Degradation of DBPs’ precursors in river water before and after slow sand filtration by photo-Fenton process at pH 5 in a solar CPC reactor

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**Abstract**

The generation of disinfection by-products during water treatment can be controlled by reducing the levels of precursor species prior to the chlorination step. The Natural Organic Matter (NOM) is the principal organic precursor and conventional removal of pollutants such as coagulation, flocculation and filtration do not guarantee the total NOM removal. In this study the degradation of NOM model compounds (dihydroxy-benzene) as well as the removal of NOM from river water via photo-Fenton process in a CPC solar photo-reactor is presented. The effect of solar activated photo-Fenton reagent at pH 5.0 before and after a slow sand filtration (SSF) in waters containing natural iron species is investigated and the details reported. The results showed that the total transformation of dihydroxy-benzene compounds along a mineralization higher than 80% was obtained. The mineralization of the organic compounds dissolved in natural water was higher than in Milli-Q water, suggesting that the aqueous organic and inorganic components (metals, humic acids and photoactive species) positively affect the photocatalytic process. When 1.0 mg/L of Fe³⁺ is added to the system, the photo-Fenton degradation was improved. Therefore the photo-Fenton reagent could be an interesting complement to other processes for NOM removal. Comparing the response of two rivers as media for the organic compounds degradation it was observed that the NOM photo-degradation rate depends of the water composition.

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**1. Introduction**

The Natural Organic Matter (NOM) present in water bodies is a complex mixture of organic substances such as humic and fulvic acids, proteins, lipids, hydrocarbons and amino-acids. NOM acts as an important precursor of disinfection by-products (DBPs) reacting with the chlorine used in the disinfection stage. It is known that the formation of DBPs highly depends on the water NOM composition and concentration (Singer, 1999). The trihalomethanes (THMs) and the haloacetic acids (HAAs) represent the two largest groups of DBPs produced. These DBPs affect the human health (Dunnick and Melnick, 1993; Yang, 2004), thus they are strictly regulated. The allowable concentration levels of total THMs and HAAs are 80 and 60 μg/L, respectively (USEPA, 2006). These values are difficult to reach as NOM has been reported to form concentrations of THMs as high as 90 μg/THM/mg of total organic carbon (TOC). The coagulation and flocculation...
(classical processes) are widely used to treat large concentrations of NOM. These processes typically remove 50–80% of dissolved organic carbon (DOC) but they are limited when trying to TOC levels of less than 1.0 mg/L (Murray and Parsons, 2004).

In developing countries drinking water production for small communities often consists of chlorination without a previous treatment. Slow sand filtration system (SSF) is frequently used. This system is inexpensive, reliable, and also useful for urban areas with better infrastructure, offering an alternative to current high rate filtration systems, which tend to be more sensitive to the system variations and require sophisticated controls up to the disinfection stage (Craun, 1993). However, SSF and classical processes are unable to decrease the aqueous TOC up to levels that prevent DBPs production above the standard level.

In order to minimize DBPs production, Advanced Oxidation Processes (AOPs) could be an alternative for NOM degradation (Murray and Parsons, 2004; Goslan et al., 2006; Liu et al, 2008; Kleiser and Frimmel, 2000; Wang et al., 2000; Uyguner and Bekbolet, 2005) or to replace chlorination as a disinfecting step (Hoffman et al., 1995; Rincon and Pulgarin, 2003, 2004, 2006, 2007). The AOPs are characterized by the presence of highly reactive hydroxyl radicals (‘OH), which are suitable for rapid and indiscriminate reaction with a great number of organic compounds inducing its almost total mineralization (Fukushima et al., 2001). Among the processes that can be used to generate hydroxyl radical, the homogeneous catalysis (Fukushima et al., 2001). The most accepted mechanism of H2O2 decomposition in acid homogeneous solution, involves the formation of hydroxyl radicals (‘OH) (Gallard and De Laat, 2000). The ‘OH radical, once in solution attacks almost every organic compound. The Fe3+ regeneration can follow different paths (Rincon and Pulgarin, 2006):

\[
\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \text{HO}^- + \cdot\text{OH} \quad (1)
\]

\[
\text{Fe}^{3+} + \text{H}_2\text{O} + h\nu \rightarrow \text{Fe}^{2+} + \text{H}^+ + \cdot\text{OH} \quad (2)
\]

The most accepted mechanism of H2O2 decomposition in acid homogeneous solution, involves the formation of hydroxyl radicals (‘OH) (Gallard and De Laat, 2000). The ‘OH radical, once in solution attacks almost every organic compound. The Fe3+ regeneration can follow different paths (Rincon and Pulgarin, 2006):

\[
\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \cdot\text{OH} + \cdot\text{OH} \quad (3)
\]

\[
\text{Fe}^{3+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{2+} + \text{HO}_2^- + \text{H}^+ \quad (4)
\]

\[
\text{Fe}^{2+} + \cdot\text{OH} \rightarrow \text{Fe}^{3+} + \cdot\text{OH} \quad (5)
\]

\[
\cdot\text{OH} + \text{H}_2\text{O}_2 \rightarrow \text{HO}_2^- + \text{H}_2\text{O} \quad (6)
\]

\[
\text{Fe}^{3+} + \text{HO}_2^- \rightarrow \text{Fe}^{2+} + \text{H}^+ + \text{O}_2 \quad (7)
\]

\[
\text{Fe}^{3+} + \text{O}_2^- \rightarrow \text{Fe}^{2+} + \text{O}_2 \quad (8)
\]

Fe2+ + HO2−→Fe3+ + HO2− (k = 1.2 × 106 L/mol s) (9)

In the photo-Fenton process (Eq. (2)), the Fenton reaction rates are strongly increased by irradiation with UV–vis light (Sun and Pignatello, 1993; Bandara et al., 1996; Zepp et al., 1992). The positive effect of irradiation on the degradation rate is due to the photo-chemical regeneration of ferrous ion (Fe2+) by photo-reduction of aqua-complexes of ferric ions, which concomitantly leads to additional ‘OH generation.

The ferrous ion generated in solution reacts with H2O2 yielding a second ‘OH radical and ferric ion (Eq. (3)), and the cycle continues. The main advantage of the photo-Fenton reagent is the light absorption up to a wavelength of 600 nm (35% of the solar irradiation). The depth of light penetration is high and the contact between pollutant and oxidizing agent is close in homogeneous solution (Bauer et al., 1999).

Fenton and photo-Fenton processes have been used with great success to treat a wide variety of contaminants at laboratory scale (Fallmann et al., 1999; Herrera et al., 1998; Kiwi et al., 1994; Rodriguez et al., 2002) and using reactors based on the use of natural sunlight (Rodriquez et al., 2005). Nevertheless in some cases, these studies have been made in Milli-Q water spiked with different iron salts (FeCl3, FeSO4, ferroxyalates) (Lipczenia-Kochany, 1991; Pignatello, 1992).

Other studies have been performed to treat different types of agro-industrial wastes with the aim to remove phenolic compounds using a Fenton and photo-Fenton reaction, but the pH was always adjusted to 3.0 (Rodriguez et al., 2005; Gernjak et al., 2003) and iron added in different concentration.

The treatment of commercial humic acid and NOM rich waters with Fenton and photo-Fenton processes were studied, at pH value near 4.0 using 5.65 mg/L of iron and 17 mg/L of hydrogen peroxide. These processes achieve greater reduction of absorbance at 254 nm and DOC compared to coagulation (Murray and Parsons, 2004). In spite that pH 3.0 is generally used along the Fenton reagent, the reduction of 254 nm absorbance and DOC was found to be insensitive in the pH range 3.0–7.0 (Goslan et al., 2006).

The aim of this work was to study the degradation via photo-Fenton of several NOM model compounds (dihydroxy-benzene) in river waters at pH 5.0 containing natural iron. Additional experiments were carried out to evaluate the beneficial effect on the dihydroxy-benzene compounds removal of the addition of 1.0 mg/L of Fe3+. Finally, NOM removal in the two types of river waters showed the feasibility of photo-Fenton treatment to abate NOM. The experiments were carried out in a CPC solar photo-reactor.

2. Experimental details

2.1. Reagents

Hydrogen peroxide 35% (Riedel-de-Haën), FeCl3·7H2O 98% (Merck) and sodium bisulphite (Fluka) were used as received. The pH was adjusted using H2SO4 0.1 M. Solutions of catechol, resorcinol and hydroquinone (Fluka > 99%) were prepared with both, Milli-Q water (18.2 MΩ cm) and water coming from
natural sources (Pance and Meléndez rivers near from Cali-Colombia), immediately prior to irradiation.

The Pance and Meléndez rivers are drinking water source for many communities (i.e. “El Retiro” and “La Sirena”), which use a SSF as pre-treatment system. Sampling was carried out before (raw water) and after SSF without chlorination. Chemical characteristic content of the waters used is reported in Table 1.

2.2. Analytical methods

The concentration of dihydroxy-benzene compounds was measured by HPLC equipment (HEWLETT–PACKARD series 1100). A column C18 Nova-Pak with particle size 4 µm; 3.9 mm i.d. and 150 mm length was used. The mobile phase utilized was methanol–water (20:80 v/v) with a flow of 1.0 mL/min. The total organic carbon (TOC) was measured using TOC analyzer SHIMADZU 5050. UV absorbance of water samples at 254 nm (UV254) was measured using a Shimadzu UV 160A spectrophotometer. The pH was measured with a pH-meter Metrohm 827 pH-lab using a glass electrode. The color and turbidity measurements were carried out following Standard Methods (APHA, 2001a,b).

2.3. Photocatalytic procedures

Samples were taken before and after slow sand filter (SSF) and spiked with dihydroxy-benzene compounds (model compounds of NOM). Experiments were carried out with and without addition of 1.0 mg/L of Fe3+. Dihydroxy-benzene compounds: catechol, resorcinol and hydroquinone, were chosen as NOM model because they are present in the natural waters resulting from the degradation of organic matter. The experiments were carried out under sunlight at Cali-Colombia between June and October of 2005 using a compound parabolic concentrator (CPC). The photo-reactor consists of six Pyrex® tubes (1.5 m length; 3.2 cm of internal diameter and 3.6 cm external diameter) placed over reflective surface of anodized aluminum and inclined 3° that correspond to the latitude of the location site. Twenty liters of studied river waters with and without dihydroxy-benzene compounds were continuously circulated through the reactor. The illuminated volume photo-reactor was 8.0 L and the recirculation flow rate was 9.1 L/min. The samples were taken from the reactor at various intervals and used for pH, H2O2 and TOC measurements. When the samples were not measured immediately after taking the sample out of the reactor the reaction was stopped with sodium bisulphite.

2.4. Evaluation of solar radiation

A global UV radiometer (ACADUS S2004 with a controller LS-3300) mounted at the same angle as the CPC reactor (3°, Cali-Colombia), provide data in terms of incident WUV/m². This gives an estimation of the energy reaching any surface at the same position with regard to the Sun. With Eq. (10), combination of data from different daily experiments and their comparison with other photocatalytic experiments is possible (Gernjak et al., 2003).

\[ Q_{UV,n} = Q_{UV,n-1} + \Delta t_n UVG_n [A_{CPC}/V_{TOT}], \]

where \( t_n \) is the experimental time of each sample, \( V_{TOT} \) the volume of reactor (20 L), \( A_{CPC} \) the illuminated surface of collector (0.905 m²), \( UVG_n \), the average \( UVG \) (UV global radiation) during \( \Delta t_n \) and \( Q_{UV,n} \) is the accumulated energy (per unit of volume, kJ/L) incident on the reactor for each sample taken during the experiment.

3. Results and discussion

3.1. Optimum H2O2 concentration

In order to determinate the suitable H2O2 concentration for hydroquinone, catechol and resorcinol (mixture) photodegradation, several experiments were carried out in Milli-Q water. The degradation rate was observed to increase with H2O2 concentration. A reduction of the pollutants >90% was reached with H2O2 concentration up to 60 mg/L (1.76 mM). Above this value the oxidation rate decreases.

Some reports (Chaly et al., 2001; Kajitvichyanukul and Suntronvipart, 2006; Moon et al., 1991) show that this trend is probably due to both auto-decomposition of H2O2 into oxygen and water (Eq. (11)), and the recombination of *OH. The excess of H2O2 will react with *OH radical (Eq. (12)) competing with the organic matter and consequently reducing the efficiency of the photo-Fenton process.

\[ 2H_2O_2 \rightarrow 2H_2O + O_2 \]  

(11)

\[ H_2O_2 + *OH \rightarrow H_2O + *O_2H \]  

(12)

3.2. Effect of river water matrix on photo-Fenton mineralization of dihydroxy-benzene compounds after addition of 1.0 mg/L of Fe3+

Fig. 1 shows the TOC abatement of an equimolar mixture of catechol, resorcinol and hydroquinone. The mineralization rate is higher in the Pance River than in Milli-Q water. With a accumulated energy of 4.0 and 11.0 kJ/L, values of TOC of 7.0
and 4.5 mg/L were reached in Pance River waters (Fig. 2, trace –○–); whereas in Milli-Q water (Fig. 2, trace –□–), with equal amounts of accumulated energy, 12.0 and 6.0 mg/L of TOC were reached. The steep mineralization rate in the first stage of treatment in river waters compared to Milli-Q water is due to the formation of photoactive intermediaries (i.e. complexes), which contributes to mineralization by photo-assisted electron transfers. Besides, in river waters, the presence of metallic ions as Fe$^{3+}$ (Table 1) photo-sensitizers allows visible light absorption producing singlet oxygen (O$_2$), H$_2$O$_2$ and •OH that attack the dihydroxy-benzene compounds. The humic acids, also presents in natural water, can contribute to the transformation of the organic compounds through the formation of complexes with Fe(III) (pH 3.0 and 5.0), increasing the amount of photoactive Fe(III) and therefore the efficiency of the photo-Fenton process (Vione et al., 2004). In addition, humic acids themselves can photo-degrade through formation of complexes with Fe(III) (Fukushima et al., 2001).

3.3. Effect of pH on photo-Fenton mineralization and degradation of dihydroxy-benzene compounds in river water after addition of 1.0 mg/L of Fe$^{3+}$

Fig. 2 shows that at pH 5.0, the degradation rate obtained for hydroquinone (as for catechol and resorcinol not shown here) was similar that obtained at pH 3.0 (discontinuous lines). However, TOC decay tends to be slower at pH 5.0 than at pH 3.0.

Fig. 1 – Catechol, resorcinol and hydroquinone mixture (each component has an initial concentration around 10 mg/L) mineralization using Milli-Q (–□–) and river (–○–) water. Recirculation flow in CPC solar photoreactor: 9.1 L/min; pH 5.0; [Fe$^{3+}$] = 1 mg/L; initial [H$_2$O$_2$] = 60 mg/L. Dark control (Fenton process) for samples (–●–) that corresponds to 15, 30, 60, 120, 180, 240 and 300 min of treatment. Real time: from 10:00 to 16:00 hours.

Fig. 2 – Catechol, resorcinol and hydroquinone mixture photo-Fenton degradation at pH 3.0 (–□–) and 5.0 (–○–) using water from Pance River taken before SSF. Recirculation flow in CPC photo-reactor = 9.1 L/min; added [Fe$^{3+}$] = 1 mg/L; initial [H$_2$O$_2$] = 60 mg/L. Only hydroquinone is shown. Catechol and resorcinol also present in the starting mixture, exhibit similar degradation rates. Initial TOC ~ 30 mg/L. Real time: from 10:00 to 16:00 hours.

Murray and Parsons (2004) evaluated the optimal pH for removing NOM from organic rich waters. The optimal pH was around 3.0 but at pH 5.0, only a little decrease of the overall performance was observed.

In strongly acidic solution, Fe(III) exists as the hexa-aquo ion, Fe(H$_2$O)$_6^{3+}$. As pH increases, this ion undergoes extensive hydrolysis (Eq. (13)), depending on the counter-ion, ionic strength, and total iron concentration, ending in precipitation of amorphous ferric oxyhydroxides.

\[
\text{Fe}^{3+} + \text{Fe(OH)}^2^+ \rightarrow \text{Fe(OH)}_2^+ \rightarrow \text{Fe(OH)}_3^+ \rightarrow \text{other polynuclear species} \rightarrow \text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O(s)} \quad (13)
\]

However, keeping the total iron below $1 \times 10^{-4}$ M in solution with pH values around 5.0 (in this work the iron concentration is around $1.8 \times 10^{-4}$ M), avoids a fast hydrolysis of Fe$^{3+}$ and therefore the precipitation of iron oxides (Pignatello et al., 2006).

In addition, at pH 5.0, species like Fe(OH)$_2^+$ are present (Gallard et al., 1999) in higher concentration than other more photoactives forms like Fe(OH)$_3^+$ and Fe$^{3+}$ and photo-Fenton process can be carried out in this weaker acidic conditions (Faust and Hoigne, 1990).

3.4. Degradation of dihydroxy-benzene compounds in river water without Fe$^{3+}$ addition

Fig. 3a shows that addition of H$_2$O$_2$ at river water (trace –○–) under sunlight (6.0 kJ/L) leads to 50% of mineralization. Low
concentration of iron (Table 1) and other metallic species present in the river waters can act as catalyst and contribute to degradation process. In Milli-Q water, the addition of H$_2$O$_2$ (Fig. 3b, trace --), leads to degradation efficiencies much lower than those observed in river waters (Fig. 3a, trace --). When 1.0 mg/L of Fe$^{3+}$ is added to complete the photocatalytic action of photoactive Fe-species naturally present in river water, the photo-Fenton process was substantially favored (Fig. 3a, trace --), reaching 80% of mineralization with 6.0 kJ/L of accumulated energy. H$_2$O$_2$ was completely consumed during the treatment.

3.5. Photo-Fenton mineralization of dihydroxy-benzene compounds mixture in Pance and Meléndez river waters before and after SSF with and without Fe$^{3+}$ addition

Results of experiments using Fenton and photo-Fenton processes to degrade dihydroxy-benzene compounds dissolved in Pance River water taken before and after SSF, in the presence of H$_2$O$_2$ (60 mg/L), with and without addition of Fe$^{3+}$ are presented in Fig. 4. Photo-mineralization in presence of H$_2$O$_2$, but without addition of Fe$^{3+}$, was significant in both cases leading to 57% TOC removal (final TOC ~15 mg/L) in water taken before SSF, and 65% (final TOC ~9.0 mg/L) in samples taken after SSF for 6.0 kJ/L of accumulated energy. This is due to (a) natural TOC present in Pance River is higher before than after SSF, (b) the lower turbidity of the samples taken after SSF (see Table 1) allowing a greater light penetration and leading to a higher photocatalytic activities in spite of the presence of a smaller iron concentration, and (c) microorganisms, organic and inorganic substances present in higher concentration in non filtered water consume a higher number of OH radicals generated in photo-Fenton process. These results demonstrate that water with low turbidity and containing natural photoactive species as Fe-complexes can be treated for their organic matter abatement via Fenton and photo-Fenton processes.

Fig. 5 shows that the degradation efficiencies of dihydroxy-benzene mixture dissolved in Meléndez River water are similar to those observed in Pance River water. Higher mineralization rates were observed for samples taken after the degradation compared to the values taken before SSF within an accumulated energy of 3.0 kJ/L. Thereafter, TOC lower than 3.0 mg/L was reached for both filtered and no filtered water with 7.0 kJ/L of accumulated energy. Lower values of turbidity and organic matter in waters of Meléndez River in comparison with Pance River (Table 1) would favor a higher light penetration in the solution and therefore the contact with the catalyst, which are in both sources at the same concentration rank. Nevertheless, it is not possible to assure that this is the most influencing factor because studies to evaluate the specific and dissociated effect of each physical-chemical parameter were not carried out in this work.

Degradation of dihydroxy-benzene compounds mixture without addition of Fe$^{3+}$, in the presence of H$_2$O$_2$ is reported in Fig. 5 (discontinuous lines) confirming that organic and inorganic components of the Meléndez River favor the photo-Fenton process to the same extend (TOC decay of 50% with an accumulated energy of 7.0 kJ/L) than the process observed for waters of the Pance River.

3.6. Removal of NOM from Pance and Meléndez rivers by Fenton and photo-Fenton processes

In order to evaluate the degradation of NOM in both rivers without the addition of dihydroxy-benzene compounds,
Additional experiments were realized with an initial H$_2$O$_2$ concentration of 20 mg/L (0.59 mM) with and without addition of Fe$^{3+}$ (1.0 mg/L). The light/H$_2$O$_2$ and light/H$_2$O$_2$/Fe$^{3+}$ systems were evaluated in the removal of UV$_{254}$ and TOC. The absorbance at 254 nm was measured to follow the degradation of chromophores.

Fig. 6 shows that on Melendez and Pance rivers, reductions of UV$_{254}$ were higher than reduction of TOC at 6.5 kJ/L of accumulated energy. Higher percentage of UV$_{254}$ compared to TOC removal means significant structural changes (simple break-up) without leading to complete mineralization of the large aromatic structures. Similar features were reported by Huang et al. (2008) and Liu et al. (2008) in TiO$_2$ photo-assisted degradation of humic acid.

For the light/H$_2$O$_2$ system (based on photocatalyst naturally present in water) considerable percentage of UV$_{254}$ and TOC removal were observed. With an accumulated energy of 6.5 kJ/L, UV$_{254}$ a TOC reduction of 68% and 55% were, respectively, obtained for samples taken from the Pance River before SSF. UV$_{254}$ and TOC reduction increased to 80% and 62%, respectively, with samples taken after SSF. Goslan et al. (2006) also reported UV$_{254}$ and DOC removal of 95 and 78% in presence of 2.0 mM of H$_2$O$_2$ and using a UV-C (22 J/cm$^2$).

Similar trend was observed for samples from Meléndez River (Fig. 6b) with the same dose applied. UV$_{254}$ removal increased from 68% to 89% for samples before and after SSF, while TOC removal did not increased and a final TOC of 1.5 mg/L was observed starting with a concentration of 3.5 mg/L.

When a 1.0 mg/L of Fe$^{3+}$ was added to water (light/H$_2$O$_2$/Fe$^{3+}$ system) UV$_{254}$ and TOC removal increased significantly in all cases, obtaining a higher UV$_{254}$ than TOC removal. For this system, TOC values below 1.0 mg/L were reached, which will presumably permit limit THMs formation above normative during a subsequent chlorination.

Previous work has demonstrated that most of TOC removed by photocatalytic process corresponds to hydrophobic components, which predominate in the NOM from raw water (Buchanan et al., 2005; Liu et al., 2008). This fraction is more aromatic than the hydrophilic fractions and has high reactivity towards oxidation interacting with other oxidative species generated in solution. Besides, photo-Fenton process is more suitable for the removal of low molecular weight organics between 0.5 and 4 KDa (Murray and Parsons, 2004). This is an interesting property as low molecular weight compounds are difficult to remove during conventional treatment processes and hence more likely to generate a higher formation potential THMs (Murray and Parsons, 2004).

The TOC removal is not complete due possibly to the presence from the beginning, or the generation during the
process, of by-products recalcitrant to the treatment. Buchanan et al. (2005) demonstrated that the hydrophilic fraction of the NOM, present in the raw water (around 1.0 mg C/L), would be refractory leading to slow mineralization rates. This fraction denoted as NEU (hydrophilic neutral fraction), include short-chain aliphatic amines, alcohols, aldehydes, esters, ketones; C₅ aliphatic amides; polyfunctional alcohols, carbohydrates; cyclic amides; and polysaccharides.

4. Conclusions

Population of many regions in developing countries drinks water from superficial source after chlorination directly without previous physical treatment leading to high THMs formation risk. This work showed that the addition of 20 mg/L of H₂O₂ (which is consumed during the treatment) at pH 5.0 (closer to natural) and using direct solar radiation lead to a considerable decrease of TOC (50–60%). This beneficial impact will be subsequently reflected in low chlorine demand and consequently in lower THMs formation. If 1.0 mg/L of Fe³⁺ is added, these percentages are increased (70–90%), in some cases reaching values of TOC below 1.0 mg/L, which could result in significantly lower THMs formation. When SSF is applied before chlorination, NOM removal is almost complete and the remaining TOC is suitable to prevent THMs formation. The beneficial effect of photoactive natural components present in the natural water on the photo-Fenton process is shown by the more efficient mineralization of organic matter.

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