the system E^0 (OH/H_2O) = 2.85 V/NHE. In addition, the high standard potentials of the HNO_2/NO and NO_3^-/HNO_2 systems (i.e., 1.00 and 1.04 V vs. NHE, respectively) also explain the oxidising effect of the nitrate ion.

Two matching phenomena account for the observed decolourisation and degradation processes. The interaction of plasma gas/BTB solution is a complex phenomenon and it is therefore important to study its kinetic in order to enlighten the main factor.

3.2. Plasma degradation of BTB without the addition of chemicals

Degradation of the dye was evidenced by means of complementary experiments performed on acidified dye solutions (initial pH 3.5 achieved using $HClO_4$). Absorbance measurements at the λ_{max} of the acid form of the dye show that the concentration of the solute molecule decreased with increasing treatment time t^* (Fig. 5). Additionally, the specific bands of the nitrite ions were observed in the range 340–400 nm with a shoulder around 302 nm; however, its conversion to NO_3^- in acidic medium remains conjectural.

Kinetically, (Fig. 6) the degradation of the dye obeyed an overall 1st order law, which was expected [27] for very dilute solutions (1.5×10^{-4} mol L^{-1}):

$$\frac{\mathrm{d}C}{\mathrm{d}t^*} = -kC, \quad \frac{\mathrm{d}(A - A_\infty)}{\mathrm{d}t^*} = -k(A - A_\infty)$$

The relevant constant, $k = 0.012$ mol min^{-1} (or 2×10^{-4} mol s^{-1}; $R^2 = 0.993$), was given by the slope of the linear plot of $\ln(A_{t^*} - A_\infty) = f(t^*)$ with $A_\infty = 0.636$.

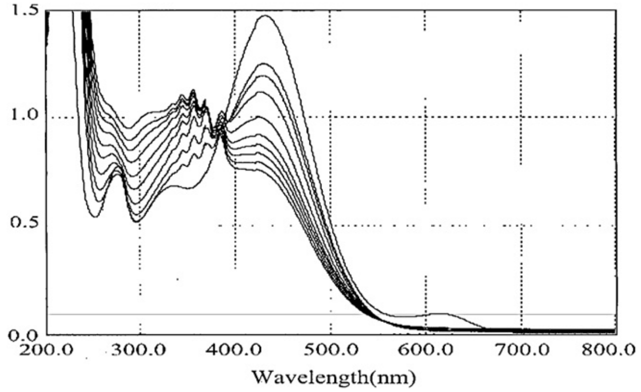

Fig. 5. Evolution of an acid BTB solution (1.5×10^{-4} mol L^{-1}) exposed to the discharge. Treatment times t^* (min): 0–10–15–25–45–65–85–105–125–145. Gas flow rate: 375 L h^{-1}; volume of liquid target: 200 mL.

3.3. Decolourisation kinetics in the presence of NaN_3

Selected chemicals were used in the plasma treatment of basicified BTB solutions; sodium azide (*caution*: explosively reactive and highly toxic – in water it forms the highly toxic gas HN_3: reaction with metals can result in very unstable and highly explosive metal azides) was first considered due to its strong reducing properties (which are related to the explosive character of some azides):

$$2HN_3 = 3N_2 + 2H^+ + 2e^- \quad (E^0 = -3.1 \text{ V/NHE})$$

This feature enables azide to degrade nitrous acid to N_2O (or its hydrated form $H_2N_2O_2$) slightly more easily than NO:

$$2HNO_2 + 4H^+ + 2e^- = H_2N_2O_2 + 2H_2O \quad (E^0 = 0.86 \text{ V/NHE})$$

$$NO + 6H^+ + 5e^- = NH_4^+ + H_2O \quad (E^0 = 0.836 \text{ V/NHE})$$

$$2NO + 4H^+ + 4e^- = N_2 + 2H_2O \quad (E^0 = 1.678 \text{ V/NHE})$$

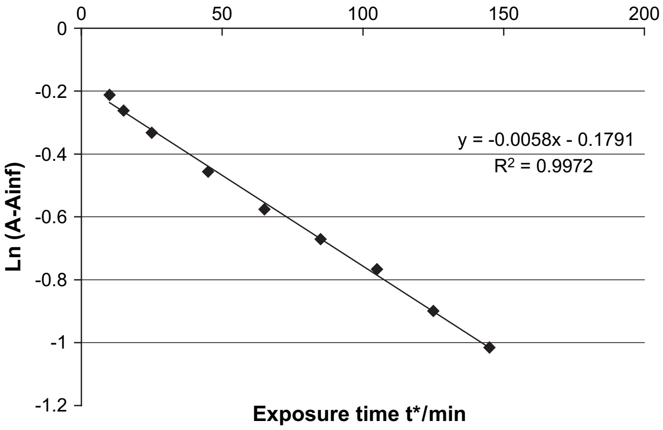

Fig. 6. Bleaching kinetics of acid BTB solutions exposed to the discharge for t^* min. The absorbance spectra are given in Fig. 5.

since the standard potentials [30] of the systems HNO_2/N_2O and NO/NH_4^+ are high enough and thermodynamically allow a complete reaction to take place. The relevant overall reaction is moreover mentioned [31]:

$$HN_3 + HNO_2 = N_2 + N_2O + H_2O \quad (\text{or } N_2 + H_2N_2O_2)$$

The matching overall reaction

$$10HN_3 + 2NO + 2H^+ = 2NH_4^+ + 15N_2 + 2H_2O$$

requires an acid medium to develop and the occurrence of protons at the surface of the liquid target. The direct reaction between HN_3 and NO:

$$4HN_3 + 2NO = 7N_2 + 2H_2O$$

concerns gaseous NO and solute HN_3; it is pH independent and may also participate in the destruction of NO.

The standard procedure in the absence of N_3^-, was

(i) the formation of NO in the discharge;
(ii) air oxidation of NO to NO_2 and the subsequent formation of nitrous and nitric acids (NO_2H and NO_3H) in the solution, and/or;
(iii) direct oxidation of NO by OH to yield the transient $HONO_2$ and, ultimately, nitric acid.

In the presence of azide the transient nitrous acid (or nitrite ions) was degraded, and the plasma-formed NO may react with HN_3 at the liquid surface, so that the formation of nitric acid was prevented or, at least, limited. The pH of the target (Fig. 4) was controlled by HN_3/N_3^- buffer, i.e., at a much higher pH than without azide, where the treated solution was buffered by HNO_2/NO_2^-. The decolourisation rate was initially very high and then reached a plateau, indicating that the bleaching reaction slowed, due to the acidity of the medium. Mineralization reactions require the breaking of numerous bonds and are thus usually less rapid than bleaching reactions [9]. The slight decrease in colour abatement (Fig. 7) for $t^* > 10$ min may be assigned to the evolution of intermediates or products.

Results relevant to the addition of NaN_3 are shown in Figs. 4 and 7. The absorbance of the treated BTB solutions was measured at the λ_{max} (605 nm); for a 10 min treatment, the decolourisation rate (expressed in % and measured at 605 nm) without NaN_3 was 99.1% and the solution turned to yellow, while the rate was only 68.2% with NaN_3 (1 g L^{-1}) and the solution remained green. Fig. 4 shows that the pH of the solution was close to 7 even for long exposure times, which may account for the observations.

These results confirm the ability of concentrated (1.5×10^{-2} M) NaN_3 solutions to limit the acidification of the medium as was previously shown in corona discharge [28]. This feature can be related to the basic character of N_3^- (pK_a (HN_3/N_3^-) = 4.7) and to its strongly reducing character (e.g., E^0 (N_2/HN_3) = −3.1 V) which, thermodynamically, allows the reagent to reduce NO to N_2 in acidic medium and thus prevents the formation of nitrous and nitric acids [29] or even that of NO.

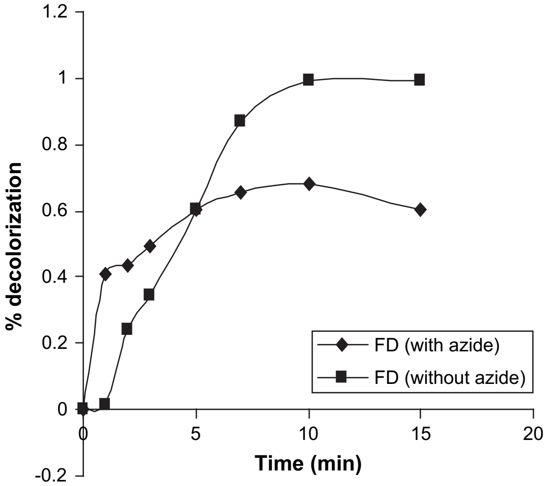

Fig. 7. Decolourisation rate (in %) of BTB with the treatment time t^* (min) with and without added azide.

The effect of the plasma gas on bleaching and mineralization of BTB was not considered herein because of the key role played by nitrogen containing compounds in the process. We know that dry gases induce only limited change in acidity; hence only the oxidising effect of the oxygenated species would be considered. A significant difference has been observed between humid air and humid oxygen, in the case of alkaline cyanide degradation, largely in favour of air [32]. In contrast, no noticeable pH effect was observed using a confined gliding arc reactor [33], except for the expected larger production of H_2O_2 in pure oxygen and the lower production of nitrates in pure nitrogen compared to air.

3.4. Decolourisation kinetics in the presence of H_2O_2

The effect of added hydrogen peroxide on the decolourisation process of BTB solutions was investigated (Fig. 8). The

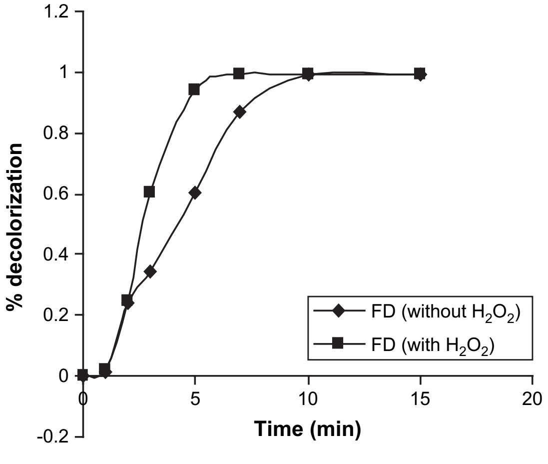

Fig. 8. Variation of the decolourisation rate (in %) of BTB against the treatment time t^* (min) with and without adding H_2O_2.

initial concentration of H_2O_2 was 10^3 ppm and the pH was adjusted to 9.25. The increase in the F_D values upon addition of hydrogen peroxide was appreciable; decolourisation rates of the basic forms of the dye were 60.6% (with) and 94.0% (without) added H_2O_2 after 5 min plasma treatment. As shown in Eq. (1), the photo-dissociation of H_2O_2 gives rise to OH radicals. This decolourisation process is mainly ascribed to the reaction of hydroxyl radicals with the solute, since the reaction of aromatic compounds with hydrogen peroxide is very slow [34]. Adding H_2O_2 may increase the concentration of active OH and thus accelerate the degradation rate.

Another explanation for the degradation of the dye may involve the occurrence of a transient species. Nitrite ions form in aqueous solutions exposed to discharge in air, and hydrogen peroxide has also been identified. These species may react to form an active intermediate:

$$NO_2^- + H_2O_2 \rightarrow ONO_2^-$$

In moderately acidic medium, the reaction:

$$HNO_2 + H_2O_2 \leftrightarrow HOONO + H_2O$$

can be considered. The peroxonitrite formed is a strong oxidant which may react with OH radicals at the dye molecule; this could explain the synergistic effect of the addition of hydrogen peroxide on decolouration reaction rate (Fig. 8).

3.5. Post-discharge phenomena for H_2O_2 containing dye solutions

Temporary, post-discharge phenomena refer to the self-evolution of the plasma treated solution when the discharge is switched off and no extra energy added, such as light or heat. Although this has been observed for other compounds, it has not hitherto been reported for the plasma treatment of BTB solutions. The H_2O_2 containing BTB solutions were first exposed to discharge ($0 < t^* < 10$ min) and were then kept for an incubation time t out of the discharge, in the dark and at ambient temperature. Under these conditions, a noticeable evolution of the solution was seen, as it continued to be bleached. Fig. 9 shows such evolution by means of the decolourisation rates relevant to selected treatment times and calculated for 30 min, 1 or 2 weeks incubation time. For long incubation times, such as for 1 or 2 weeks, the plots overlap. Since the lifetime of OH radicals is short (of the order of a few nanoseconds), these radicals cannot be considered to be directly responsible for the observed decolourisation. The increase in decolourisation during post-discharge can thus be ascribed to dissolved hydrogen peroxide and to nitrites. After 1 week incubation, there was no more H_2O_2 or nitrite present in solution, which explains why the plots overlap.

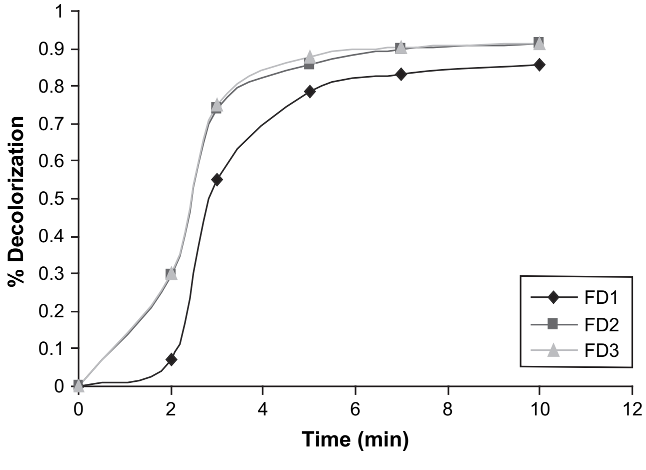

Fig. 9. Variation of the decolourisation rate against post-discharge time t (for various treatment times t^* FD1 = 30 min, FD2 = 1 week, FD3 = 2 weeks).

4. Conclusions

Dye decolourisation was rapid and developed within 10 min. Adding sodium azide to the dye solution limits the reduction in pH and thereby, limits the decolourisation process. The presence of added hydrogen peroxide markedly increases decolourisation rate. Post-discharge phenomena increase decolourisation by 10% in the absence of discharge; two stages in dye removal are then observed namely a rapid step ascribed to the hydroxyl radical OH and a slower one, attributable to hydrogen peroxide, H_2O_2.

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