



UNIVERSIDAD
POLITECNICA
DE VALENCIA

CAMPUS DE ALCOY

1ª Jornada Técnica sobre procesos de oxidación avanzada en el ciclo integral del agua

"Proceso foto-Fenton: Estrategias de aplicación en condiciones próximas a la neutralidad"



Castellón - 15 de enero de 2016

Grupo de Procesos de Oxidación Avanzada
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APPLICATION OF PHOTO-FENTON PROCESSES AT CIRCUMNEUTRAL pH

- INTRODUCTION PHOTO-FENTON
- FENTON –LIKE PROCESS:
 - PROCESSES USING OTHER METALS AS PHOTOCATALYSTS
 - PHOTO-FENTON PROCESSES AT CIRCUMNEUTRAL pH
 - Fe⁰ ; supported Iron Fe (Films; Zeolites...)
 - Complex Formation (Oxalate, Citrate, EDTA, EDDS, Humic acids, Fulvic acids, SBO)
- CONCLUSIONS

FENTON PROCESSES

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- **1894:** Discovered by **J. Fenton**
- **1934 :** **Haber y Weiss** the oxidant specie: $\cdot\text{OH}$
Barb y cols. Propose mechanism ($\cdot\text{OH}$)
- **The 60th :** Pollutants oxidation
- **The 90th** Application in wastewater treatment
- **2000s :** Real wastewater at pilot plant
- **2010s :** Photo-Fenton at Circumneutral pH

HOMOGENEOUS PHOTOCATALYSIS

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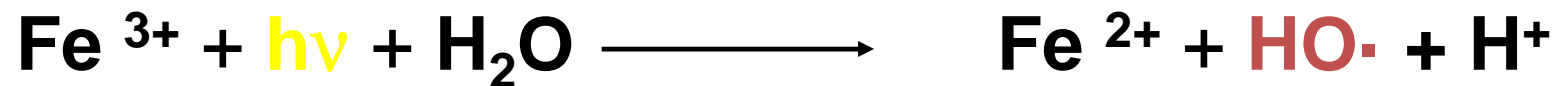
FENTON PROCESS:



Reduction **Fe (III) to Fe (II).**

UV Light or **SOLAR Light**

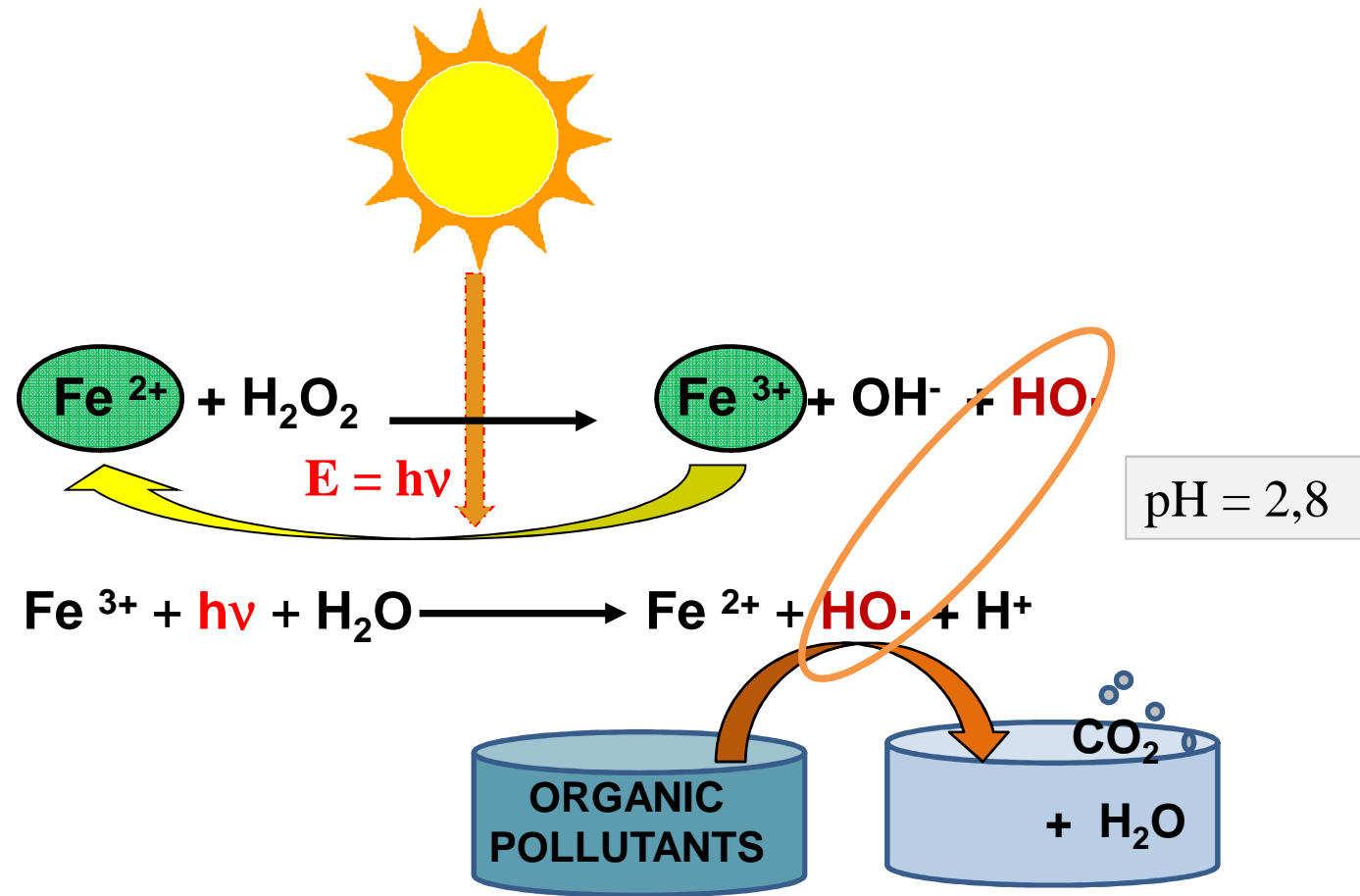
PHOTO-FENTON:



PHOTOCATALYTIC

SOLAR PHOTO-FENTON

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FENTON MECHANISM

- $\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \text{OH}^- + \text{OH}^\bullet$ $k = 70\text{M}^{-1}\text{s}^{-1}$
The required amount of oxidizer to the degradation of the organic pollutants are generated: (OH^\bullet) which is trapped by both Fe^{2+} and H_2O_2

- $\text{Fe}^{3+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{2+} + \text{H}^+ + \text{HO}_2^\bullet$ $k = 1\text{-}2 \cdot 10^{-2} \text{M}^{-1}\text{s}^{-1}$
Very slow reaction: the rate-limiting step in the degradation of the organic pollutants

- $\text{HO}^\bullet + \text{H}_2\text{O}_2 \rightarrow \text{HO}_2^\bullet + \text{H}_2\text{O}$ $k = 1.7\text{-}4.5 \cdot 10^{-7} \text{M}^{-1}\text{s}^{-1}$

- $\text{Fe}^{2+} + \text{HO}^\bullet \rightarrow \text{Fe}^{3+} + \text{OH}^-$ $k = 3.2 \cdot 10^{-8} \text{M}^{-1}\text{s}^{-1}$

OH^\bullet generate Fe^{3+} that as the pH increases precipitated as ferric oxyhydroxides

- $\text{Fe}^{3+} + \text{HO}_2^\bullet \rightarrow \text{Fe}^{3+} + \text{O}_2 + \text{H}^+$ $k = 1.2 \cdot 10^{-6} \text{M}^{-1}\text{s}^{-1}$
- $\text{Fe}^{2+} + \text{HO}_2^\bullet + \text{H}^+ \rightarrow \text{Fe}^{3+} + \text{H}_2\text{O}_2$ $k = 1.2 \cdot 10^{-6} \text{M}^{-1}\text{s}^{-1}$
- $\text{HO}_2^\bullet + \text{HO}_2^\bullet \rightarrow \text{H}_2\text{O}_2 + \text{O}_2$

Photo Fenton Reaction

- $\text{Fe}(\text{OH})^{2+} + h\nu \rightarrow \text{Fe}^{2+} + \text{OH}^\bullet$

Water Res. 44: 545-554

FENTON REACTIONS

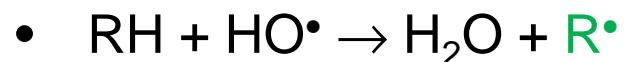
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Reacción	k (M ⁻¹ s ⁻¹)	Referencia	
$\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \text{OH}^- + \cdot\text{OH}$	76	Wailing (1975)	(1.1)
$\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{2+} + \text{H}^+ + \cdot\text{OOH}$	0,01	Wailing (1975)	(1.2)
$\text{Fe}^{2+} + \cdot\text{OH} \rightarrow \text{Fe}^{3+} + \text{OH}^-$	$3,2 \times 10^8$	Stuglik yZagorski (1981); Buxton et al. (1988)	(1.3)
$\text{OH} + \text{H}_2\text{O}_2 \rightarrow \cdot\text{OOH} + \text{H}_2\text{O}$	$2,7 \times 10^7$	Buxton et al. (1988);Christensen et al. (1982)	(1.4)
$\cdot\text{OOH} \rightarrow \text{O}_2^{\cdot-} + \text{H}^+$	$1,58 \times 10^5 \text{ s}^{-1}$	Bielski et al. (1985)	(1.5)
$\text{O}_2^{\cdot-} + \text{H}^+ \rightarrow \cdot\text{OOH}$	$1,0 \times 10^{10}$	Bielski et al. (1985)	(1.6)
$\text{Fe}^{2+} + \cdot\text{OOH} \xrightarrow{+\text{H}^+} \text{Fe}^{3+} + \text{H}_2\text{O}_2$	$1,2 \times 10^6$	Jayson et al. (1969); Rush y Bielski (1985)	(1.7)
$\text{Fe}^{3+} + \cdot\text{OOH} \rightarrow \text{Fe}^{2+} + \text{H}^+ + \text{O}_2$	$3,1 \times 10^5$	Rush y Bielski (1985)	(1.8)
$\cdot\text{OH} + \cdot\text{OOH} \rightarrow \text{H}_2\text{O} + \text{O}_2$	$1,0 \times 10^{10}$	Sehested et al. (1969)	(1.9)
$\cdot\text{OH} + \cdot\text{OH} \rightarrow \text{H}_2\text{O}_2$	$4,29 \times 10^9$	Sehested et al. (1969)	(1.10)
$\cdot\text{OOH} + \cdot\text{OOH} \rightarrow \text{H}_2\text{O}_2 + \text{O}_2$	$8,23 \times 10^5$	Bielski et al. (1985)	(1.11)
$\text{Fe}^{3+} + \text{O}_2^{\cdot-} \rightarrow \text{Fe}^{2+} + \text{O}_2$	$5,0 \times 10^7$	Rothschild y Allen (1958)	(1.12)
$\text{Fe}^{2+} + \text{O}_2^{\cdot-} \xrightarrow{+\text{H}^+} \text{Fe}^{3+} + \text{H}_2\text{O}_2$	$1,0 \times 10^7$	Rush y Bielski (1985)	(1.13)
$\cdot\text{OH} + \text{O}_2^{\cdot-} \rightarrow \text{O}_2 + \text{OH}^-$	$1,0 \times 10^{10}$	Sehested et al. (1969)	(1.14)
$\cdot\text{OOH} + \text{O}_2^{\cdot-} \xrightarrow{+\text{H}^+} \text{H}_2\text{O}_2 + \text{O}_2$	$9,7 \times 10^7$	Bielski et al. (1985)	(1.15)
$\text{RH} + \cdot\text{OH} \rightarrow \text{R}^{\cdot} + \text{H}_2\text{O}$		Beltrán de Heredia et al. (2001)	(1.16)
$\text{R}^{\cdot} + \text{Fe}^{2+} \xrightarrow{+\text{H}^+} \text{RH} + \text{Fe}^{3+}$		Beltrán de Heredia et al. (2001)	(1.17)
$\text{R}^{\cdot} + \text{R}^{\cdot} \rightarrow \text{R}-\text{R}$		Beltrán de Heredia et al. (2001)	(1.18)
$\text{R}^{\cdot} + \text{Fe}^{3+} \rightarrow \text{R}^+ + \text{Fe}^{2+}$		Beltrán de Heredia et al. (2001)	(1.19)

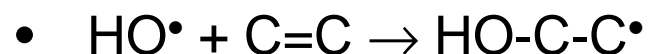
FENTON MECHANISM

REACTION MECHANISM OF OH• WITH ORGANICS COMPOUNDS

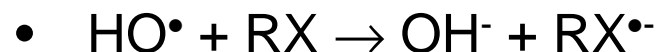
A) Hydrogen abstraction C-H, N-H, O-H



B) Addition to the double bonds



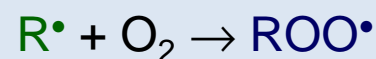
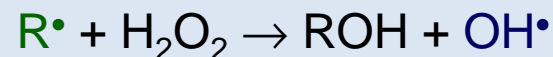
C) Electronic transfer



D) Addition to aromatic rings

E) Other.....

Organic radicals generated ($R\bullet$) react with H_2O_2 or O_2 generating hydroxyl radicals ($OH\bullet$) or peroxide radicals ($ROO\bullet$)



These reactions can produce mineralización of organic compounds to CO_2 , H_2O and inorganic ions

PHOTO-FENTON TREATMENT

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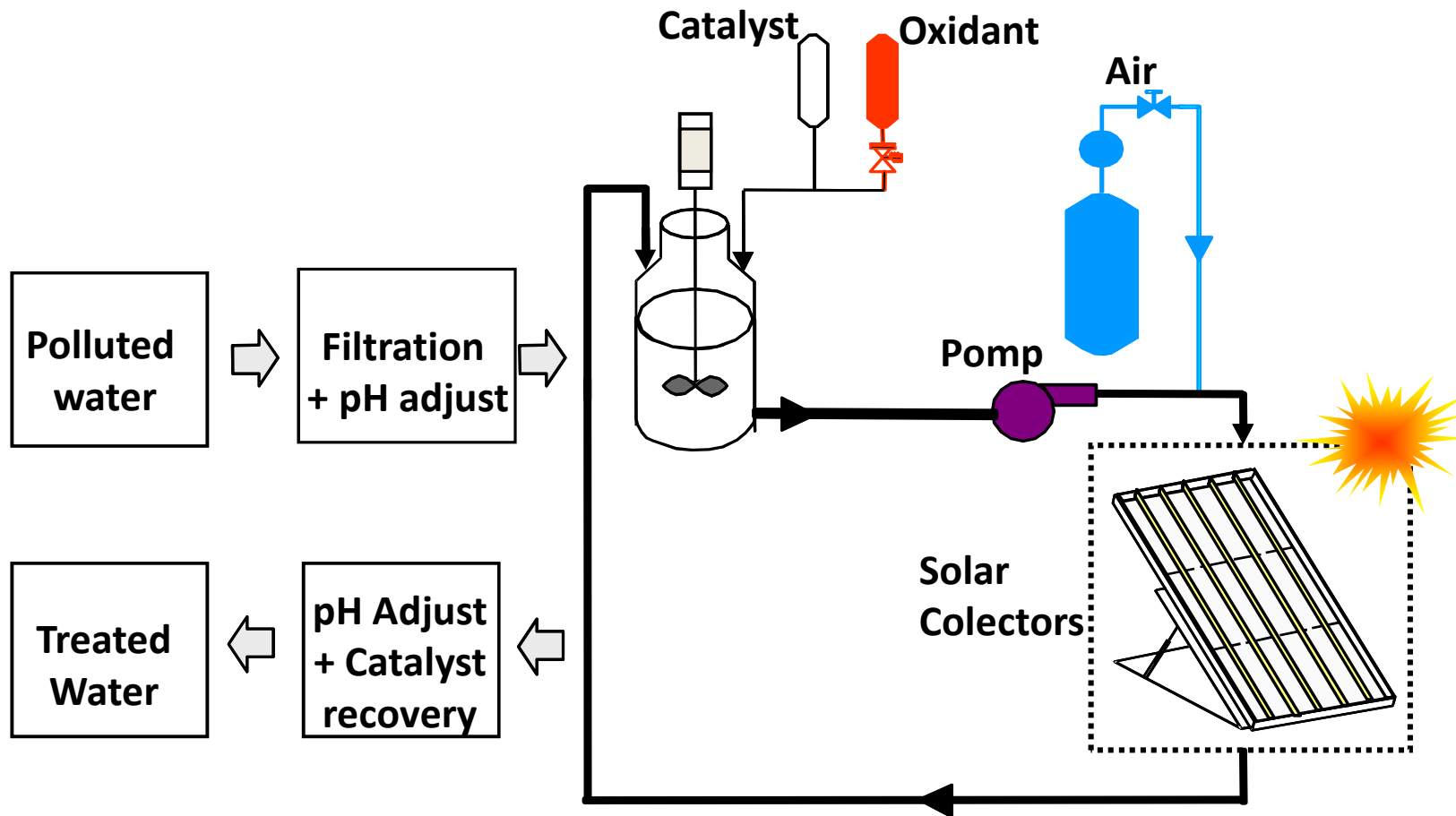
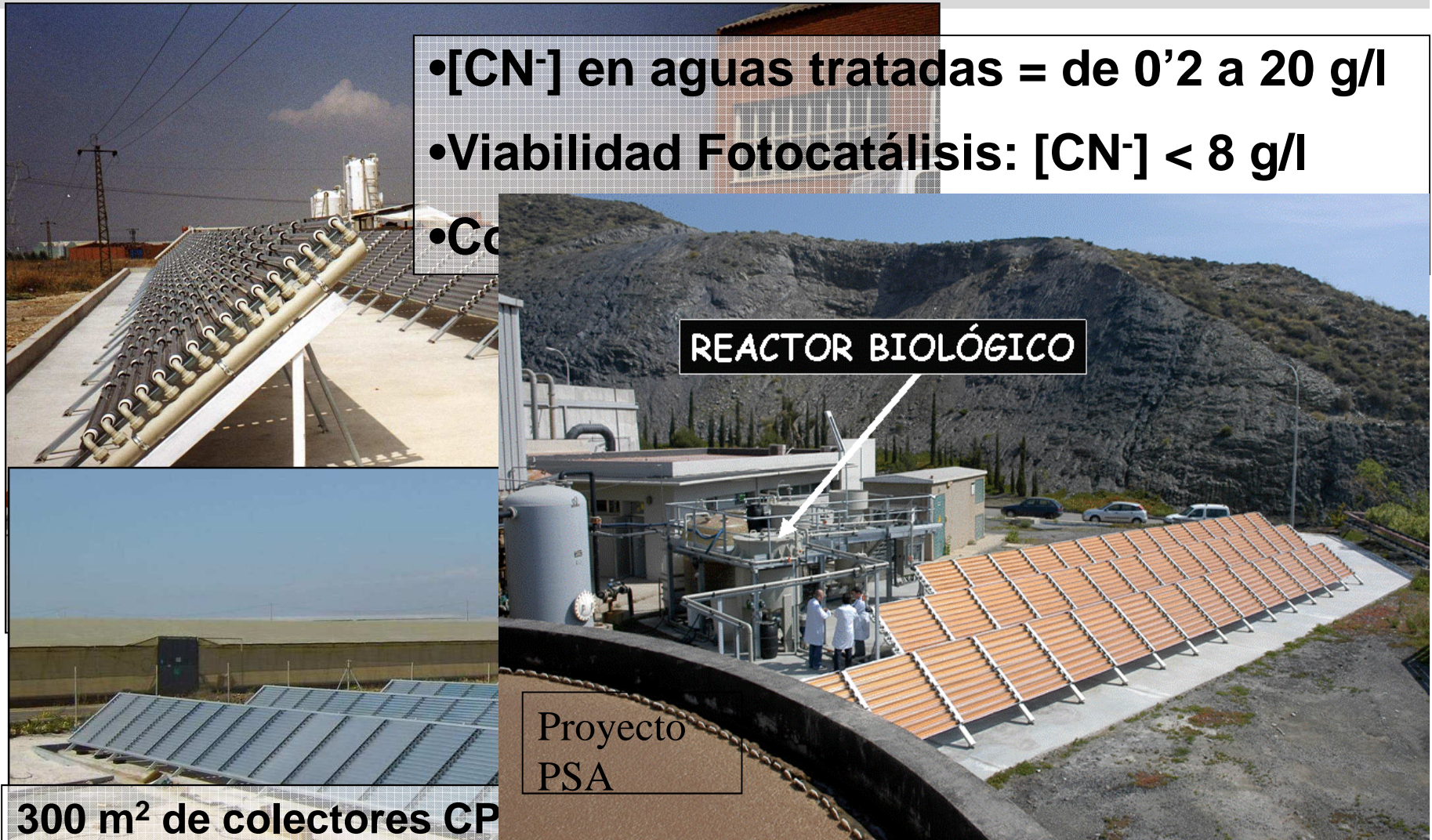


PHOTO-FENTON TREATMENT

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- $[\text{CN}^-]$ en aguas tratadas = de 0'2 a 20 g/l
- Viabilidad Fotocatálisis: $[\text{CN}^-] < 8 \text{ g/l}$
- Co



REACTOR BIOLÓGICO

Proyecto
PSA

300 m² de colectores CP

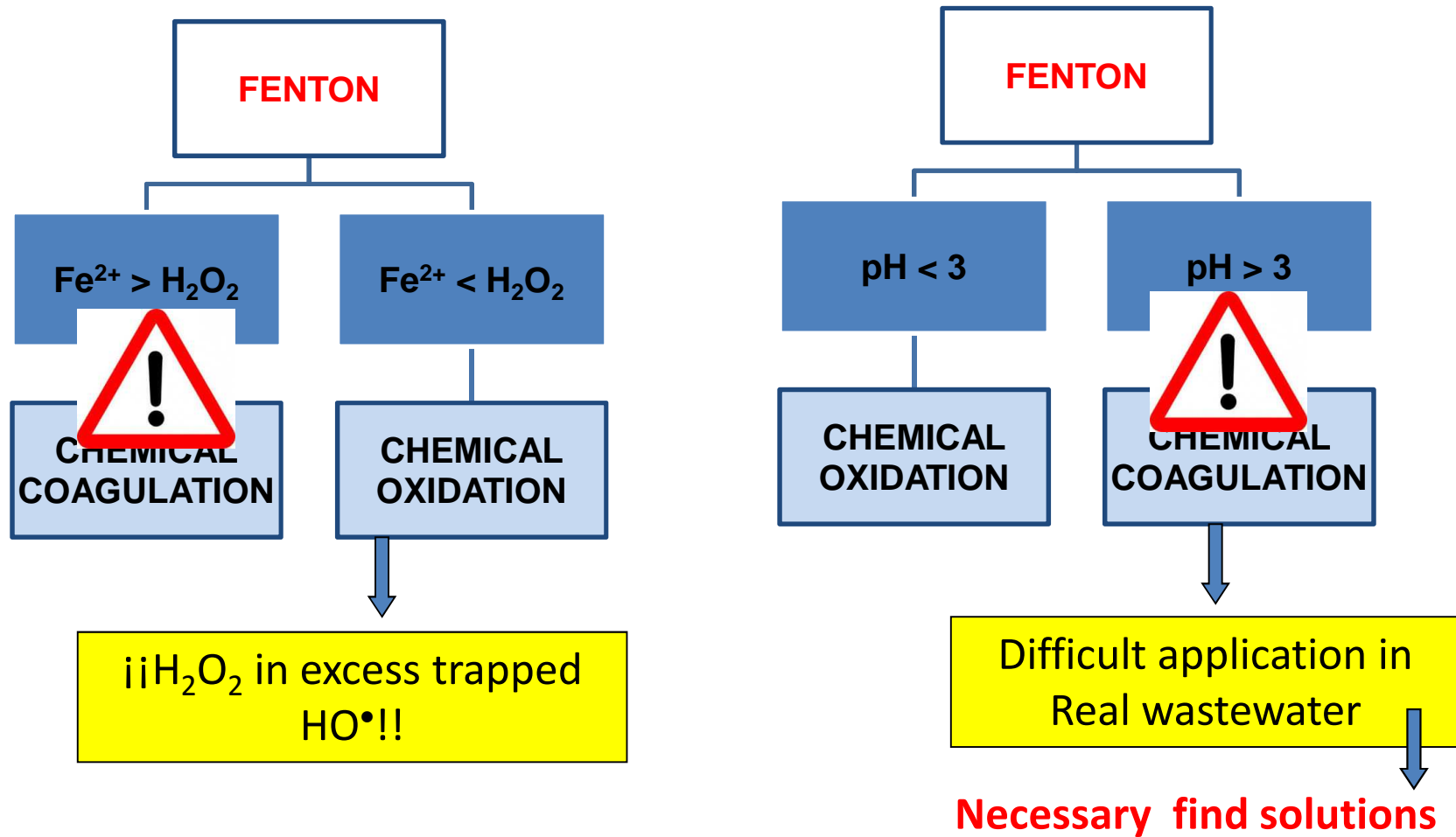
750.000 envases/año (50% comercializados en Almería)

41.4 kJ/L para mineralizar 108 mg/L de COT.

Application of Photo-Fenton Processes at circumneutral pH

INTRODUCTION PHOTO-FENTON PROCESS

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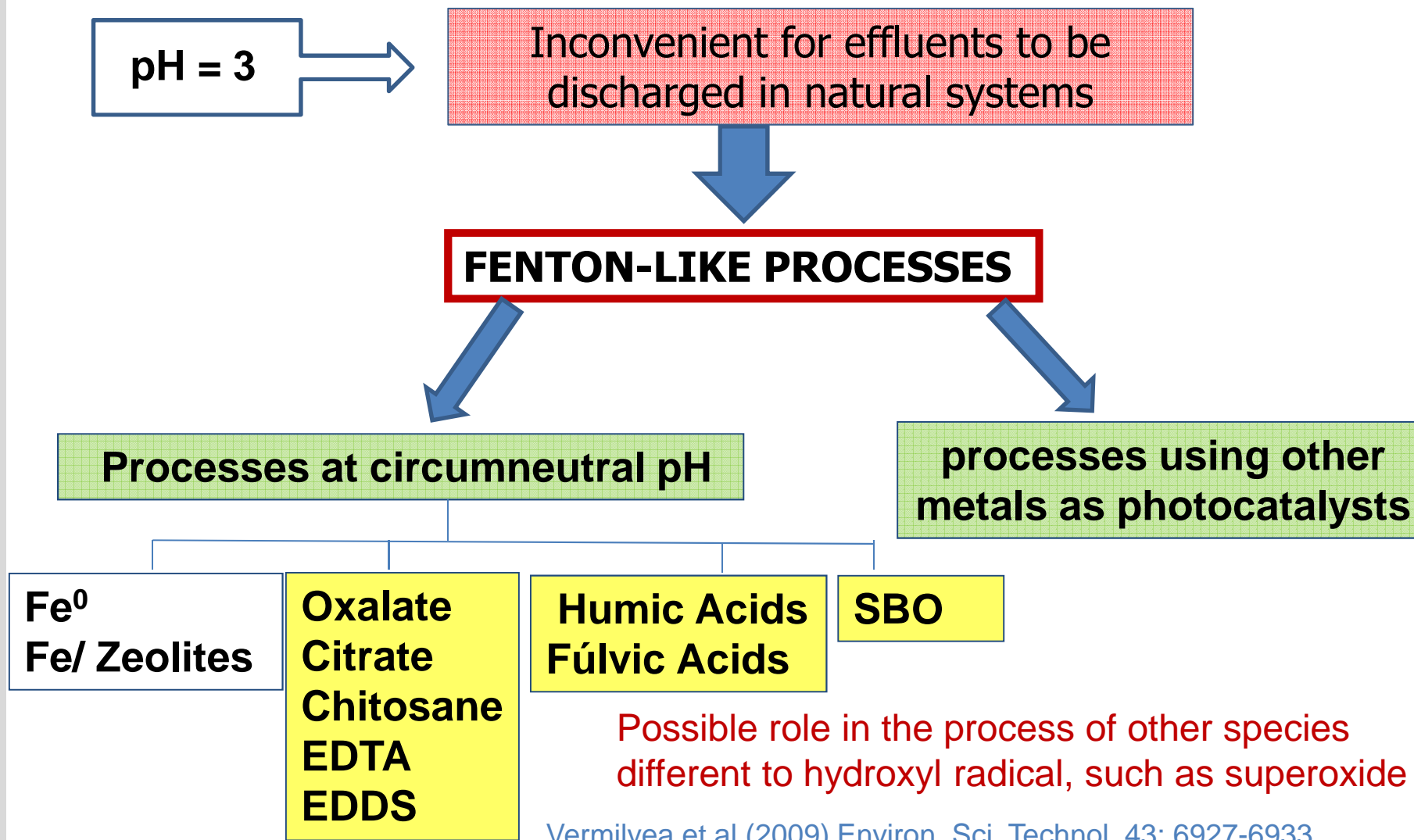


Klamerth et al. (2010) Water Res. 44: 545-554.
Bernabeu et al. (2011) Catal. Today 161: 233-240; (2012) Chem. Eng. J. 198-199: 65-72

Application of Photo-Fenton Processes at circumneutral pH

SOLUTIONS OF PHOTO-FENTON PROCESS

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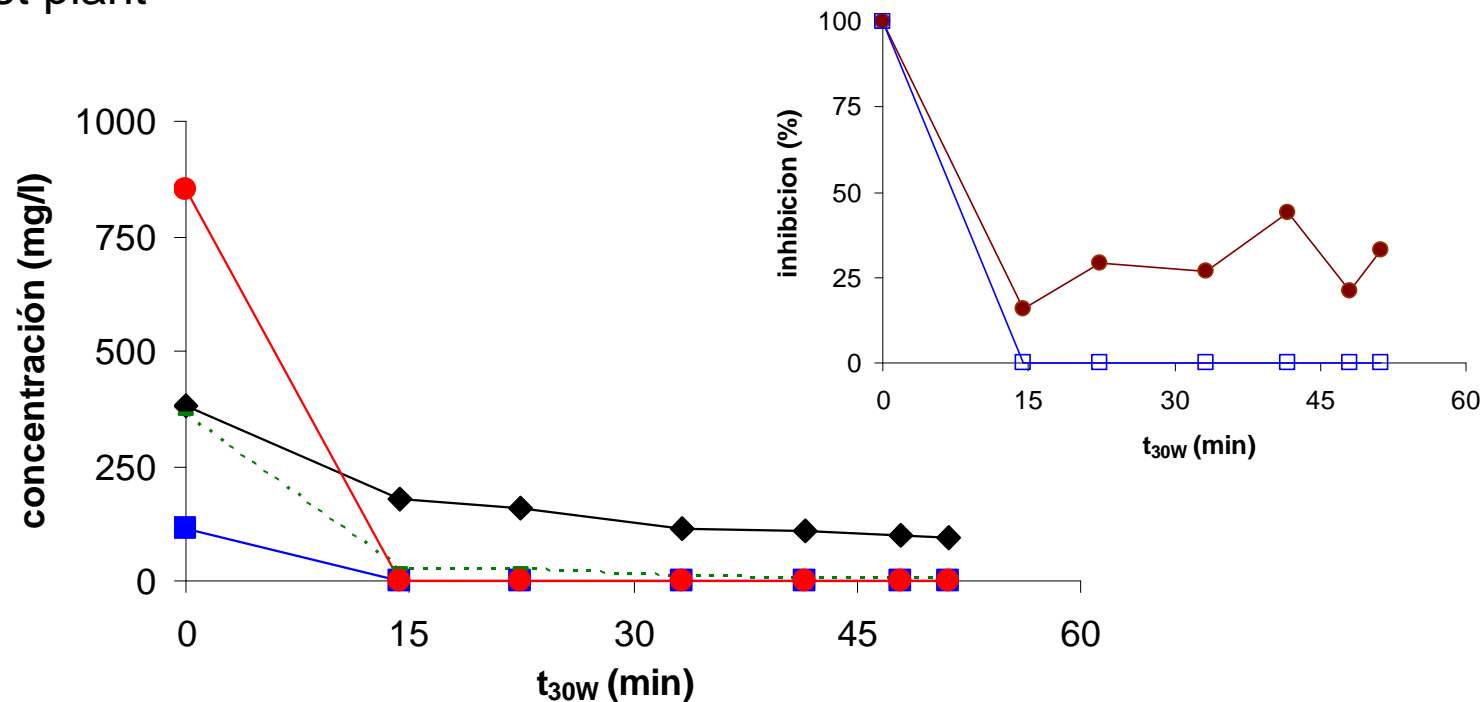
Vermilyea et al.(2009) Environ. Sci. Technol. 43: 6927-6933.
Applied Catalysis B: Environmental 146 (2014) 192– 200

Application of Photo-Fenton Processes at circumneutral pH

FENTON-LIKE PROCESSES: processes using other metals as photocatalysts (copper)

Finishing metal Industry wastewater

Toxicity (%) to the activated sludge of industrial wastewater treated by irradiation in pilot plant



Solar treatment with H_2O_2 . Cyanide degradation (●), free cyanide (■), Total carbono (◆) Copper degradation (▲). Toxicity (*vibrio fischeri*) (●), Activated sludge toxicity (□).

FENTON-LIKE PROCESSES: processes using other metals as photocatalysts (copper)

Finishing metal Industry wastewater

Toxicity (%) to the activated sludge of industrial wastewater treated by irradiation in pilot plant



t= 0; Toxicity = 75%

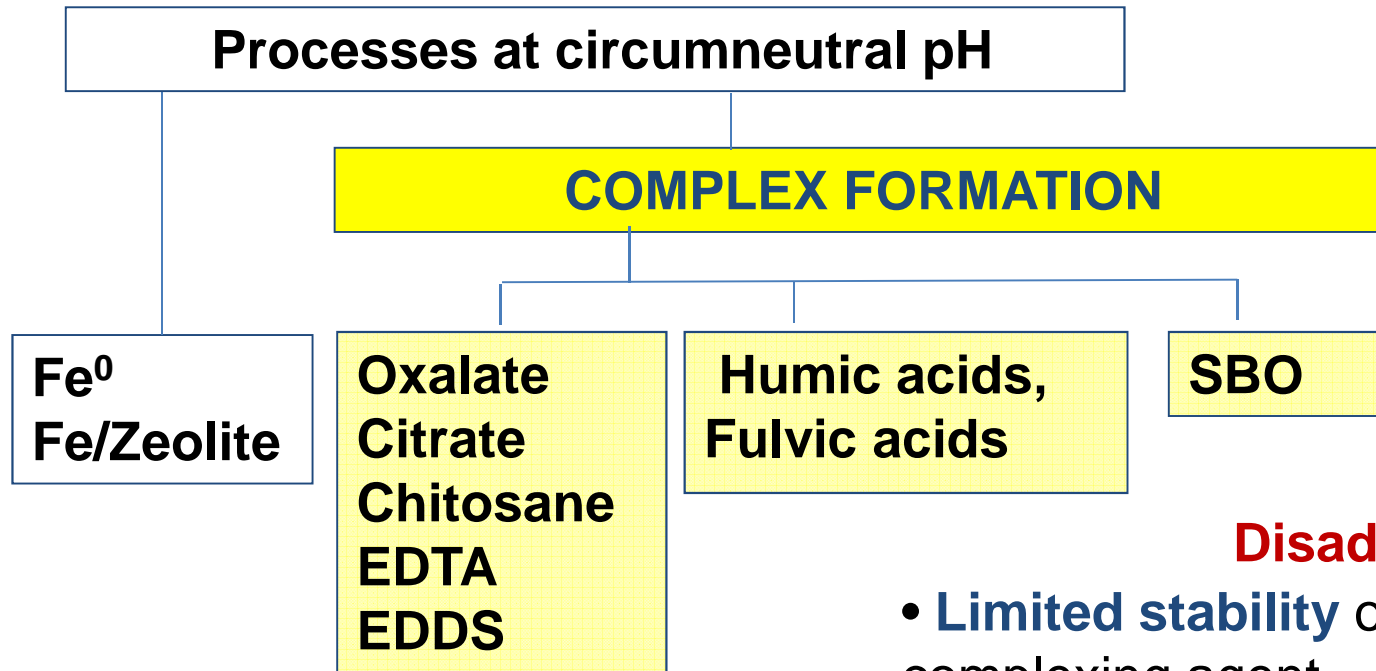


t= 3h, Toxicity = 37%



t= 6h, Toxicity = 13%

FENTON-LIKE PROCESSES: processes a circumneutral pH



Advantageous:

- **Stabilization of iron ions** over a wider pH
- High absorption of UV–vis radiation,



Disadvantageous:

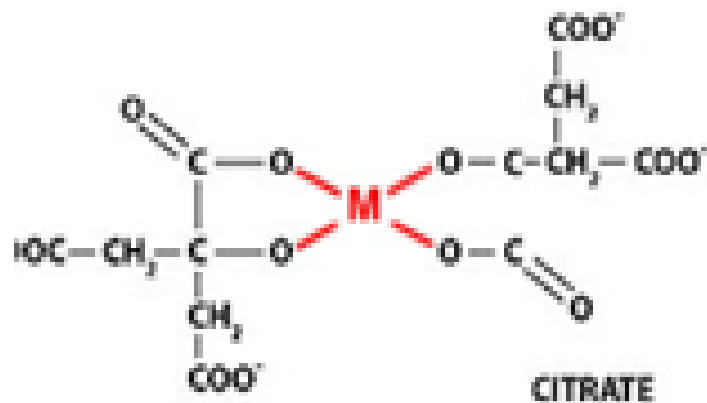
- **Limited stability** of the employed complexing agent
- Formation of very **stable non-photoactive iron complexes** (may hinder the participation of iron in the Fenton reaction)
- **Potential Toxicity** of chemicals auxiliaries

FENTON-LIKE PROCESSES: processes a circumneutral pH: citric Acid

COMPLEX FORMATION: CITRIC ACID

Iron ligand for application in photo-Fenton processes:

- Availability and the possibility of improving the Fenton reaction at pH values above 2.8 (up to neutrality), with high quantum yields of Fe(II) ($\Phi_{\text{Fe(II)}}$) and hydroxyl radical generation .



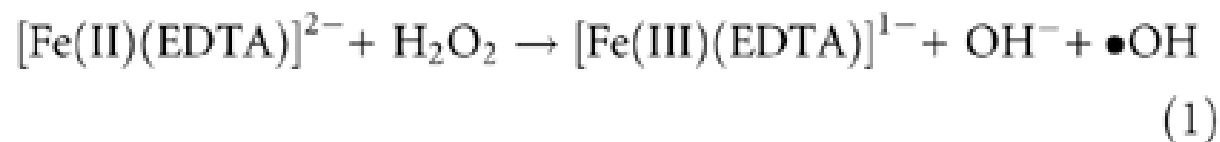
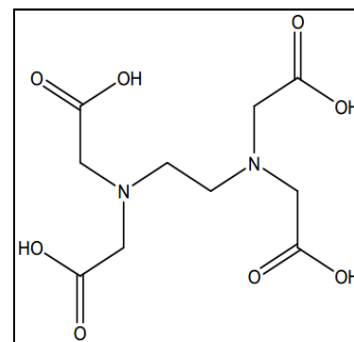
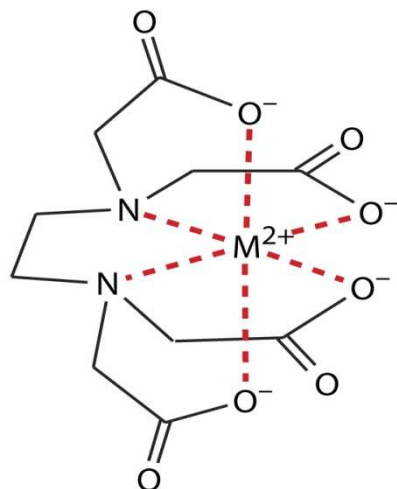
Furthermore, citric acid is **biodegradable**, and therefore environmentally friendly in contrast to other ligands, such as EDTA, that show poor biodegradability.

([Sillanpaa and Pirkanniemi, 2001](#)),

([Zepp et al., 1992](#), [Abrahamson et al., 1994](#), [Zhang et al., 2006](#) and [Kwan and Chu, 2007](#)).

FENTON-LIKE PROCESSES: processes a circumneutral pH: EDTA

COMPLEX FORMATION: EDTA

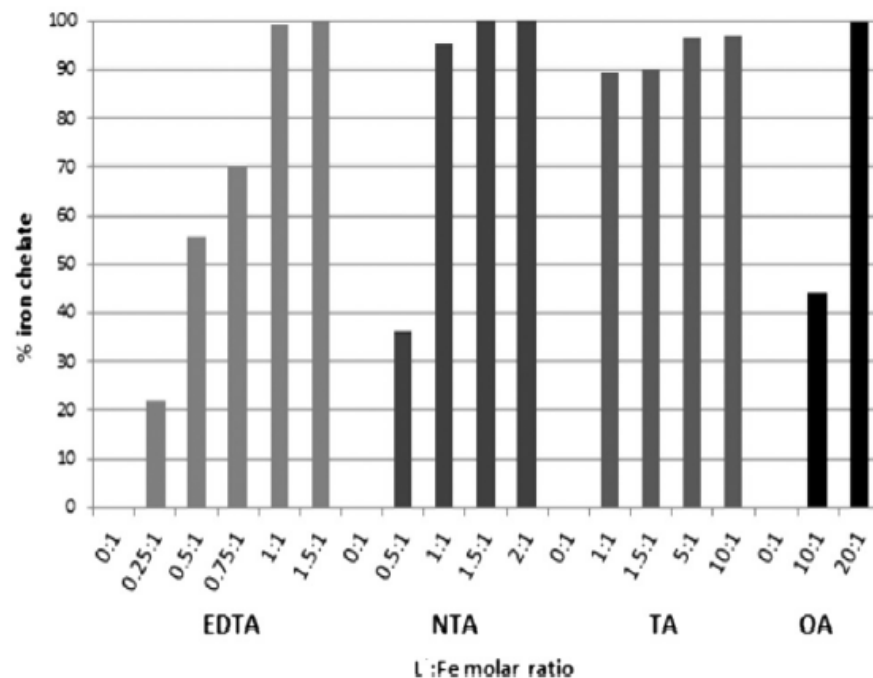


Low biodegradability
Toxicity

C.Y. Kwan, W. Chu / Chemosphere 67 (2007) 1601–1611
DeLuca, Dantas, Esplugas, Water research 61 (2014) 232-242

FENTON-LIKE PROCESSES: processes a circumneutral pH

COMPLEX FORMATION:



ethylenediaminetetraacetic acid (**EDTA**);
nitrilotriacetic acid (**NTA**);
oxalic acid (**OA**)
tartaric acid (**TA**).

Chelates of EDTA and NTA presented more stability than OA and TA,

Fig. 1 – Percentage of iron chelates formed with several L:Fe(III)molar ratio tested.

FENTON-LIKE PROCESSES: processes a circumneutral pH: EDTA

COMPLEX FORMATION: EDTA

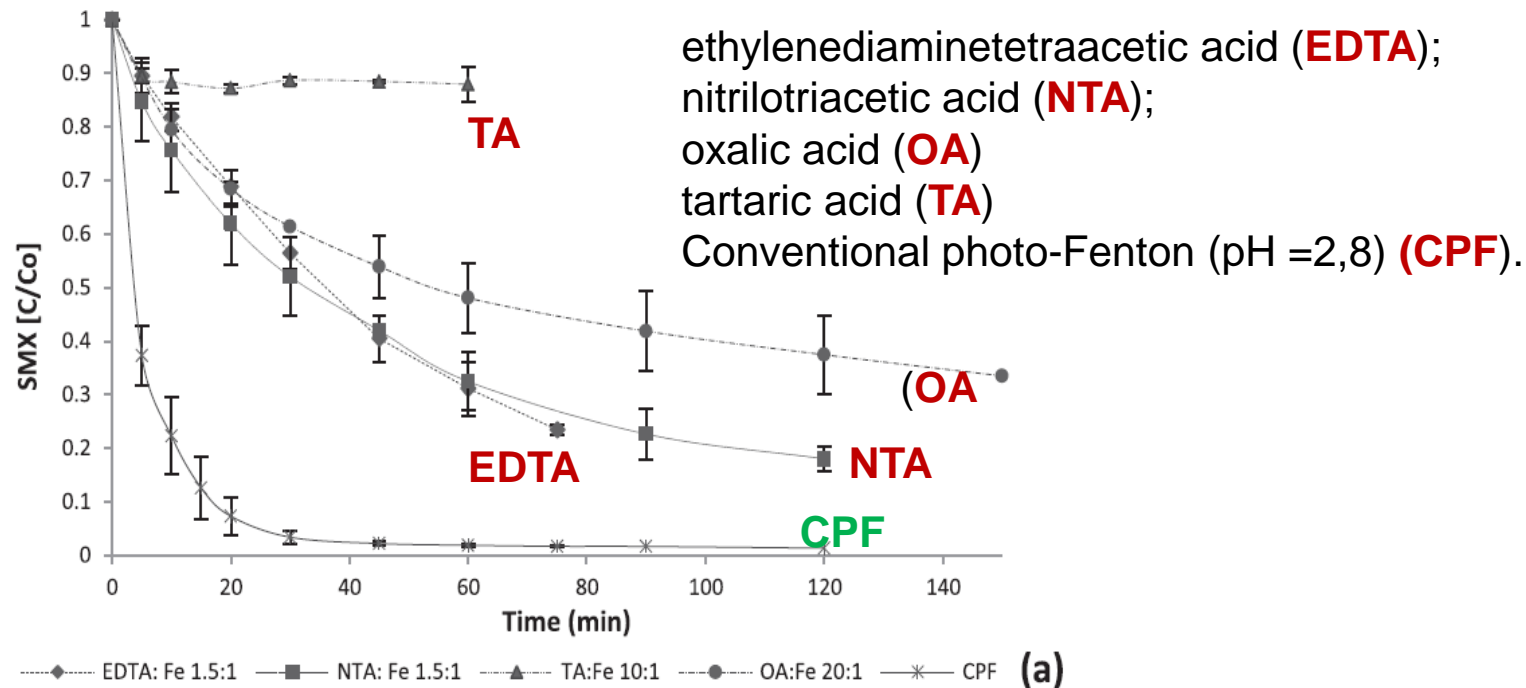


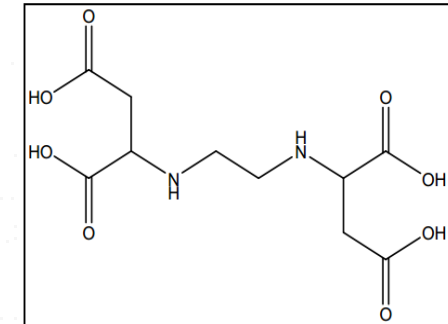
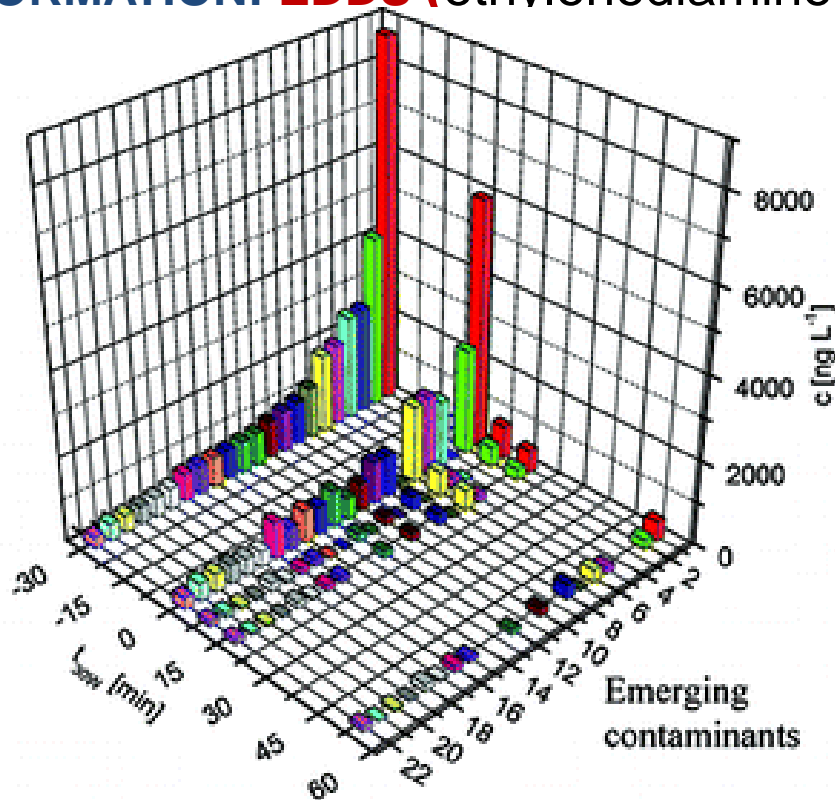
Fig. SMX removal by photo-Fenton like at neutral pH with Fe(III)-L chelates and trend of pH values over time reaction.
(pH = 7.0), ([SMX]₀=0.079 mM; [Fe²⁺]₀ = 0.089 mM; [H₂O₂]₀ = 0.294 mM;

DeLuca, Dantas, Esplugas, Water research 61 (2014) 232-242

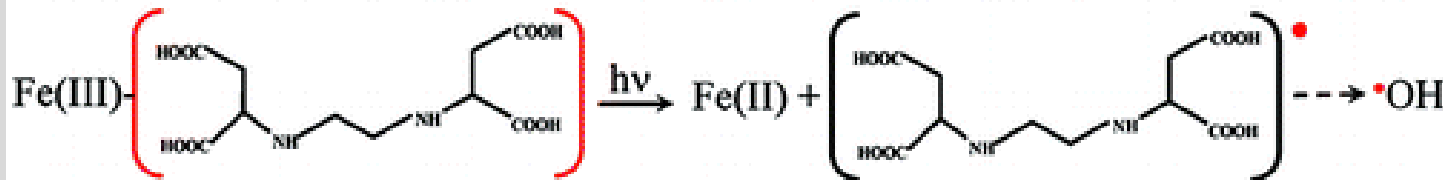
FENTON-LIKE PROCESSES: processes a circumneutral pH: EDDS

COMPLEX FORMATION: **EDDS** (ethylenediamine-N,N'-disuccinic acid)

- Neutral pH
- CPC
- Degradation of 22 Eps In MWTP



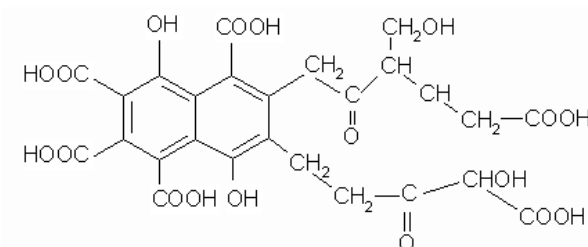
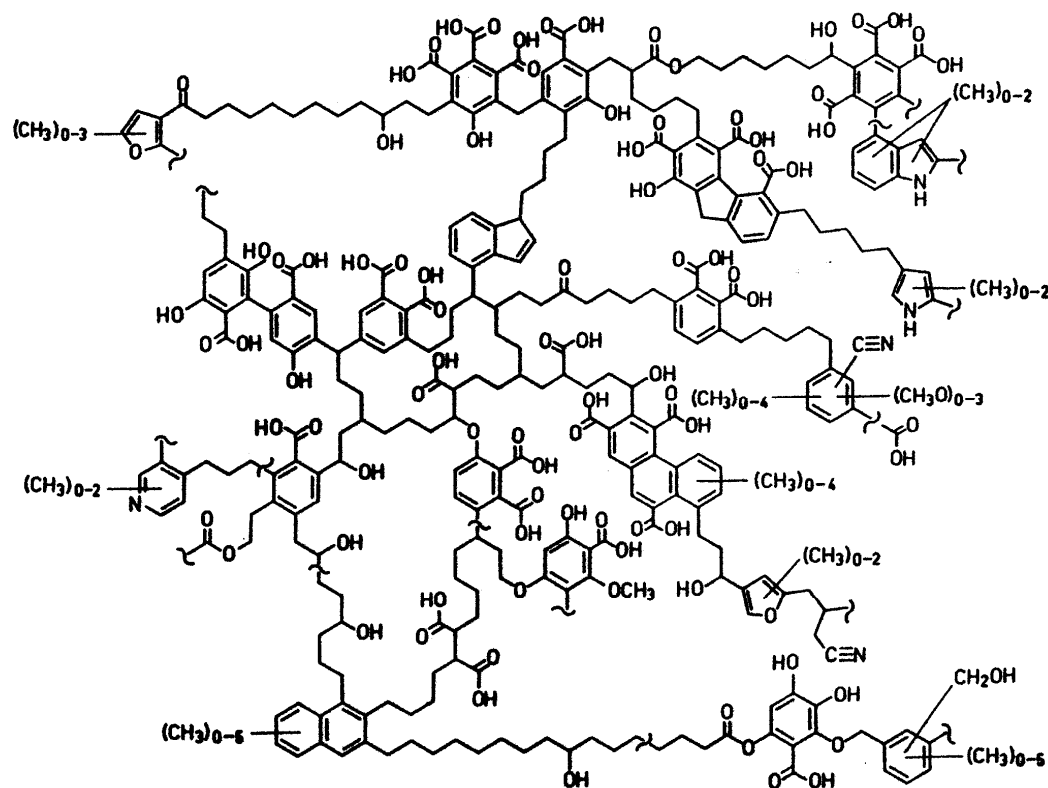
degradation rate 95.5%
in $t_{30W} = 63$ min,
Lower peroxide consumption:
(61 mg L⁻¹ pH =6,3
80 mg L⁻¹ at pH 3).



(Klamerth (2011)Catal. Today, 161: 241-246.

FENTON-LIKE PROCESSES: processes a circumneutral pH: Humic Acids

COMPLEX FORMATION: HUMIC and FULVIC ACIDS



These substances contain functional groups that are able to complex iron

Mikutta et al (2011) *Environ. Sci. Technol.*, 45: 9550–9557
 Lipczynska-Kochany et al. (2008) *Chemosphere* 73: 745-750.
 Georgi et al.(2007) *Appl. Catal. B: Environ.* 72: 26-36.
 Fan et al. (2011) *Chemosphere* 82: 229-236.

FENTON-LIKE PROCESSES:

processes a circumneutral pH: Humic acids

COMPLEX FORMATION: HUMIC and FULVIC ACIDS

Dreissena polymorpha (zebra mussel) is an invasive freshwater bivalve mollusc that causes important technical and environmental problems

Neutral photo-Fenton could be a promising alternative as ca.90% damaged larvae were detected in only 3 hours irradiation in the presence of H_2O_2 (10 mg/l).

This process was clearly more effective than sunlight irradiation, H_2O_2 , or dark Fenton.

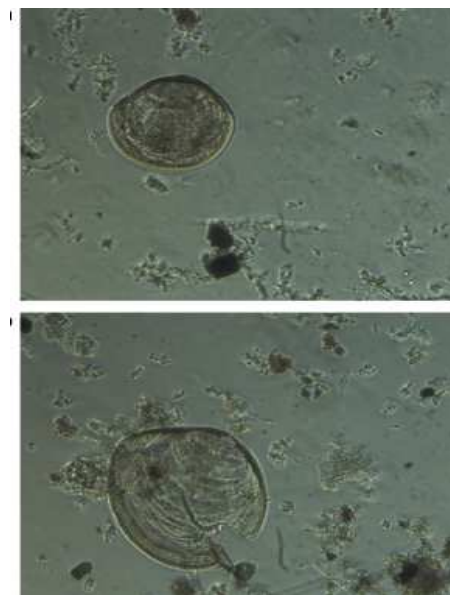
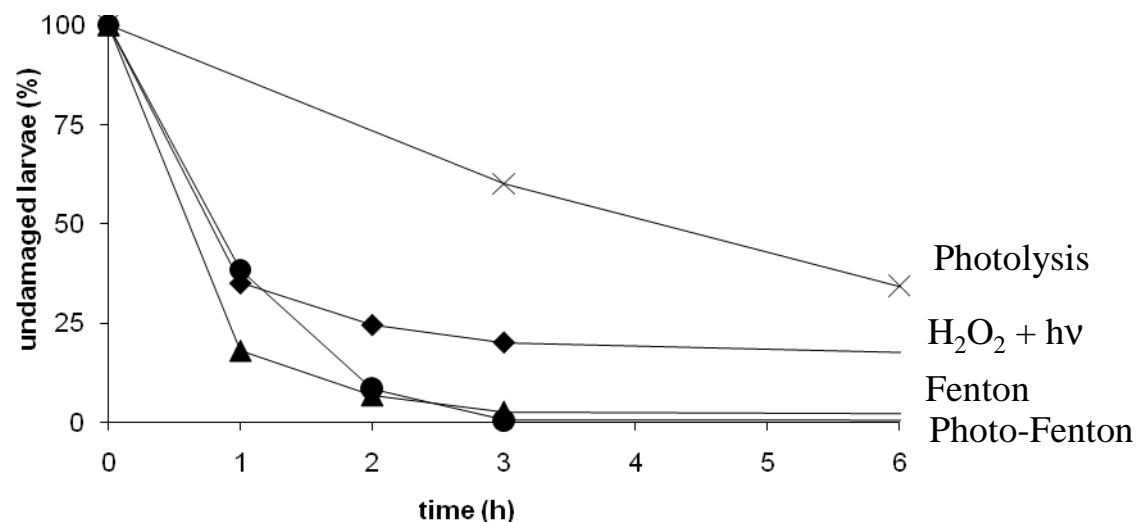


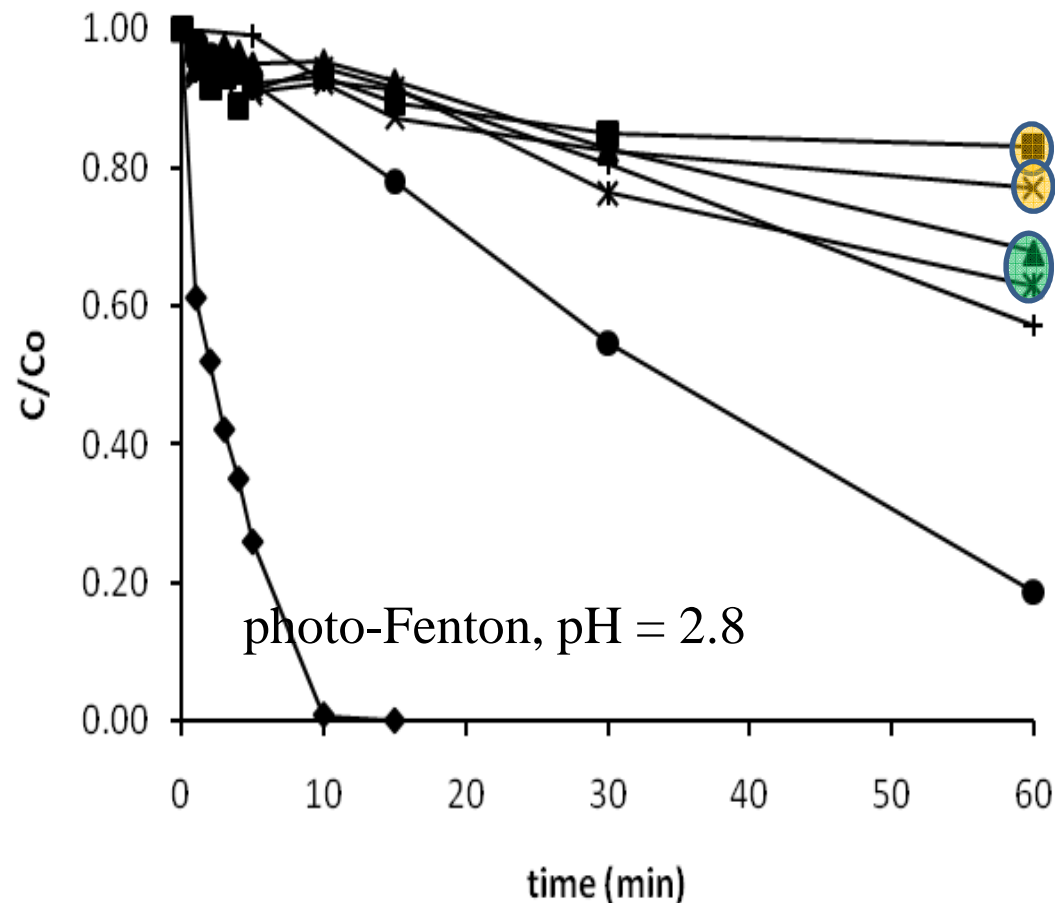
Photo-Fenton (5 mg/l of iron and 10 mg/l of H_2O_2 , sunlight) (▲), Fenton (5 mg/l of iron and 10 mg/l of H_2O_2) (●), 10 mg/l of H_2O_2 (♦) and control experiment (×).

FENTON-LIKE PROCESSES:

processes a circumneutral pH: Humic acids

COMPLEX FORMATION: HUMIC ACIDS

Relative concentration of caffeine (50 mg/l) vs. irradiation time

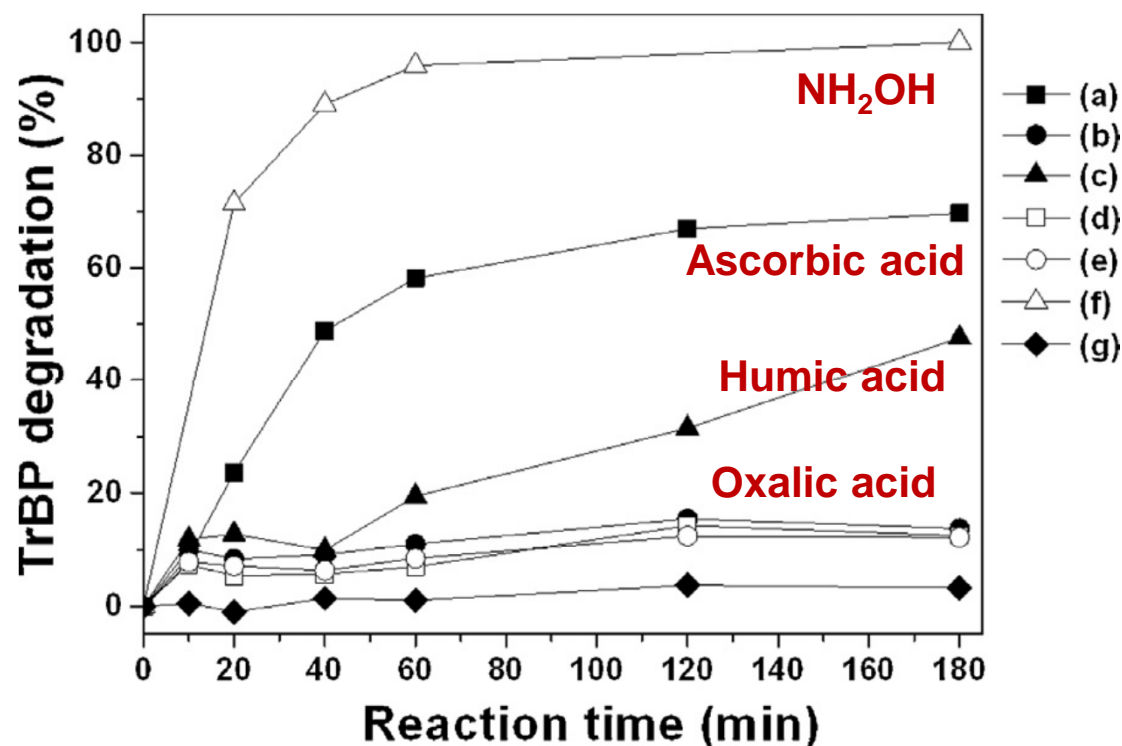


- (■) photo-Fenton, pH = 7, distilled water;
- (×) photo-Fenton, pH = 7, tap water
- (▲) photo-Fenton, pH = 7, humic acids, distilled water;
- (*) photo-Fenton, pH = 7, humic acids, tap water

FENTON-LIKE PROCESSES:

processes a circumneutral pH: humic acids

COMPLEX FORMATION:



Effects of reducing agents on the kinetics of **TriBromoPhenol degradation**.

(a) Ascorbic Acid
 (b) oxalic acid,
 (c) p-hydroquinone,
 (d) humic acid,
 (e) gallic acid,
 (f) NH₂OH,
 (g) Without Reducing Agents,

[TrBP]₀ 100 M, [H₂O₂] 20 mM,
 Fe-Z 109 mg L⁻¹ (30 M),

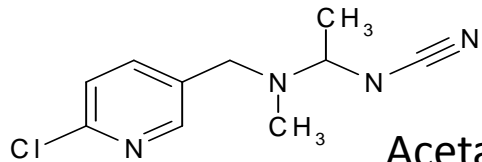
Applied Catalysis B: Environmental 147 (2014) 411– 419

FENTON-LIKE PROCESSES:

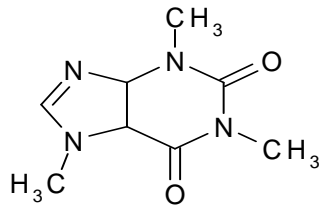
processes a circumneutral pH: Humic acids

COMPLEX FORMATION: HUMIC and FULVIC ACIDS

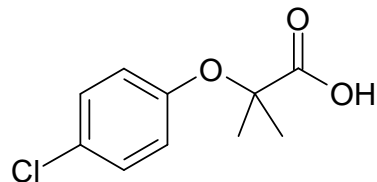
In WTP



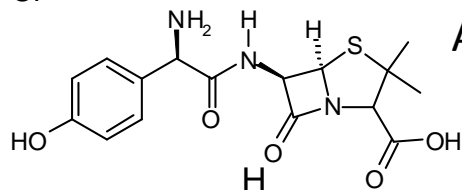
Acetamiprid



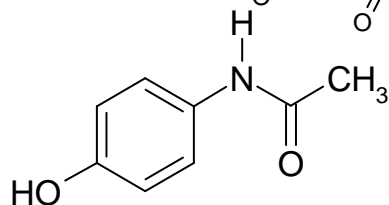
Caffeine



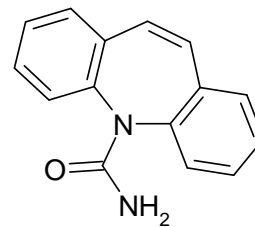
Clofibric Acid



Amoxiciline



Acetaminophen



Carbamazepine



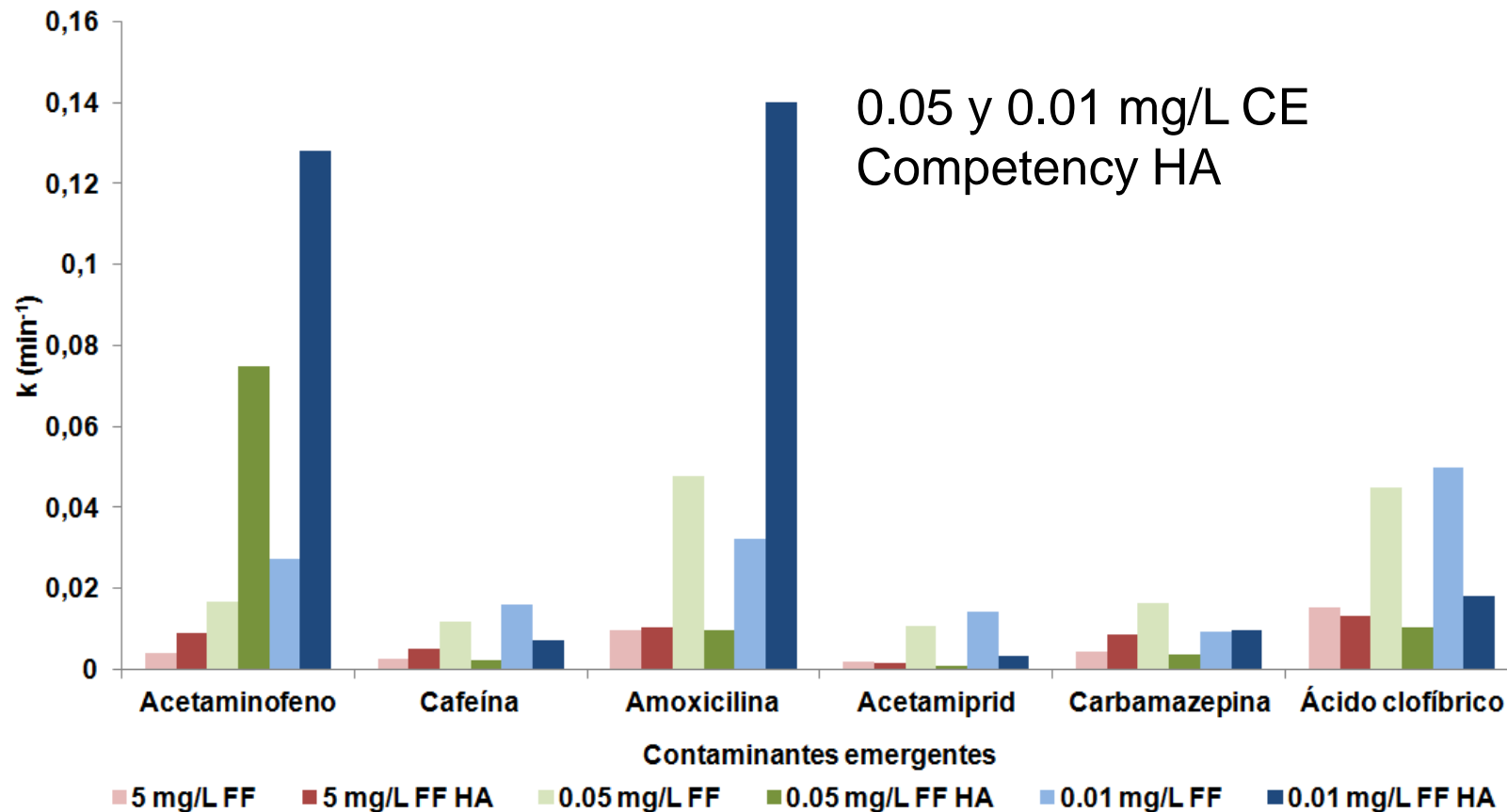
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Application of Photo-Fenton Processes at circumneutral pH

FENTON-LIKE PROCESSES: processes a circumneutral pH: Humic acids

COMPLEX FORMATION: HUMIC and FULVIC ACIDS

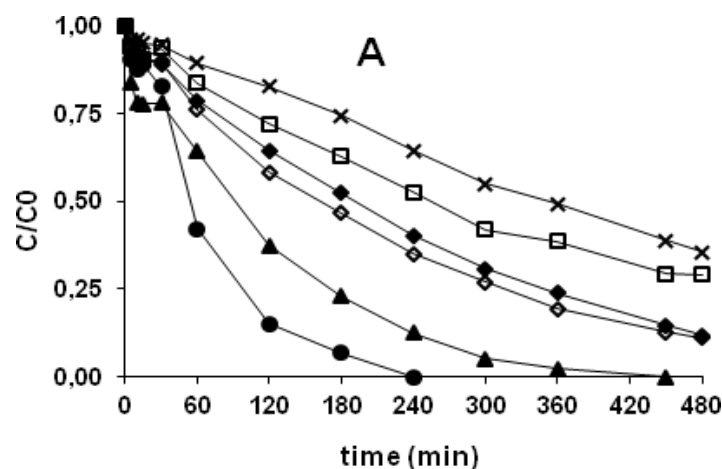
In WTP



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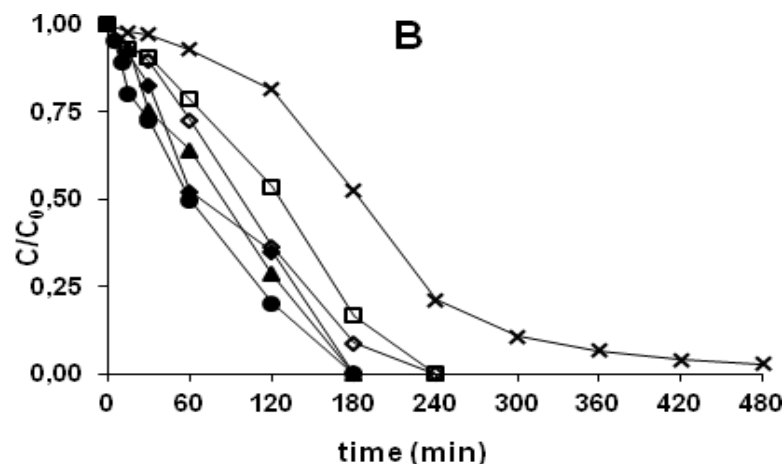
FENTON-LIKE PROCESSES:

processes a circumneutral pH: Humic acids



neutral photo-Fenton in distilled water (A)

amoxicillin (▲),
acetaminophen (◆),
acetempirid (×),
caffeine (□),
clofibric acid (●)
carbamazepine (◇).

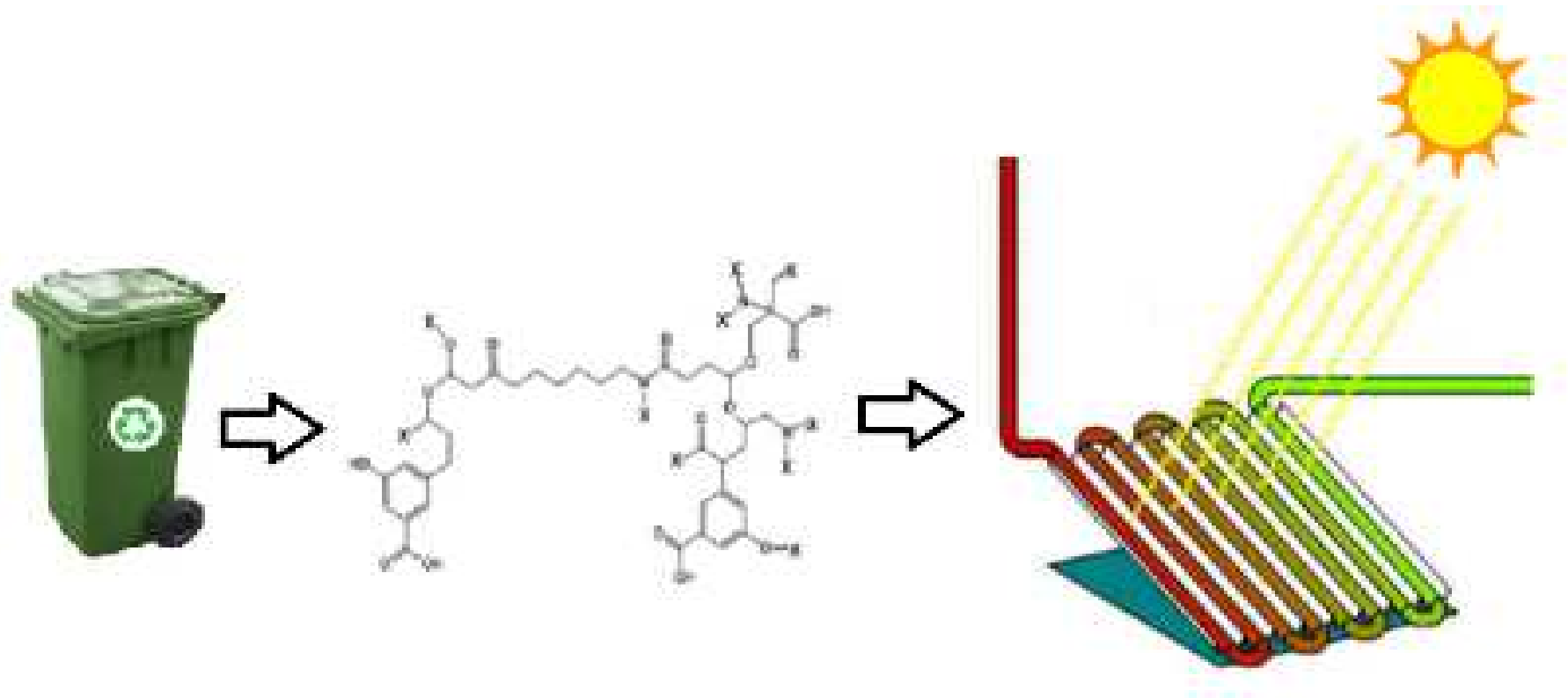


neutral photo-Fenton in distilled water and **humic acids** (10mg/L)(B)

FENTON-LIKE PROCESSES: processes a circumneutral pH

COMPLEX FORMATION: SBO

Constituted by macromolecules (67 to 463 kg mol^{-1}) with similar characteristics as humic substances



FENTON-LIKE PROCESSES: processes a circumneutral pH

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COMPLEX FORMATION: **SBO**

Urban wastes



Organic fraction

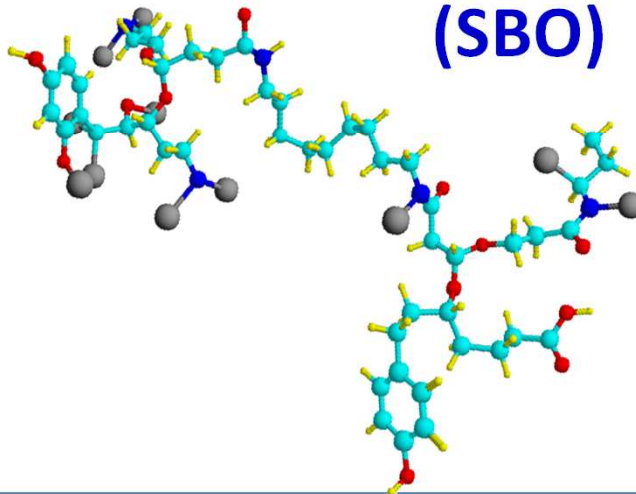


- Pre-treatments
- Anaerobic digestion
- Aerobic biodegradation

Biomass



Soluble Bio-Organics
(SBO)



- Digestion at basic conditions
- Centrifugation to remove the non-soluble fraction
- Ultrafiltration of supernatant
- Drying of the retentate

FENTON-LIKE PROCESSES:

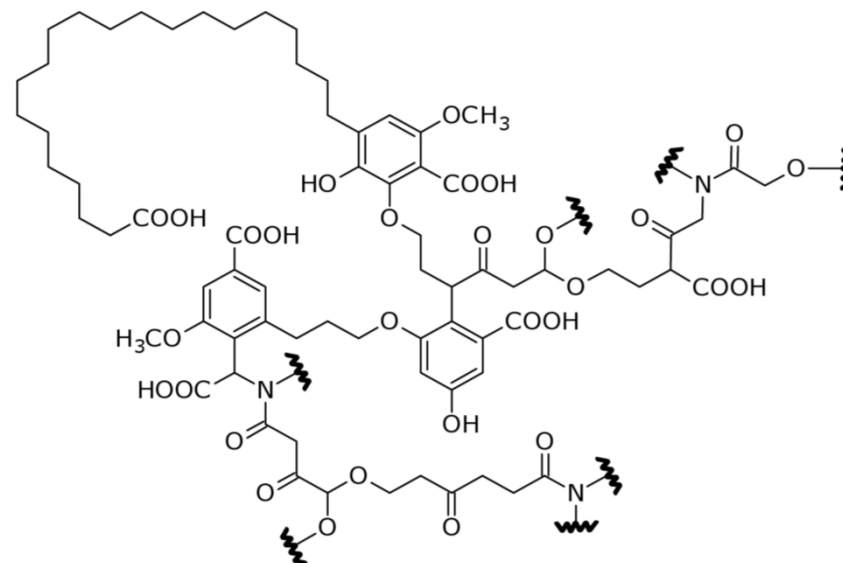
processes a circumneutral pH

COMPLEX FORMATION: SBO

A (FORSUD): urban waste organic humid fraction mixed with the digestate from an anaerobic reactor.

B (CVDFT110): mixture of FORSUD, gardening residues and urban sewage sludge

C (CVT230): home gardening and park trimming residues (GR) aerated for 230 days



Functional groups present in SBO (e.g. carboxylic or amide) indicate that these substances are able to act as chelating agents for iron, what might be useful to apply photo-Fenton at neutral conditions

Gomis et al.(2013) Catalysis Today. 209: 176-180.

Gomis et al. (2014) Chem. Eng. J. 235: 236-243.

FENTON-LIKE PROCESSES:

processes a circumneutral pH

COMPLEX FORMATION: SBO

	A (FORSUD)	B (CVDF110)	C (CVT230)
Volatile solids (% w/w)	84.6	72.7	72.1
Carbon (% w/w)	45.1	35.5	38.2
Nitrogen (% w/w)	7.8	4.3	4.0
Si (% w/w)	0.36	0.92	2.55
Fe (% w/w)	0.16	0.53	0.77
Al (% w/w)	0.78	0.44	0.49
Mg (% w/w)	0.18	0.49	1.13
Ca (% w/w)	1.32	2.59	6.07
K (% w/w)	9.2	5.4	3.6
Na (% w/w)	0.39	0.15	0.16
Cu (mg/l)	100	216	202
Ni (mg/l)	27	71	92
Zn (mg/l)	185	353	256
Cr (mg/l)	11	30	19
Pb (mg/l)	44	75	85
Hg (mg/l)	0.2	0.4	0.2

FENTON-LIKE PROCESSES: processes a circumneutral pH

COMPLEX FORMATION: SBO

	A (FORSUD)	B (CVDF110)	C (CVT230)
Aliphatic carbon	43	31	37
Amine	10	8	7
Methoxy	4	-	-
Alkoxy	10	20	14
Anomeric carbon	3	7	4
Aromatic	10	16	13
Phenolic carbon	2	6	5
Phenoxy	1	2	2
Carboxylic	7	9	12
Amide	9	1	1
Carbonilic	1	-	5
Lipophilic/hydrophilic ratio	9.3	5.3	3.6
Aliphatic/aromatic ratio	3.3	1.3	1.8
E ₂ /E ₃ ratio	3.83	2.31	2.38

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Application of Photo-Fenton Processes at circumneutral pH

FENTON-LIKE PROCESSES: processes a circumneutral pH: SBO

APPLICATION EXAMPLE

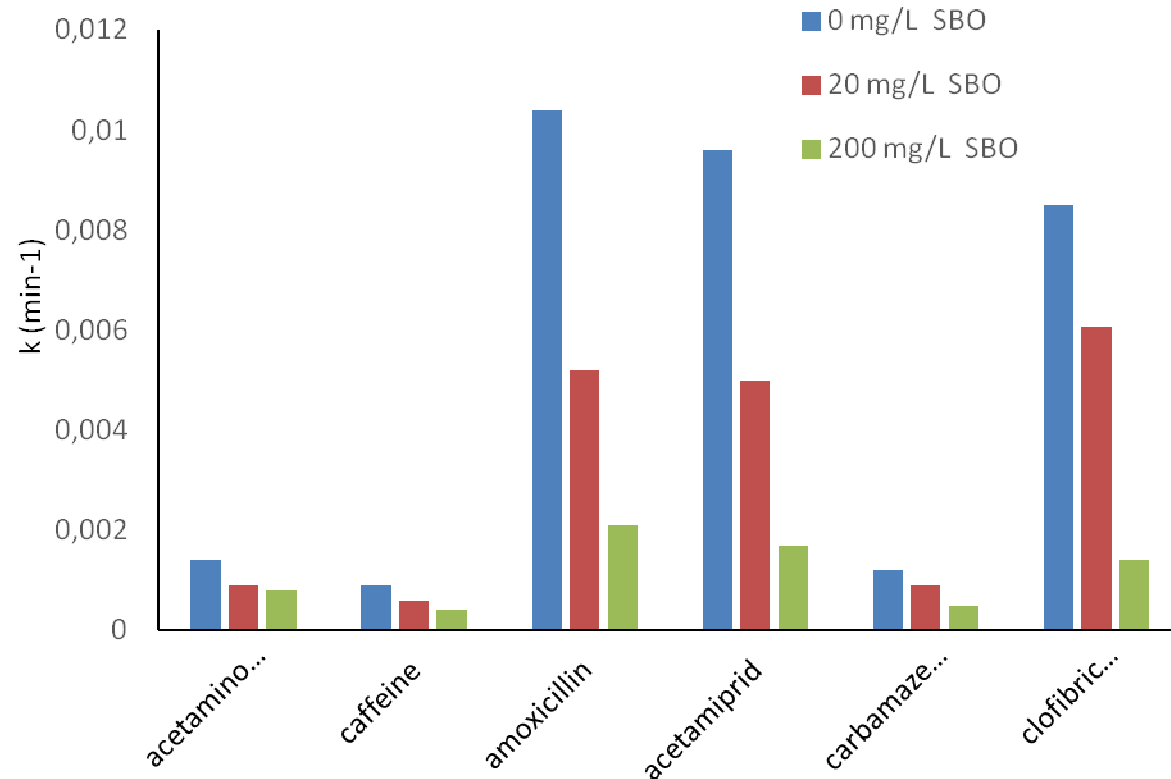
sensitizing effect photolysis

EP(5 mg·L⁻¹) + BOS

Results

Higher SBO concentration resulted in lower k values

The screen effect due to the brown color of SBOs might explain this fact

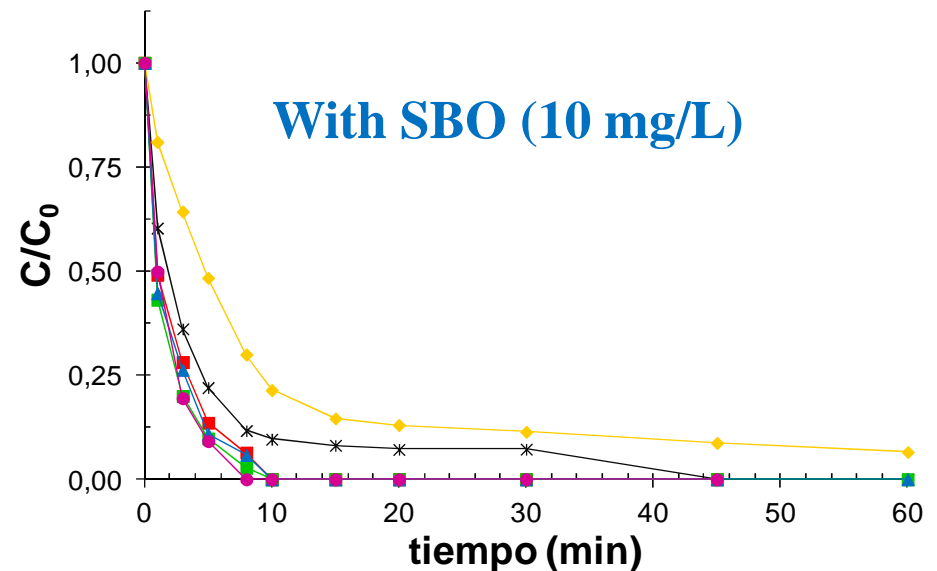
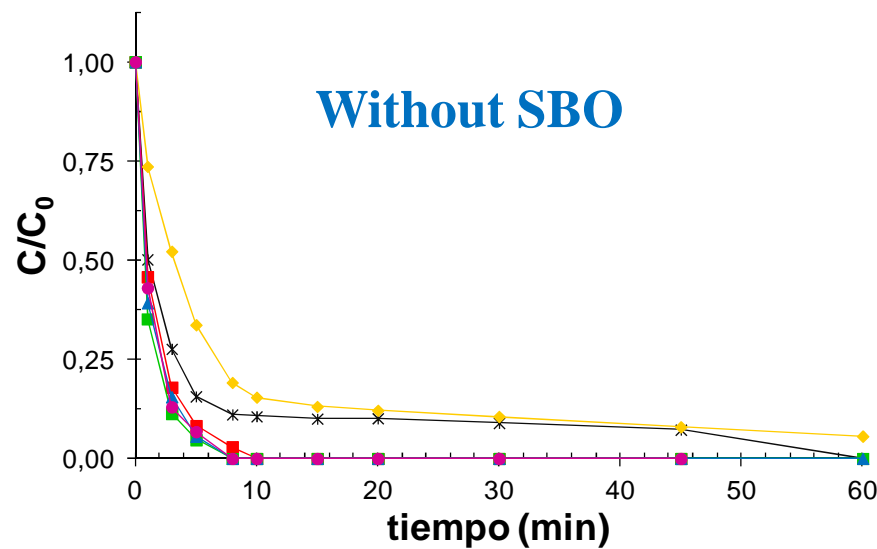


FENTON-LIKE PROCESSES: processes a circumneutral pH: SBO

APPLICATION EXAMPLE : photo-Fenton pH 2,8

EP (5 mg·L⁻¹) + Fe II (5 mg·L⁻¹) + H₂O₂ (75 mg·L⁻¹)

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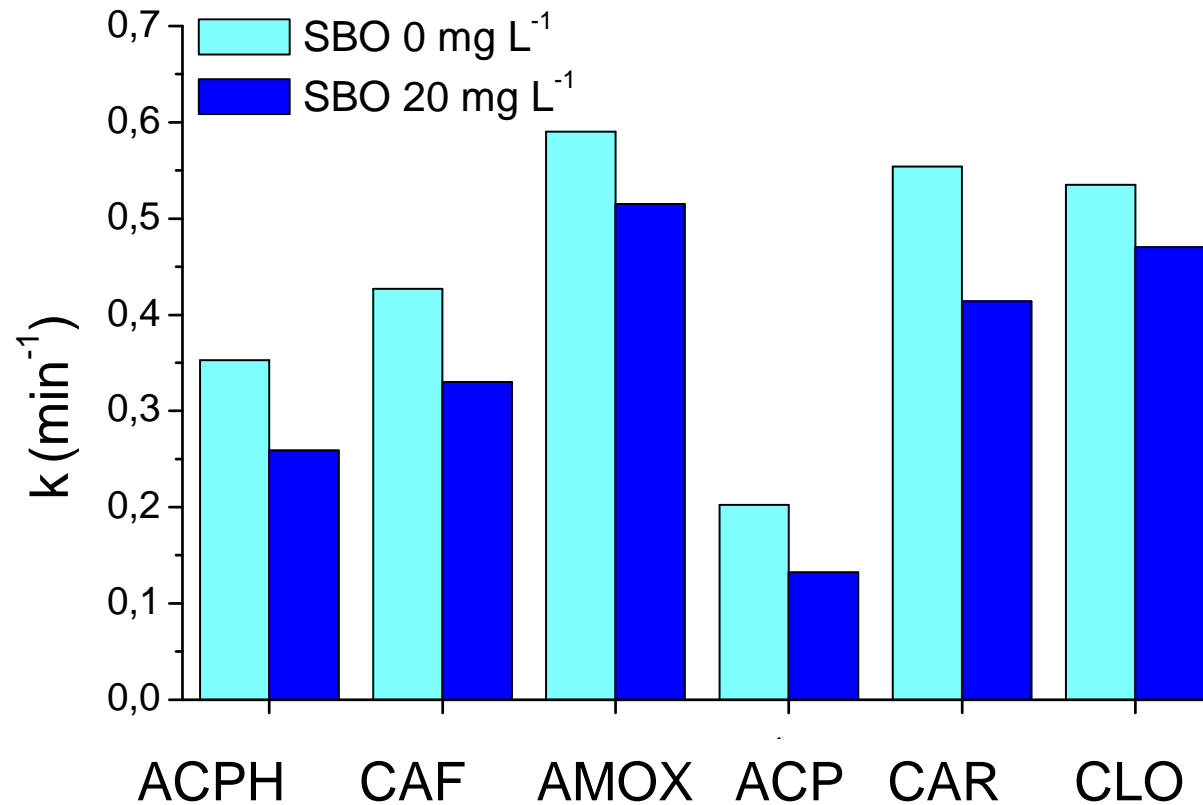
- * Acetaminophen
- Caffeine
- Amoxiciline
- ◆ Acetamidrid
- ▲ Carbamacepine
- Clofibric Acid

FENTON-LIKE PROCESSES:

processes a circumneutral pH: SBO

photo-Fenton

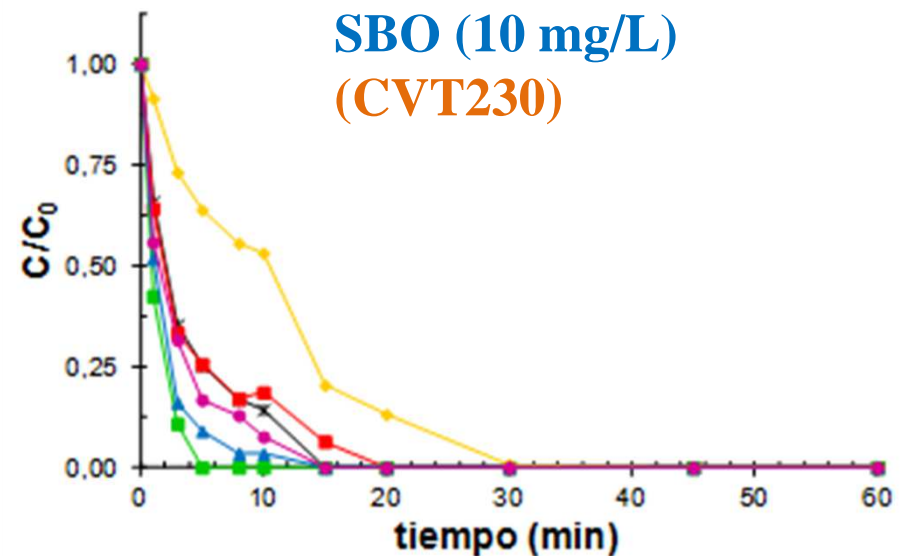
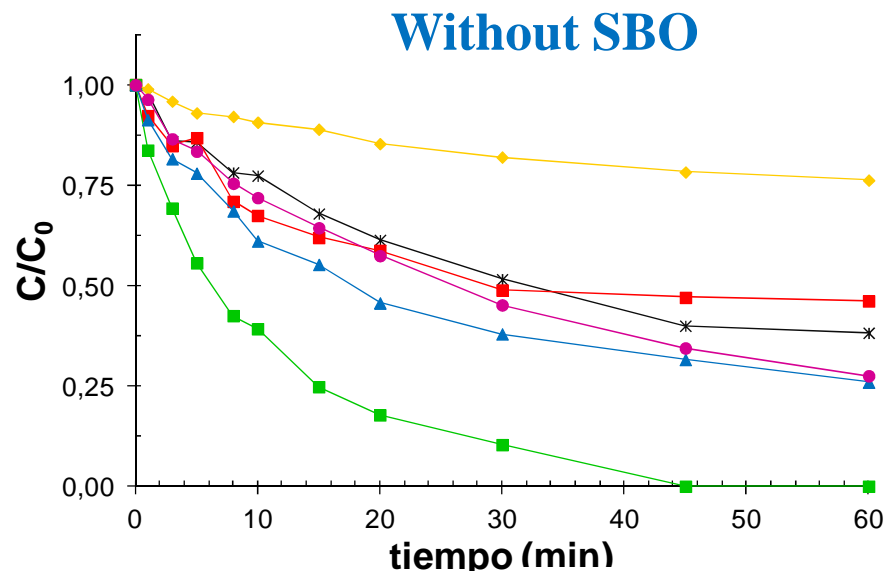
pH 2,8



FENTON-LIKE PROCESSES: processes a circumneutral pH: SBO

APPLICATION EXAMPLE photo-Fenton pH 5,3

EP (5 mg·L⁻¹) + Fe II (5 mg·L⁻¹) + H₂O₂ (75 mg·L⁻¹)



* Acetaminophen ■ Caffeine ■ Amoxiciline
◆ Acetamidrid ▲ Carbamacepine ● Clofibric Acid

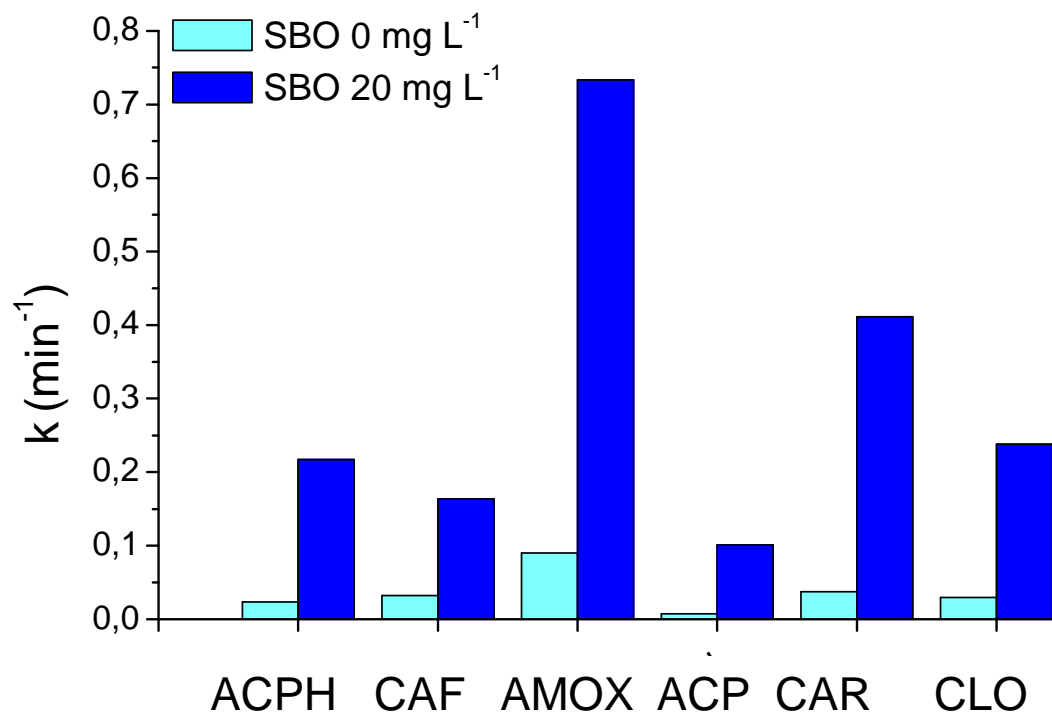
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FENTON-LIKE PROCESSES:

processes a circumneutral pH: SBO

APPLICATION EXAMPLE photo-Fenton pH 5,3

EP (5 mg·L⁻¹) + Fe II (5 mg·L⁻¹) + H₂O₂ (75 mg·L⁻¹)



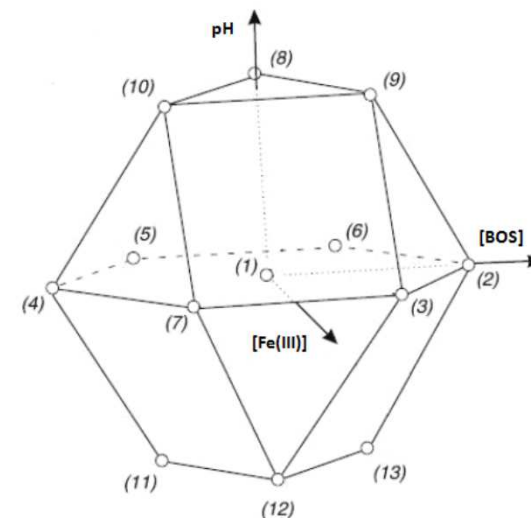
FENTON-LIKE PROCESSES:

processes a circumneutral pH: SBO

DOEHLERT

3 Factores	Niveles	MIN	MAX	Incremento
[SBO] mg/L	7	15	25	1,7
[Fe ³⁺] mg/L	5	2	6	1
pH	3	3	7	2

Exp.	[Fe (III)]	[SBO]	pH
1	4	20	5
1'	4	20	5
1''	4	20	5
2	6	20	5
3	5	25	5
4	2	20	5
5	3	15	5
6	5	15	5
7	3	25	5
8	5	21,7	7
9	3	21,7	7
10	4	16,7	7
11	3	18,3	3
12	5	18,3	3
13	4	23,3	3



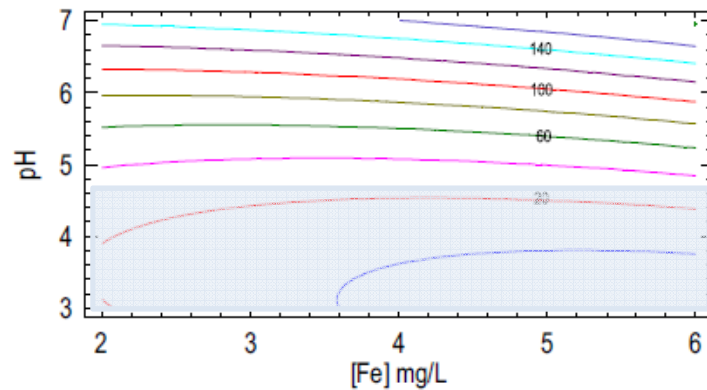
Application of Photo-Fenton Processes at circumneutral pH

FENTON-LIKE PROCESSES: processes a circumneutral pH: SBO

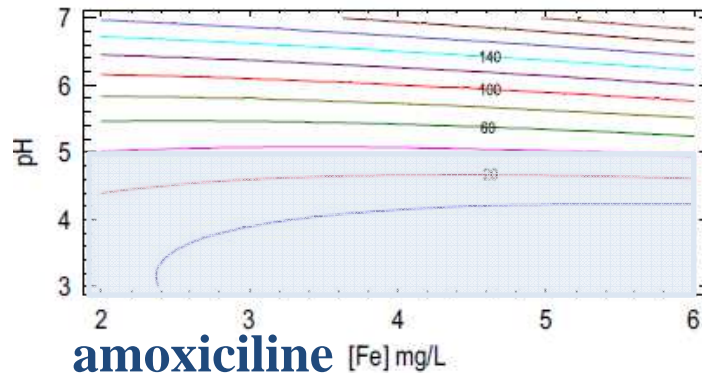
EXPERIMENTAL DESIGN DOEHLERT PHOTO-FENTON BOS
Time to remove 50% with BOS (CVT230)

The photo-Fenton reaction can be extended to pH values close to 5, without too remarkable loss of efficiency

[SBO] = 15 mg L⁻¹

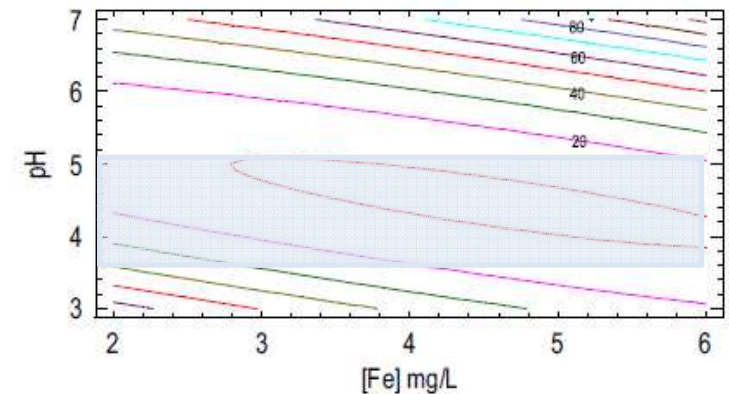
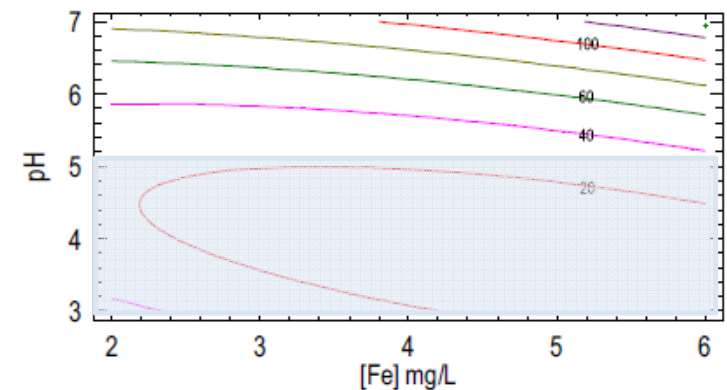


carbamazepine



amoxiciline [Fe] mg/L

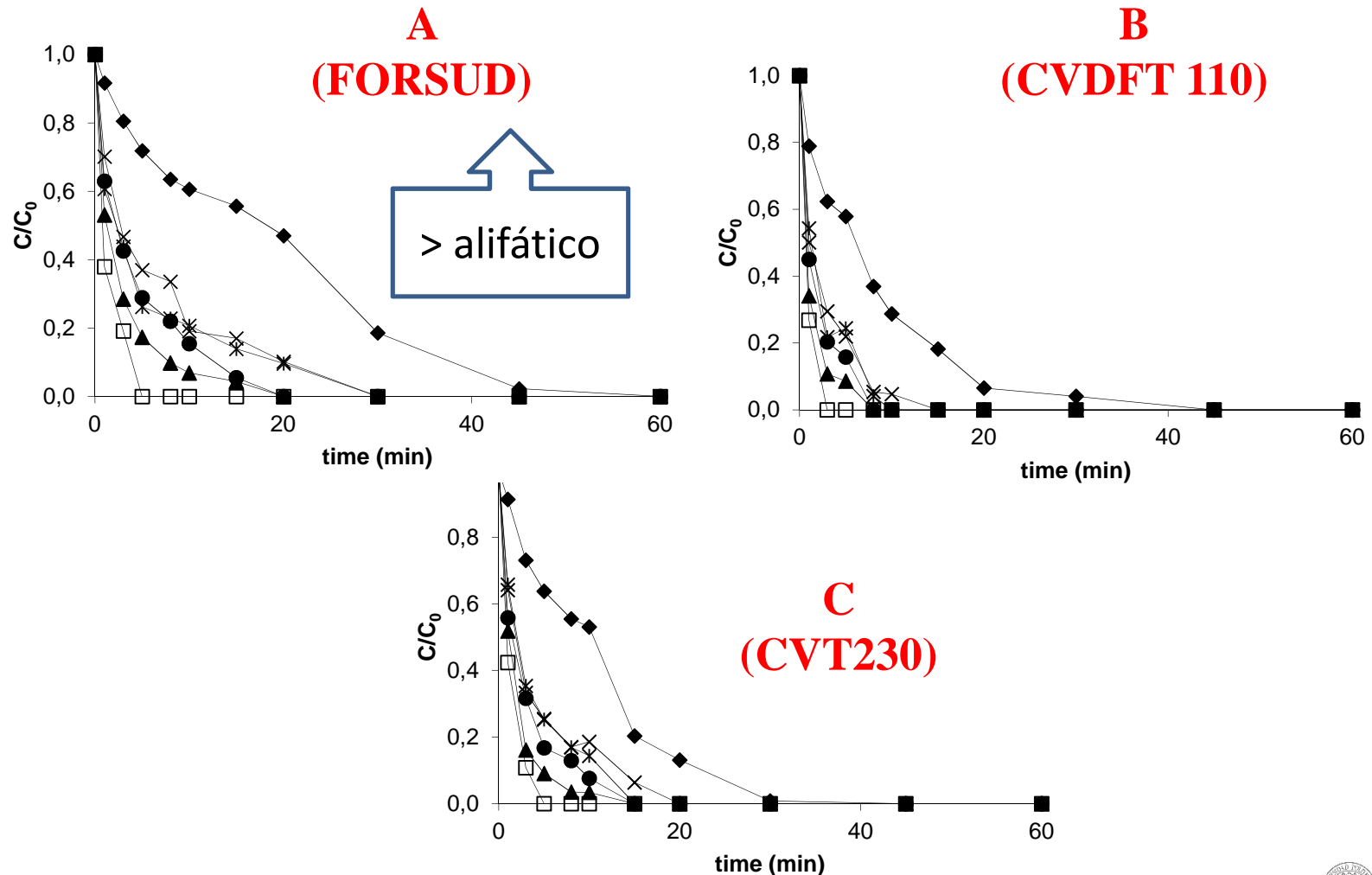
[SBO] = 25 mg L⁻¹



Application of Photo-Fenton Processes at circumneutral pH

FENTON-LIKE PROCESSES: processes a circumneutral pH: SBO

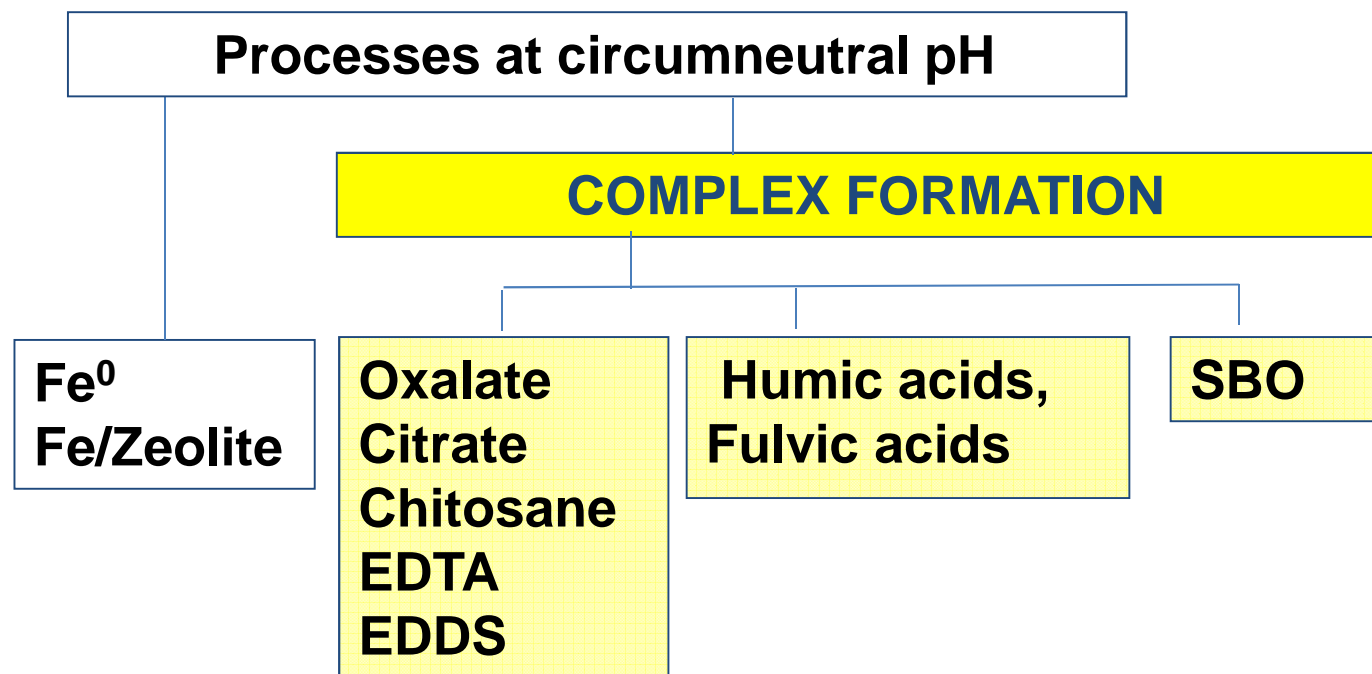
EFFECT OF DIFFERENT SBO



CONCLUSSIONS:

processes a circumneutral pH

- Existen numerosos estudios que demuestran que es posible aplicar procesos tipo foto-Fenton en condiciones próximas a la neutralidad con buenos porcentajes de degradación de contaminantes y de detoxificación de efluentes.





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1ª Jornada Técnica sobre procesos de oxidación avanzada en el ciclo integral del agua



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