Integrated Advanced Oxidation Processes used for the treatment of synthetic aqueous solutions containing organic micropollutants

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Sections

- Context - Micropollutants – long-term hazard to ecosystems
- Recently implemented technologies for MPs removal
- Efficiency of Ozonation and AC/Ozonation Coupling
- Objectives
- Main results
- Conclusions

Micropollutants

Households products: detergents, paints

Pharmaceutical products

Veterinary pharmaceuticals

Dyes and microplastics from textiles

Cosmetics

Pesticides

> 3000 chemicals are used as human pharmaceuticals

Micropollutants

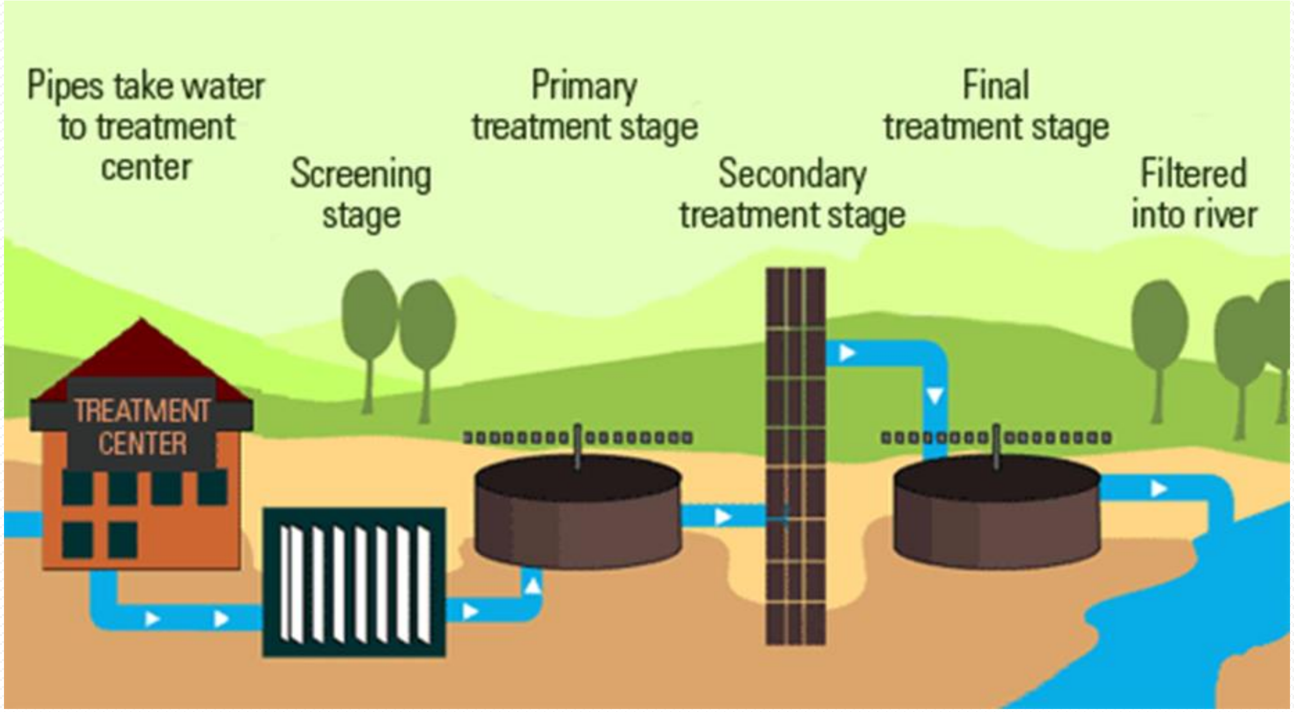
- > 100’000 synthetic compounds are registered in the EU
- About 30’000 synthetic compounds are in daily use
- Several hundred pesticides
- > 8000 chemicals as food supplements
- > 3000 chemicals are used as human pharmaceuticals

Micropollutants - sources

- Micropollutants end up in wastewater treatment plants (WWTPs).

Pharmaceuticals in WWTPs:

- 70% due to household use.
- 20% from livestock farming.
- 5% from hospital effluent.
- 5% from non-specific sources.



Micropollutants -effects of prenatal and early postnatal exposure

Doi: 10.1111/j.1742-7843.2007.00114.x
Journal compilation © 2007 Nordic Pharmacological Society. *Basic & Clinical Pharmacology & Toxicology*, **102**, 73–75

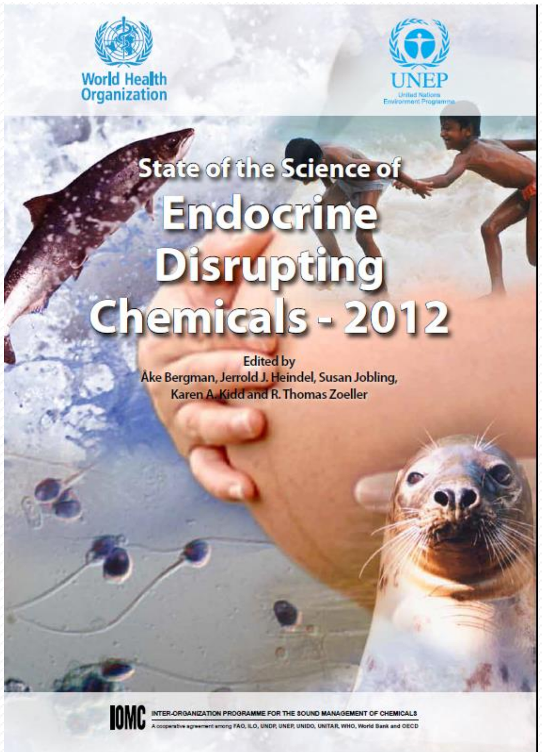
The Faroes Statement: Human Health Effects of Developmental Exposure to Chemicals in Our Environment

Philippe Grandjean[1,2], David Bellinger[2], Åke Bergman[3], Sylvaine Cordier[4], George Davey-Smith[5], Brenda Eskenazi[6], David Gee[7], Kimberly Gray[8], Mark Hanson[9], Peter van den Hazel[10], Jerrold J. Heindel[8], Birger Heinzow[11], Irva Hertz-Picciotto[12], Howard Hu[13], Terry T-K Huang[14], Tina Kold Jensen[1], Philip J. Landrigan[15], I. Caroline McMillen[16], Katsuyuki Murata[17], Beate Ritz[18], Greet Schoeters[19], Niels Erik Skakkebæk[20], Staffan Skerfving[21] and Pal Weihe[22]

- The ***Faroes Statement*** warned of fetal exposure to toxic substances resulting in **lifelong effects**: reduced immune system, obesity, diabetes, cancers, ADHD, Parkinson's, Alzheimer's.
- Old toxicology "the dose makes the poison" must be replaced by "**the timing makes the poison**".

Prevention should **<u>not</u>** await definitive evidence of causality when delays in decision-making would lead to the propagation of toxic exposures and harmful consequences.

The *State of the Science of Endocrine Disrupting Chemicals (EDCs)—2012*



Key concerns:

- Human and wildlife health depends on the ability to reproduce and develop normally (healthy endocrine system).
- Global rise in many hormonal diseases and disorders are also due to EDCs.
- Many endocrine-related diseases and disorders are on the rise.
- ~800 chemicals are known or suspected to be capable of interfering with hormone receptors, synthesis or conversion.

Source: A. Bragman et al. State of the Science of Endocrine Disrupting Chemicals – 2012, An assessment of the state of the science of endocrine disruptors prepared by a group of experts for the UNEP and WHO (2013)

The *State of the Science of Endocrine Disrupting Chemicals (EDCs)—2012*

Metabolic disorders:

•Obesity, diabetes and metabolic syndrome are potentially sensitive to EDCs ("obesogens").

•Exposures of animal models to a variety of chemicals during early development have been shown to result in weight gain

Immune function and diseases:

•EDCs are at least partially responsible for the rise in the development of immune-related disorders.

•Systemic inflammation, immune dysfunction and immune cancers such as lymphoma and leukemia in humans are associated with EDC exposures.

Neurodevelopmental disorders:

•Exposure to EDCs causes cognitive and behavioral deficits in humans

•involvement of thyroid hormone mechanisms in neurodevelopmental disorders in humans and wildlife

Bone disorders:

•decreased bone mineral density or increased risk of bone fractures

Female reproductive health:

•EdCs exposures can interfere with endocrine signaling of pubertal timing, fecundity and fertility and with menopause.

Evidence for endocrine disruption in humans and wildlife

Population declines:

•Wildlife species and populations continue to decline worldwide.

Hormone-related cancers:

•increase in incidence of endocrine-related cancers in humans

Male reproductive health:

•the incidence of testicular cancer has further increased in the European countries

•increases of genital abnormalities in babies and testis germ cell cancer in men

•The feminizing effects of estrogenic chemicals from sewage effluents on male fish is a widespread phenomenon.

Sex ratio imbalances:

•EDC-related sex ratio imbalances have been seen in wild fish and mollusks

•Result in fewer male offspring in humans

Thyroid-related disorders:

•The strength of evidence supporting a role for EDCs in disrupting thyroid function in wildlife adds credence to the hypothesis that this could occur in humans

Adrenal disorders:

•the hypothalamic– pituitary–adrenal (HPA) axis and the adrenal gland are targets for EDCs

Source: A. Bragman et al. State of the Science of Endocrine Disrupting Chemicals – 2012, An assessment of the state of the science of endocrine disruptors prepared by a group of experts for the UNEP and WHO (2013)

Pharmaceuticals in the environment—global occurrences*

Environmental Toxicology and Chemistry, Vol. 35, No. 4, pp. 823–835, 2016

PHARMACEUTICALS IN THE ENVIRONMENT—GLOBAL OCCURRENCES AND PERSPECTIVES

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(Submitted 27 February 2015; Returned for Revision 3 July 2015; Accepted 11 December 2015)

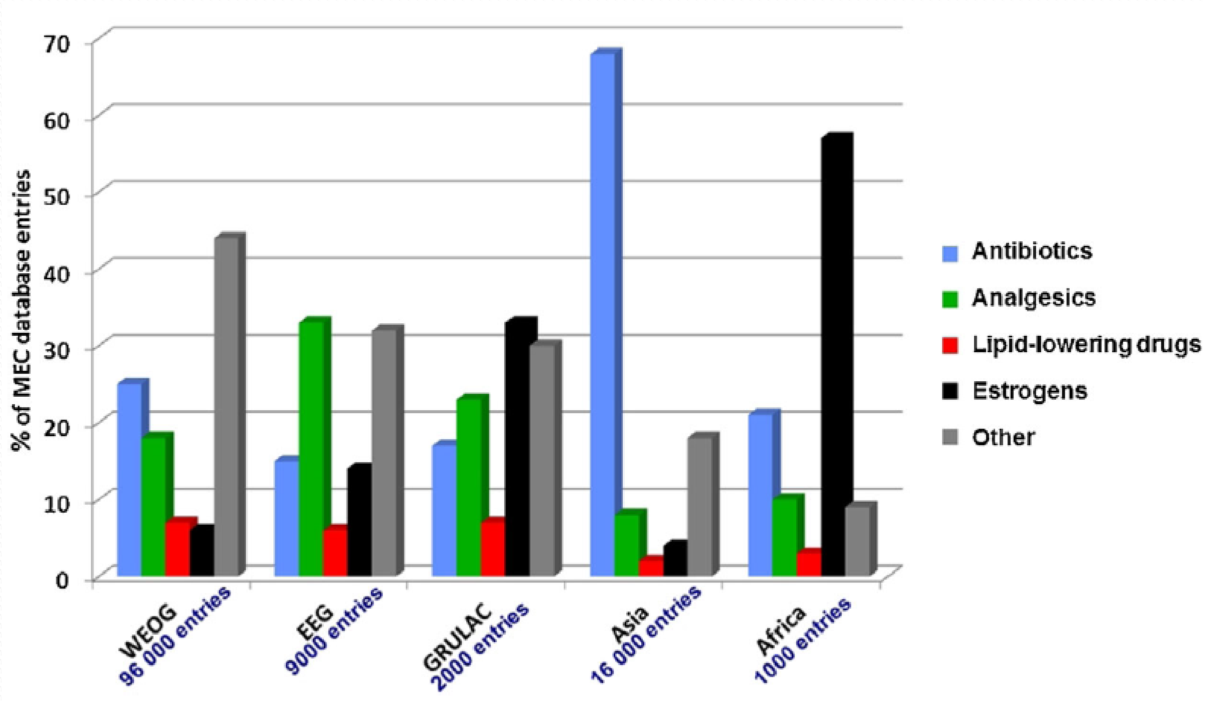
Source: Beek et al., Environmental Toxicology and Chemistry, 2016, 35(4) 823-835

Pharmaceuticals in the environment—global occurrences*

- In 2016, Beek & co reported a comprehensive literature review gathering results from 1016 original papers and 160 reviews.
- The authors collected measured environmental concentrations (**MECs**) for pharmaceuticals reported worldwide in surface water, groundwater, tap/drinking water, manure, soil, and other environmental matrices.
- Pharmaceuticals or their transformation products have been detected in the environment of 71 countries (from the 5 regions recognized by the United Nations).
- In total, 631 different pharmaceuticals were found at MECs above the detection limit.
- Sixteen substances were detected in each of the 5 UN regions.
- The anti-inflammatory drug diclofenac has been detected in environmental matrices in 50 countries, and concentrations found in several locations exceeded predicted no-effect concentrations
- The authors conclude that pharmaceuticals are a global challenge calling for approaches to prevent, reduce, and manage their entry into and presence in the environment.

Source: Beek et al., Environmental Toxicology and Chemistry, 2016, 35(4) 823-835

Pharmaceuticals in the environment—global occurrences*



- MEC = measured environmental concentration;
- EEG = Eastern Europe Group;
- GRULAC = Latin American and Caribbean States;
- WEOG = Western Europe and Others Group (North America, New Zeeland, Australia).

Source: Beek et al., Environmental Toxicology and Chemistry, 2016, 35(4) 823-835

Pharmaceuticals in the environment—global occurrences*

Table 1. Number of countries in each United Nations group in which positive detection of pharmaceutical substances in surface waters, groundwater, and/or tap or drinking water has been reported[a]

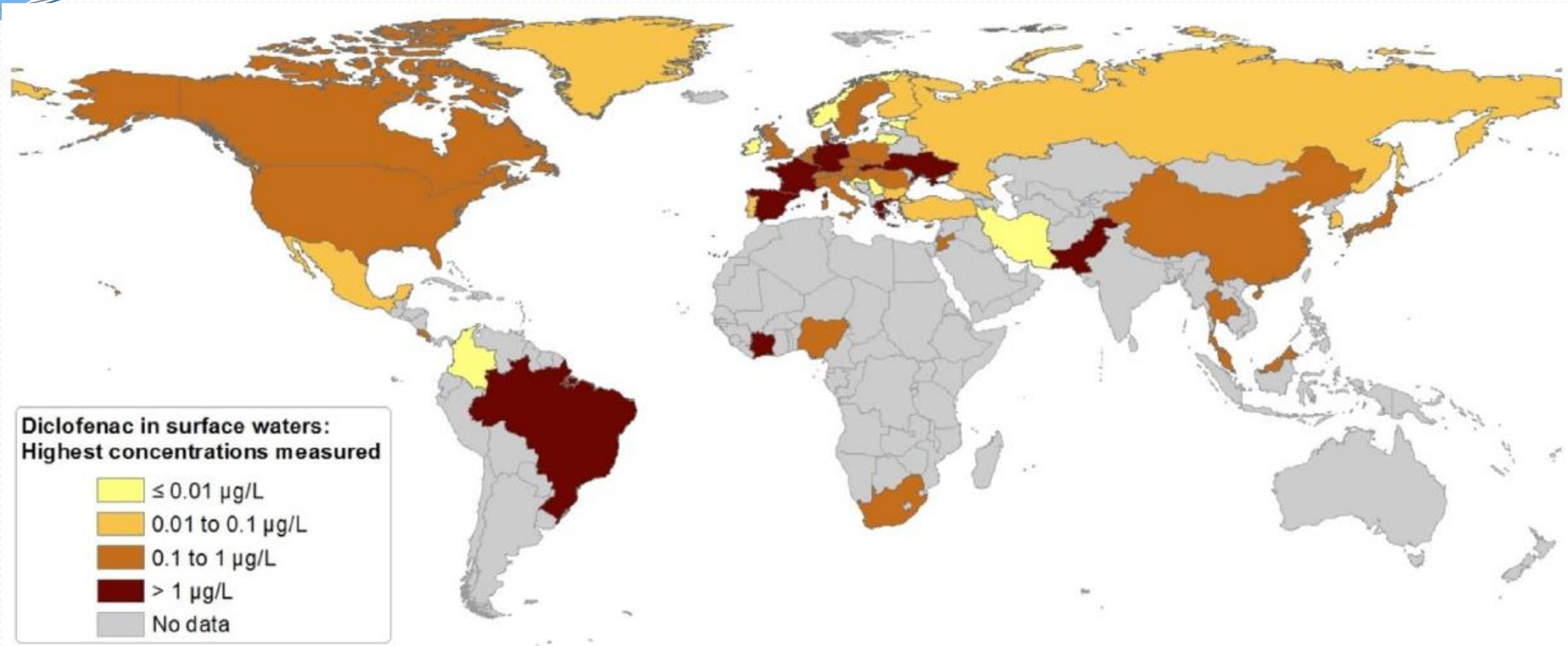
Pharmaceutical substance	Therapeutic group	Africa	Asia-Pacific	EEG	GRULAC	WEOG	Global
Diclofenac	Analgesics	3	8	13	3	23	50
Carbamazepine	Antiepileptics	3	6	13	2	24	48
Ibuprofen	Analgesics	3	8	10	2	24	47
Sulfamethoxazole	Antibiotics	5	9	10	2	21	47
Naproxen	Analgesics	2	8	10	2	23	45
Estrone	Estrogens	1	10	6	2	16	35
Estradiol	Estrogens	2	9	4	2	17	34
Ethinylestradiol	Estrogens	1	8	3	2	17	31
Trimethoprim	Antibiotics	2	9	3	2	13	29
Paracetamol	Analgesics	1	6	4	3	15	29
Clofibric acid	Lipid-lowering drugs	1	3	5	2	12	23
Ciprofloxacin	Antibiotics	1	5	1	2	11	20
Ofloxacin	Antibiotics	1	4	1	1	9	16
Estriol	Estrogens	1	1	2	1	10	15
Norfloxacin	Antibiotics	1	4	1	2	7	15
Acetylsalicylic acid	Analgesics	1	4	1	2	7	15

[a]These 16 substances are the only ones that have been found in each region.
EEG = eastern Europe; GRULAC = Latin America and Caribbean; WEOG = western Europe and others.

- Residues of 16 pharmaceutical substances were detected in the surface, drinking, and groundwater of all the UN regions.
- Other 4 pharmaceutical substances have been found in the environment nearly as often as diclofenac: carbamazepine (antiepileptic), sulfamethoxazole (antibiotic), ibuprofen, and naproxen (both analgesics).
- Other therapeutic groups that have been detected in the environment include estrogens, such as estrone and ethinylestradiol.

Source: Beek et al., Environmental Toxicology and Chemistry, 2016, 35(4) 823-835

Pharmaceuticals in the environment—global occurrences*



Maximum diclofenac concentrations reported in surface waters in each country

- Diclofenac is the most often detected pharmaceutical in the environment.
- Maximum concentrations of >1 **mg/L** often occur downstream of sewage-treatment plants in densely populated areas.

Source: Beek et al., Environmental Toxicology and Chemistry, 2016, 35(4) 823-835

Micropollutants – “Rhine” solutions

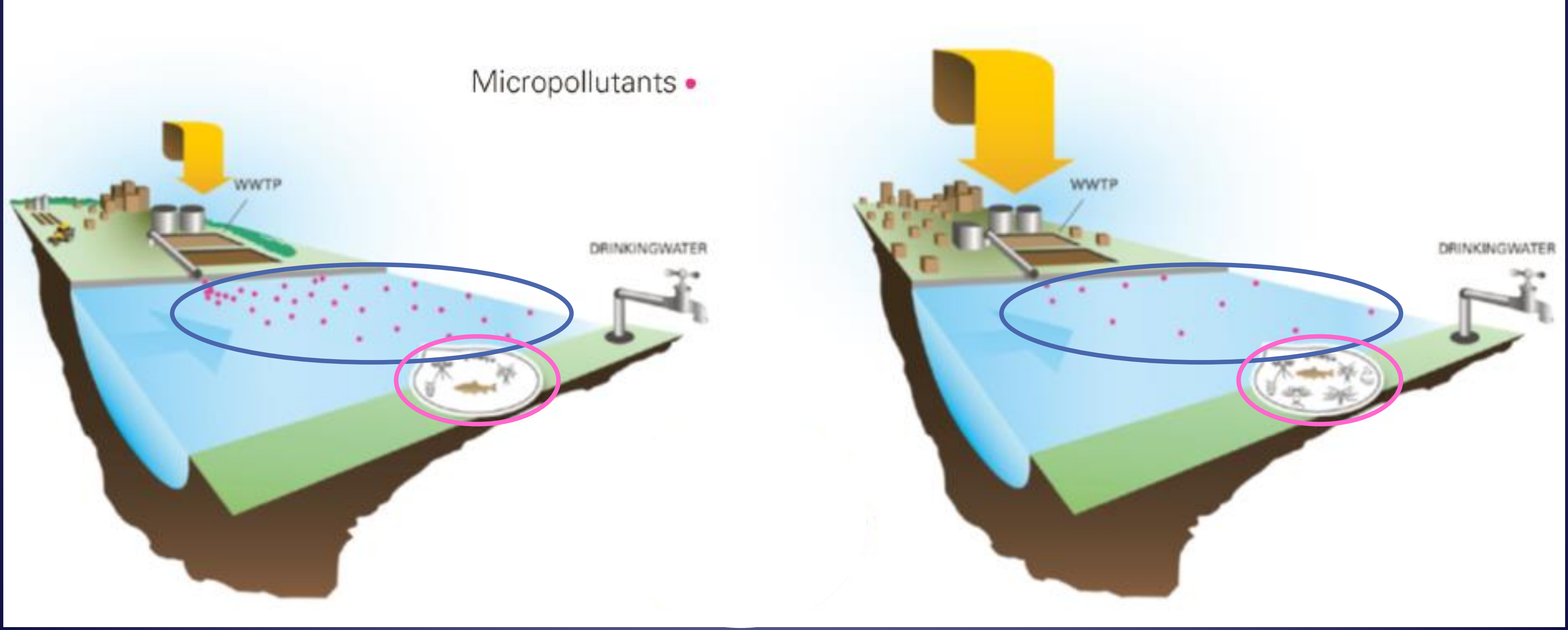
- Since 1950 Germany and France, and afterwards Luxemburg, the Netherlands and Switzerland have founded the **International Commission for the Protection of the Rhine** (ICPR).
- **October 2013** -Communiqué of the 15th conference of Rhine Ministers, Basel.
- An agreement was made toward a significant **reduction** in the discharge of persistent MPs derived from different sources.

Source: https://www.iksr.org/

Micropollutants -
THE SWISS National STRATEGY for upgrading WWTPs

March 2014 – water protection act

Wastewater treatment towards MPs removal



Source: R.I.L. Eggen et al., Reducing the Discharge of Micropollutants in the Aquatic Environment: The Benefits of Upgrading Wastewater Treatment Plants, Environ. Sci. Technol., 2014, 48 (14), pp 7683–7689

Micropollutants - THE SWISS National STRATEGY for upgrading WWTPs

Sound scientific and technical basis:
2002-2007: Swiss National Research Program (NRP) 50, “Endocrine Disruptors: Relevance to Humans, Animals and Ecosystems”
2006-2010, project “Strategy MicroPoll”: quantifying loads and toxicities of MPs from WWTP effluents

Based on Broad Societal and Political Acceptance: Reports in the Swiss media on the occurrence of pharmaceuticals in streams and drinking water raised public awareness and stimulated public concern

Technically Feasible: Most MPs are removed by O_3 and PAC adsorption followed by filtration.

Manageable: A limited number of proxy compounds are monitored by HPLC-MS/MS. Removal performance can be controlled by the ozone concentration or PAC dose.

Pragmatic: 100 out of 700 WWTPs are considered for upgrading based on the anticipated MP load and capacity for dilution in the receiving water.

Adaptable in time: The upgrade will be realized over 20 years, allowing enough time to select the optimal technologies.

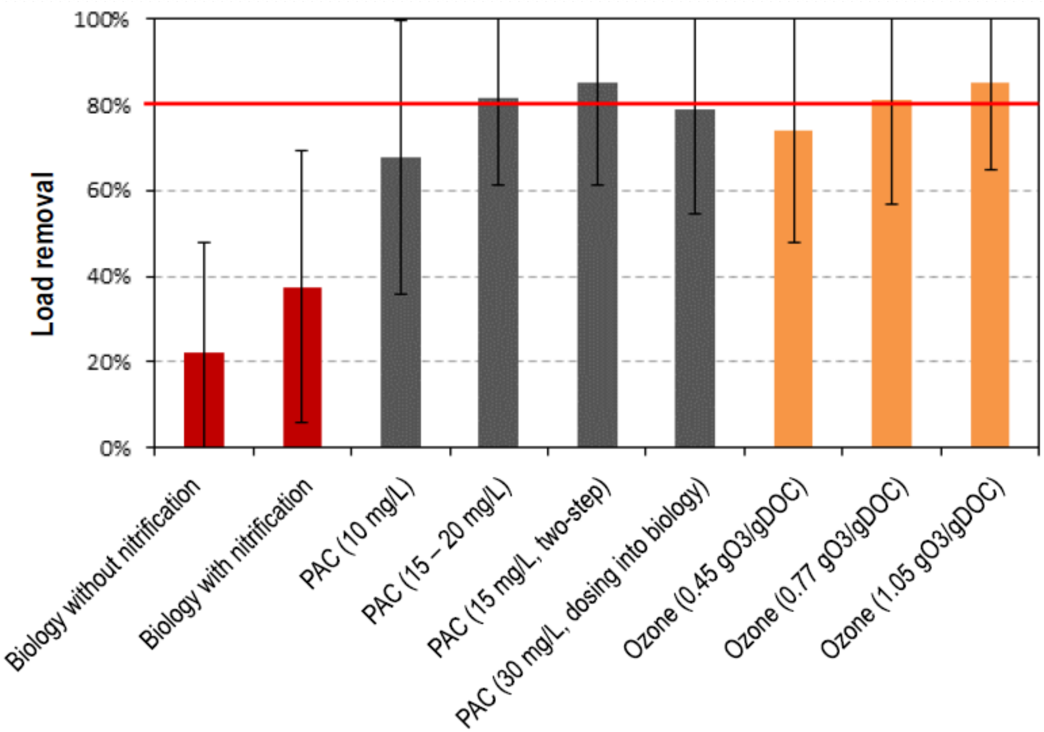
Financially Feasible: Are expected 20% higher costs for WWTPs serving >80 000 persons, and up to 50% for WWTPs of lower capacity. Investment costs: 1.3 billion $ (yearly costs: 0,130 billion $)

Source: R.I.L. Eggen, et al. Reducing the Discharge of Micropollutants in the Aquatic Environment: The Benefits of Upgrading Wastewater Treatment Plants, Environ. Sci. Technol., 2014, 48 (14), pp 7683–7689

Recently implemented technologies for MPs removal

- Two technologies considered at **real-scale**:
  - Ozonation
  - Powdered Activated Carbon
- PAC removes MPs
- Ozonation transforms MPs
- Ozonation leads also to partial disinfection
- <u>**No**</u> full mineralization of target compounds is achieved

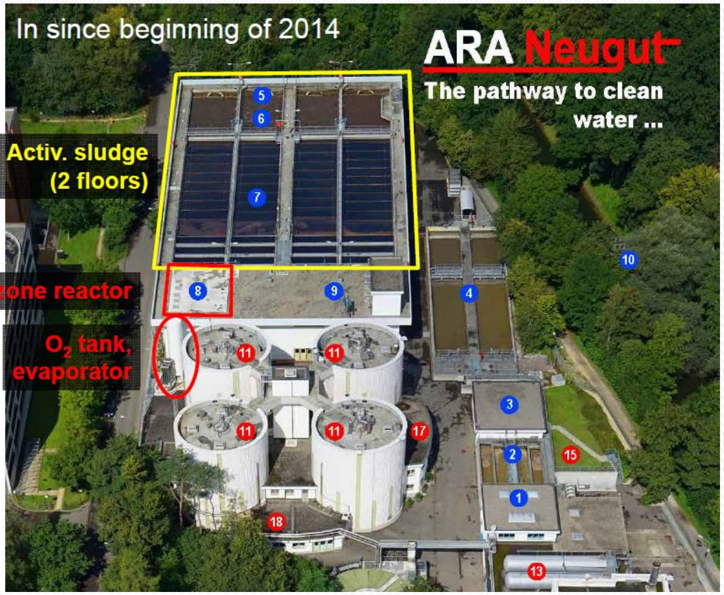
Comparison of MPs removal by Biological treat., PAC and Ozonation



Source: Pilot experiments Eawag, Kloten/Opfikon, Lausanne, Regensdorf

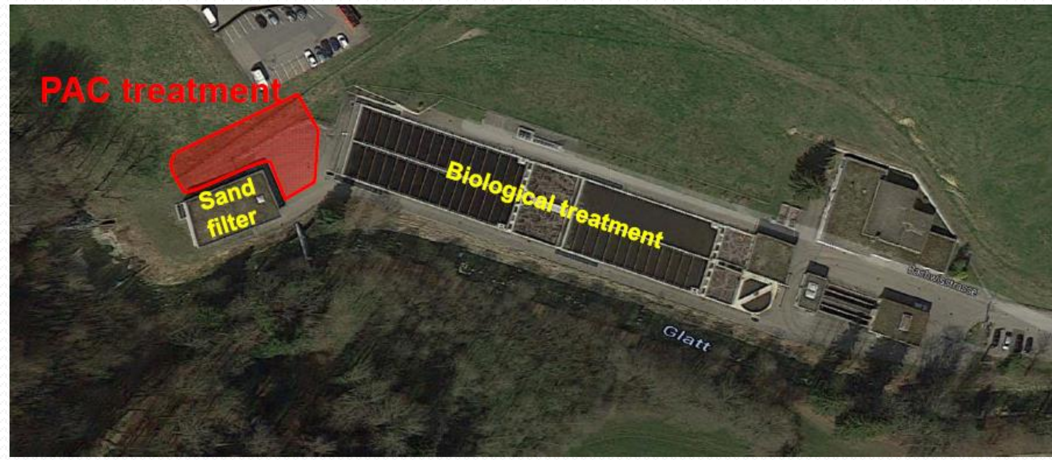
Recently implemented technologies for MPs removal

AraNeugut WWTP, Dübendorf, Switzerland



In 2014 the WWTP in Dubendorf has been provided with an ozonation treatment step

WWTP Bachwis, Herisau, Switzerland



Set into operation in May 2015

Influent: municipal + textile industry - industrial compounds with low reactivity towards Ozone

PAC unit prior to sand filter

Source: A. Joss et al., Micropollutants in Swiss Waters – Measures in Wastewater Treatment Plants2015, Swiss Tech Convention Center, EPFL Lausanne

Efficiency of Ozonation and AC/Ozonation Coupling - prior work -

- Principle : Simultaneous use of O_3 and AC
- Action : Synergistic effects

In bulk liquid

- **1. Direct degradation**
  Oxydation by O_3
- **2. Indirect degradation**
  Oxydation by $HO^{\bullet}$

On AC Surface

- **3. Adsorption** on AC surface
- **4. Oxydation of adsorbed compounds**
- **5. Interaction O_3/AC :** $HO^{\bullet}$ generation

O_3 : oxidant

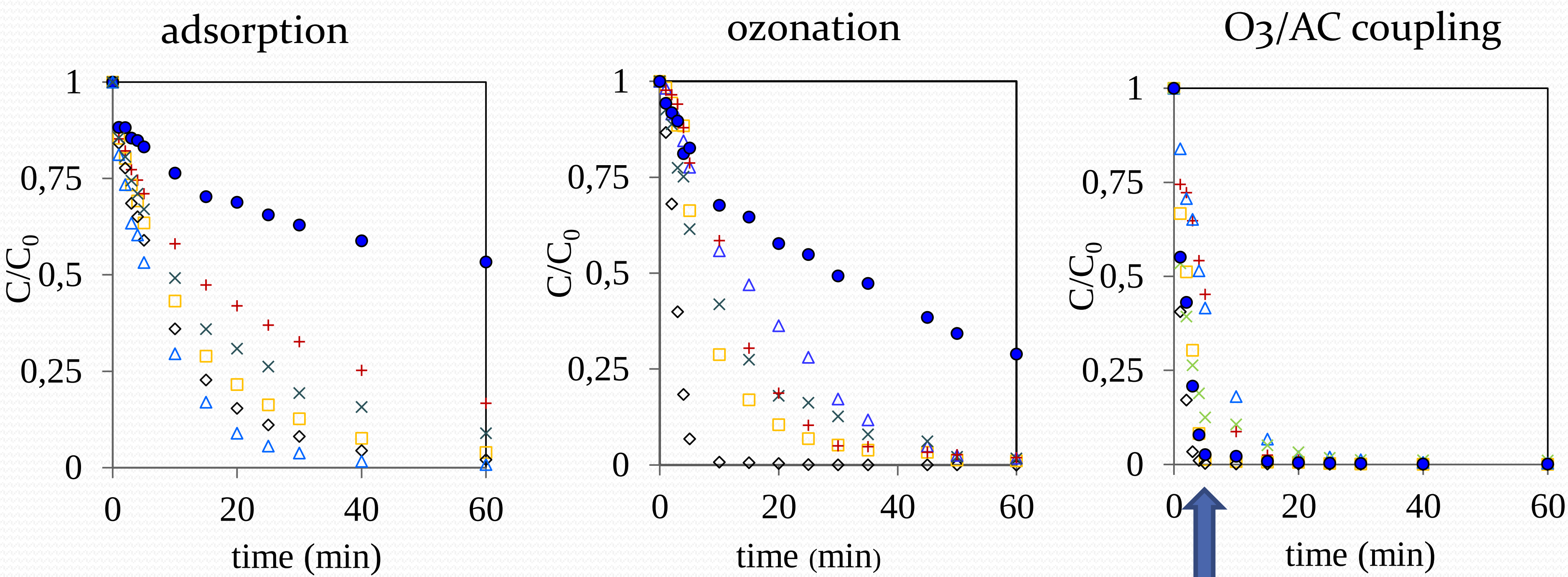
AC: adsorbent, reactional support, radical initiator and promotor

Model pharmaceutical micropollutants

Molecules	Class	pKa	Solubility (g/L) (25°C)	Molecular Volume (Å^3)
Carbamazepine	Anti-epileptic	13.9	0.018	210.32
Sulfamethoxazole	Antibiotic	1.7; 5.6	0.6	204.59
Ketoprofen	Analgesic	4.1 - 4.4	0.051	233.79
Fluoxetine hydrochloride	Antidepressive	9.5 – 10.1	14	274.37
Metoprolol tartrate	Betablocker	9.7	50	274.25
Terbutaline hemisulfate	Bronchodilatator	8.8	213	222.28

Criteria : different pharmaceutical classes, weakly removed by conventional methods

Efficiency of Ozonation and AC/Ozonation Coupling



terbutaline

metoprolol

fluoxetine

sulfamethoxazole

carbamazepine

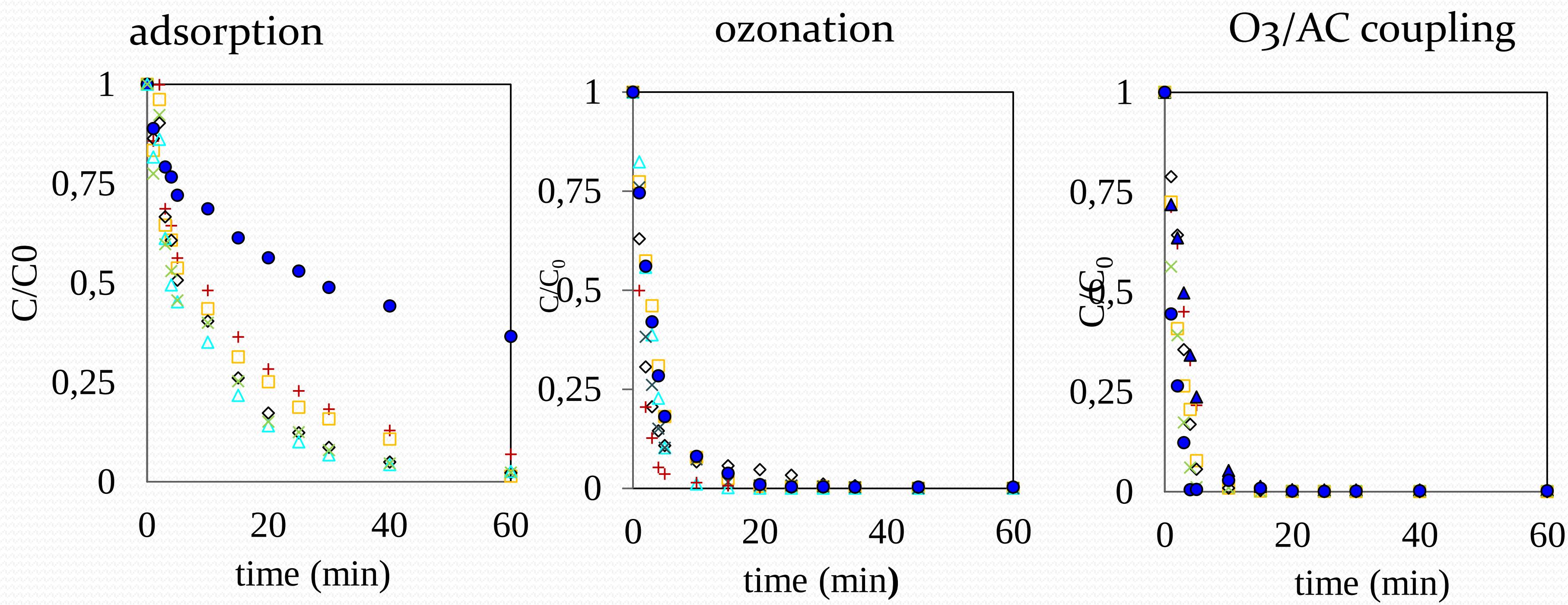
ketoprofen

Complete removal of CBZ after 3min

pH 3

C_i = 18 mg/L

Efficiency of Ozonation and AC/Ozonation Coupling



terbutaline

metoprolol

fluoxetine

sulfamethoxazole

carbamazepine

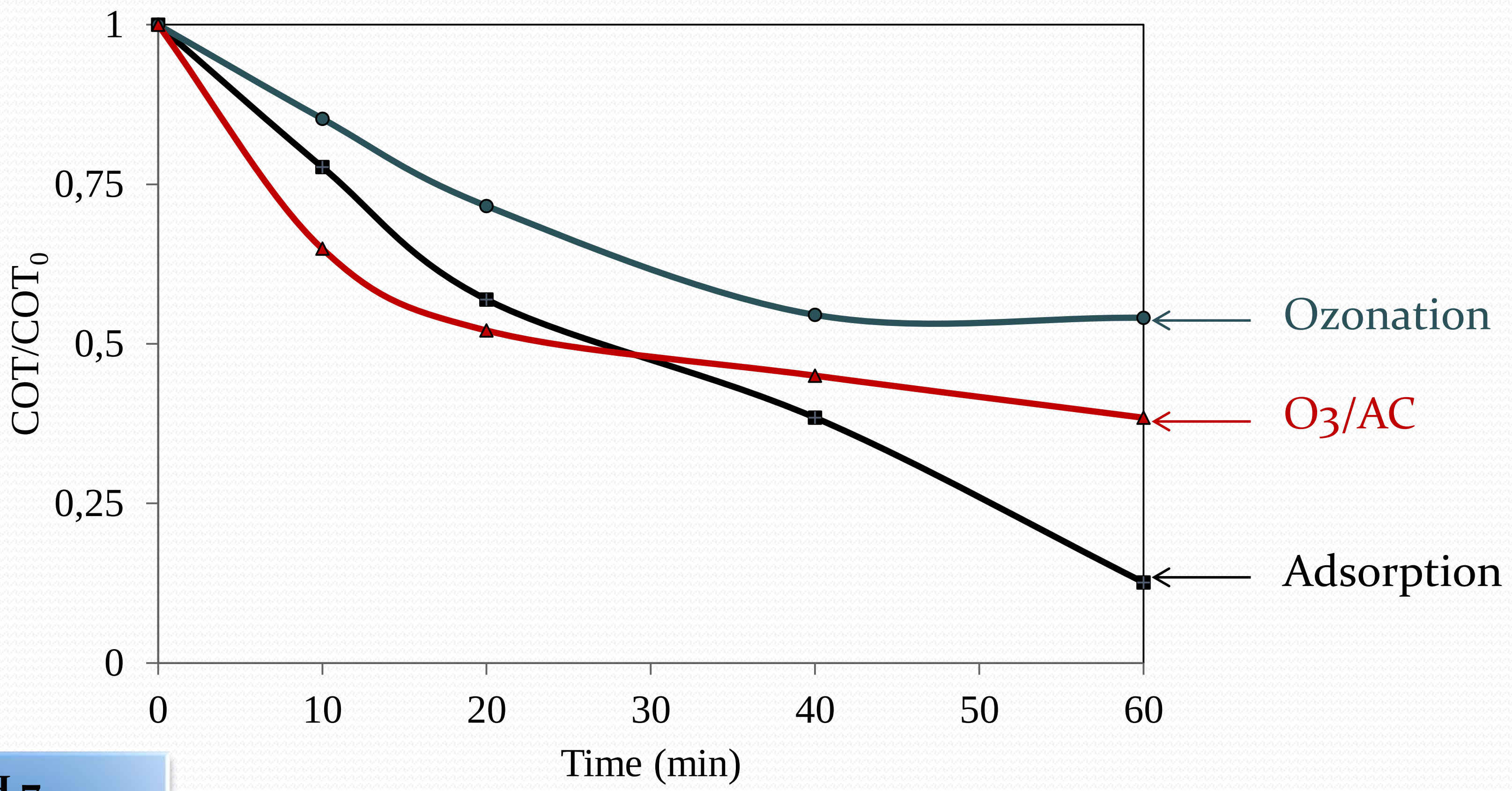
ketoprofen

pH 7

C_i = 18 mg/L

Efficiency of Ozonation and AC/Ozonation Coupling

TOC evolution



pH 7

C_i = 18 mg/L

O3/AC : Mineralization close to 60 %

Efficiency of Ozonation and AC/Ozonation Coupling

Conclusions and perspectives

- **Adsorption**
  - No by-product
  - Slow
  - Depends on pollutant
  - Pollutant load not destructed
  - Regeneration of AC
- **Ozonation**
  - Faster than adsorption
  - Toxic by-products poorly removed
  - Depends on pollutant
  - Depends on operating conditions
- **O_3/AC coupling**
  - Fast pharmaceuticals removal
  - No influence of operating conditions
  - No influence of compounds
  - Advanced mineralization can be obtained
  - Toxic by-products still present
  - Full mineralization still not achieved

Developing a new advanced treatment technique for micropollutants removal from water and wastewater

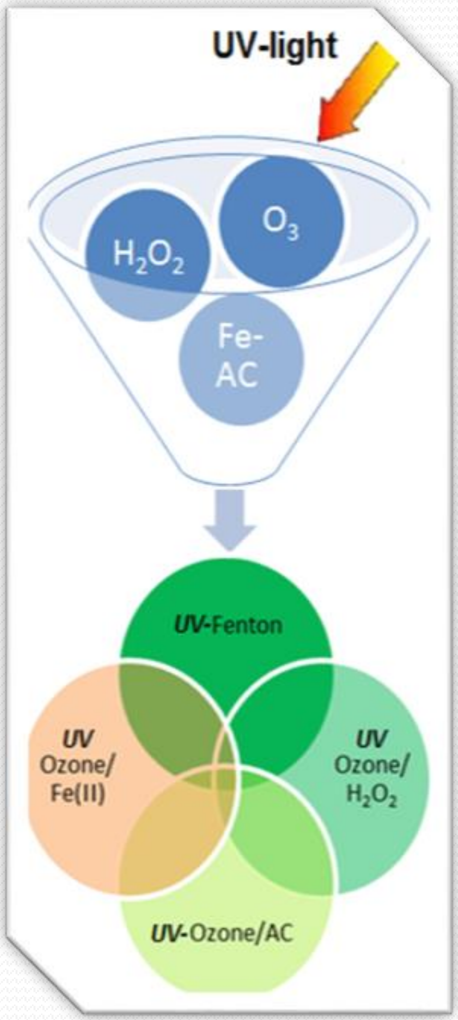


Photo-Fenton-Peroxone System

- **Principle : Simultaneous use of UV-light, Fe-AC, O_3 and H_2O_2**
- **Action : Synergetic effects**

Objective: Degradation of micropollutants (pharmaceuticals) commonly found in municipal wastewater

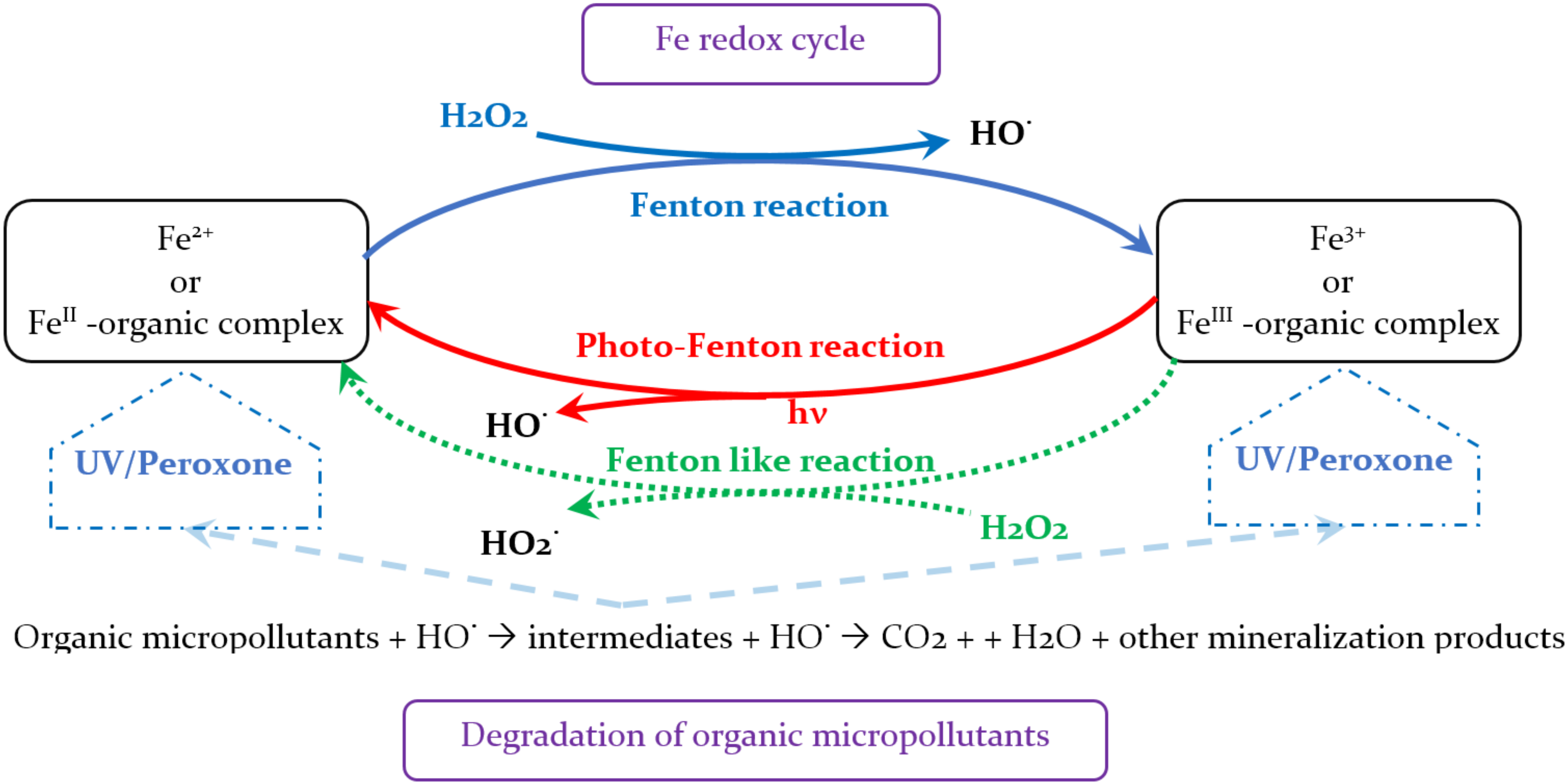
Degradation of MPs

In bulk liquid → **Peroxone process**

On Fe-AC → **Fenton process**

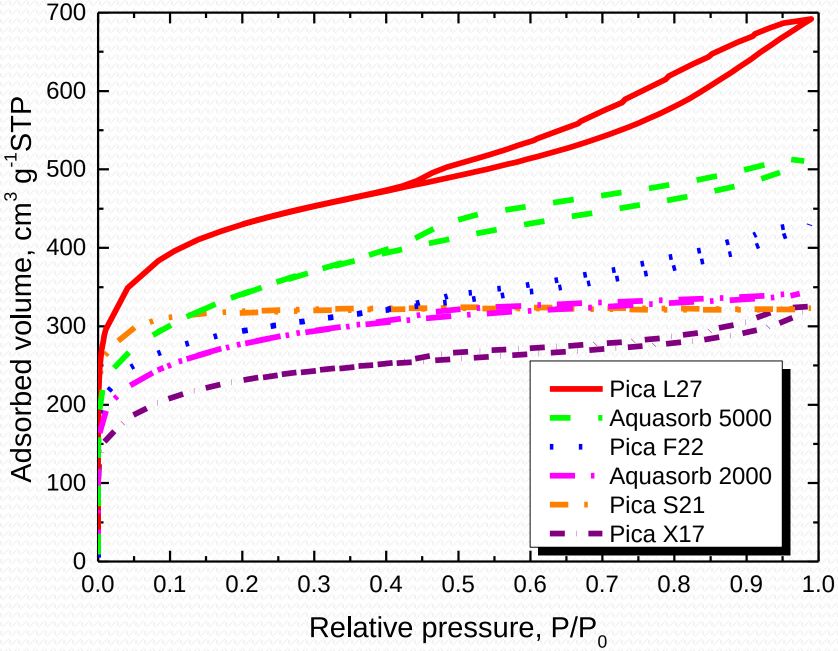
Applications: upgrade of wastewater treatments plants, valorization of agricultural waste as activated carbon

Developing a new advanced treatment technique for micropollutants removal from water and wastewater



Characterization of Activated Carbon Materials - textural properties -

N2 adsorption isotherms (77 K) -Micromeritics ASAP 2020



L27, AS5000, F22 X17: Hysteresis indicates the presence of mesopores

S21, AS2000: micropores

L27, F22: well developed porosity

AS5000: average porosity

AS2000, X17: low porosity

Textural properties of several commercial activated carbons

Material adsorbant	W_o (cm^3 g^{-1})	L_o (Å)	S_{ext} (m^2 g^{-1})	S_{micro} (m^2 g^{-1})	S_{total} (m^2 g^{-1})
Pica *L27*	0,57	18,5	444	616	1060
Aquasorb *AS5000*	0,44	15,4	257	571	828
Pica *F22*	0,40	12,7	256	630	886
Aquasorb *AS2000*	0,36	13,5	75	533	608
Pica *S21*	0,40	8,7	43	920	963
Pica *X17*	0,33	14,3	157	462	619

Characterization of Activated Carbon Materials - chemical properties -

pH_{PZC} : Point of zero charge – pKa distribution method
Functional groups: Boehm method

L27: numerous surface groups

AS2000, AS5000, S21, X17:

few surface groups

L27: acid surface

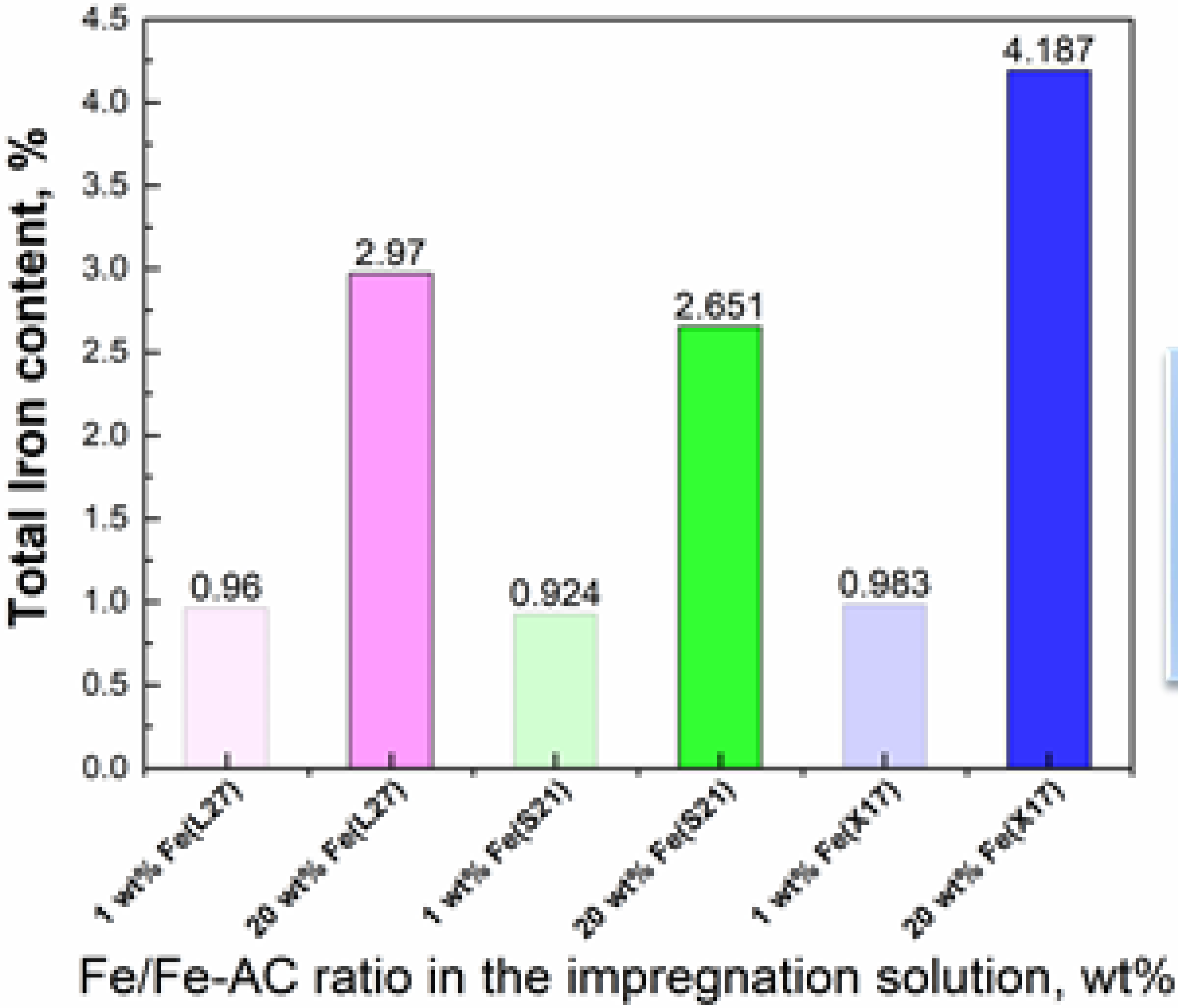
F22, S21: neutral surface

AS2000, AS5000, X17: alkaline surface

Chemical properties of several commercial activated carbons

	***Carboxilic groups* (meq g⁻¹)**	***Phenolic groups* (meq g⁻¹)**	***Lactone groups* (meq g⁻¹)**	***Acid groups* (meq g⁻¹)**	***Alkaline groups* (meq g⁻¹)**	***pH_{PZC}***
Pica *L27*	0.81	0.30	0.46	1.57	0.18	**3.0**
Aquasorb *AS5000*	0.11	0.16	0.05	0.32	0.38	**8.3**
Pica *F22*	0.13	0.14	0.05	0.32	0.26	**7.5**
Aquasorb *AS2000*	0.03	0.07	0.02	0.12	0.16	**9.3**
Pica *S21*	0.05	0.30	0.03	0.35	0.33	**7.4**
Pica *X17*	0.15	0	0.02	0.20	0.85	**9.7**

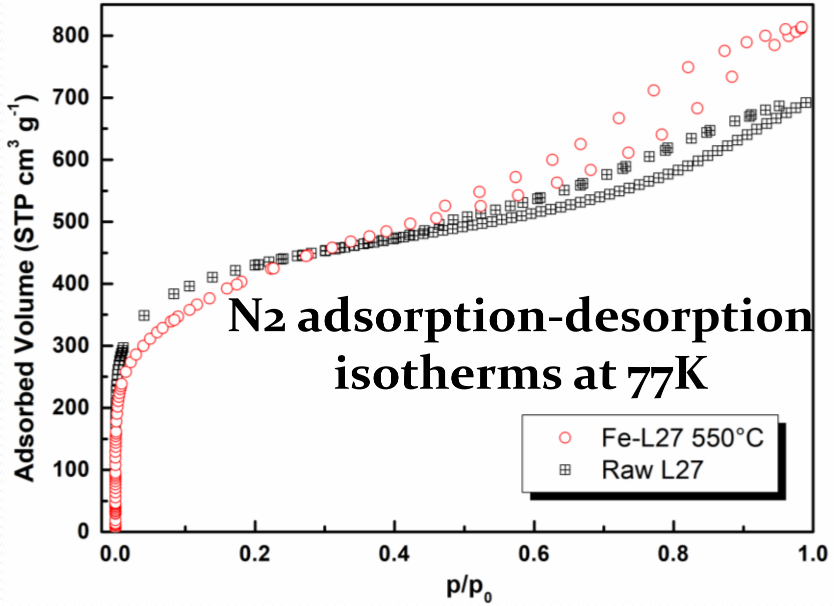
Characterization of Activated Carbon Materials
- total Iron content -



Iron content increases with the augmentation of the Fe amount contacted with AC

Total iron content increases in the order: Fe-S21<Fe-L27<Fe-X17

Characterization of Fe-Activated Carbons - textural properties-



N2 adsorption-desorption isotherms at 77K

Textural properties of Fe-impregnated ACs

Material adsorbant	W_o ($cm^3\ g^{-1}$)	L_o (Å)	S_{ext} ($m^2\ g^{-1}$)	S_{micro} ($m^2\ g^{-1}$)	S_{total} ($m^2\ g^{-1}$)
raw L27	0.57	18.5	444	616	1060
Fe-L27, T 550°C	0.50	26.4	748	379	1127
raw S21	0.47	9.7	18	969	987
Fe-S21, T 550°C	0.57	9.7	85	1175	1260
raw X17	0.29	15.1	130	384	514
Fe-X17,T 550°C	0.31	16.2	192	383	575

Fe-L27 – developed mesoporosity (micropores partially blocked by Fe impregnation).

Fe-S21 – developed microporosity

Fe-X17 – developed mesoporosity

Experimental set-up – batch photoreactor

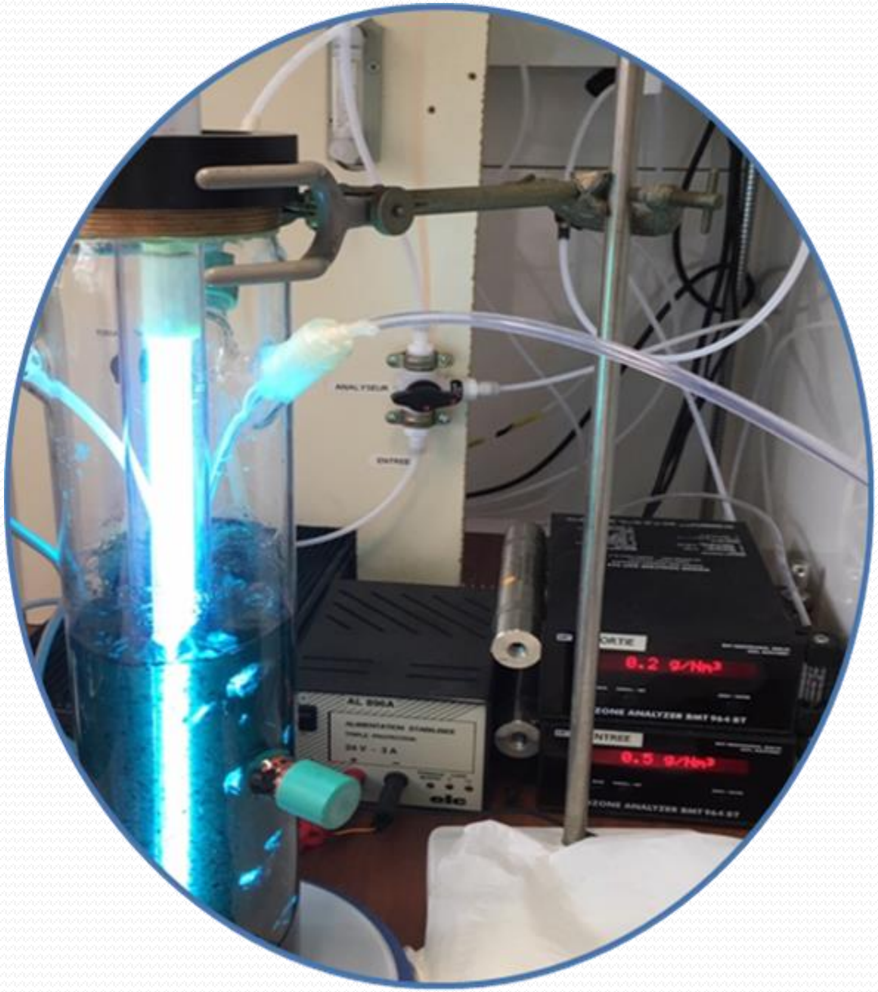
- Initial pH values: 3, 7 and 9

- Monosolute: C = 200 mg/L
- Mixture: C = 18 mg/L

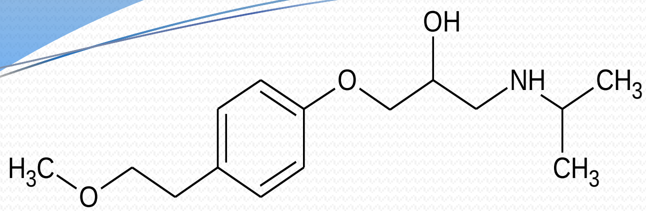
- $[O_3] \approx 20\ g/Nm^3$

- catalyst : 0.4 g/L

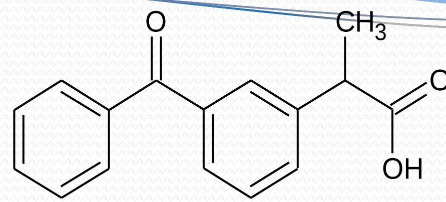
- $[H_2O_2] = 250$ mg/L



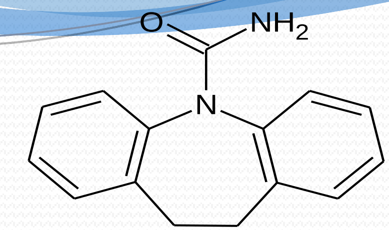
Model pharmaceutical micropollutants



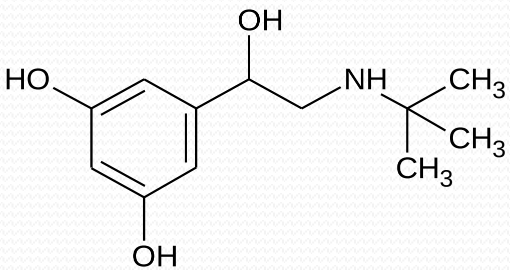
Metoprolol (MTP)
Betablocker
Solubility (g/L) = 50



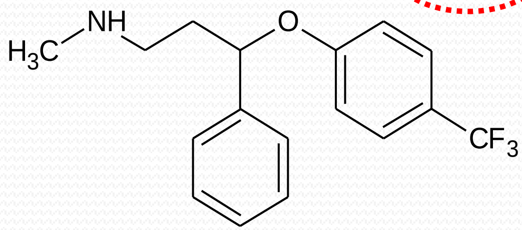
Ketoprofene (KTP)
Analgesic
Solubility (g/L) = 0.051



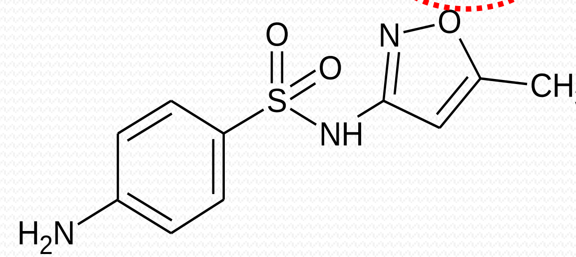
Carbamazepine (CBZ)
Anti-epileptic
Solubility (g/L) = 0.018



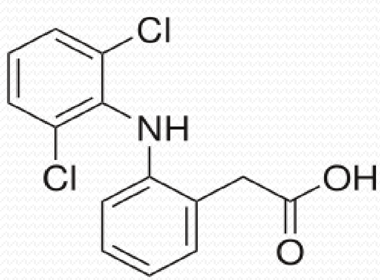
Terbutaline (TBL)
Bronchodilatator
Solubility (g/L) = 213



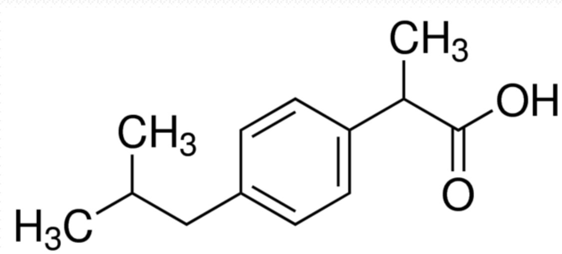
Fluoxetine (FXT)
Antidepressive
Solubility (g/L) = 14



Sulfamethoxazole (SFX)
Antibiotic
Solubility (g/L) = 0.6



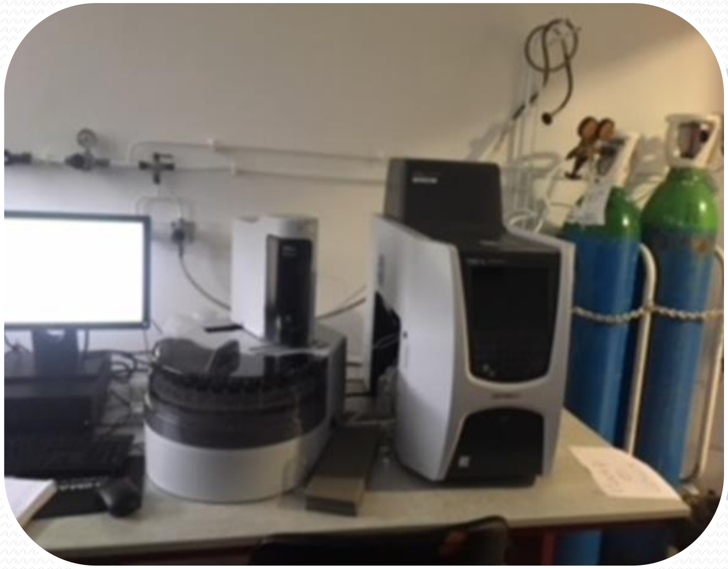
Diclofenac (DCF)
Analgesic
Solubility (g/L) = 50



Ibuprofen (IBP)
Analgesic
Solubility (g/L) = 100

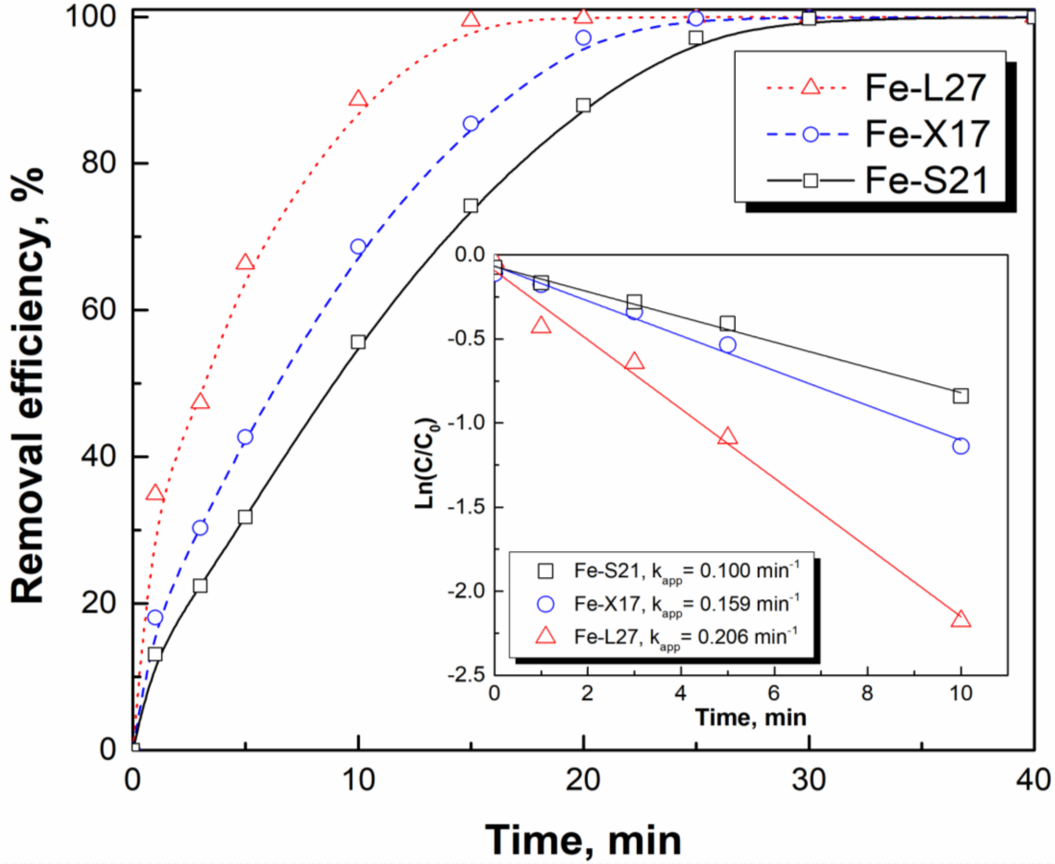
Analytical methods

- HPLC – High Performance Liquid Chromatographie



- TOC – Total Organique Carbon

Photocatalytic tests
- effect of AC matrix -



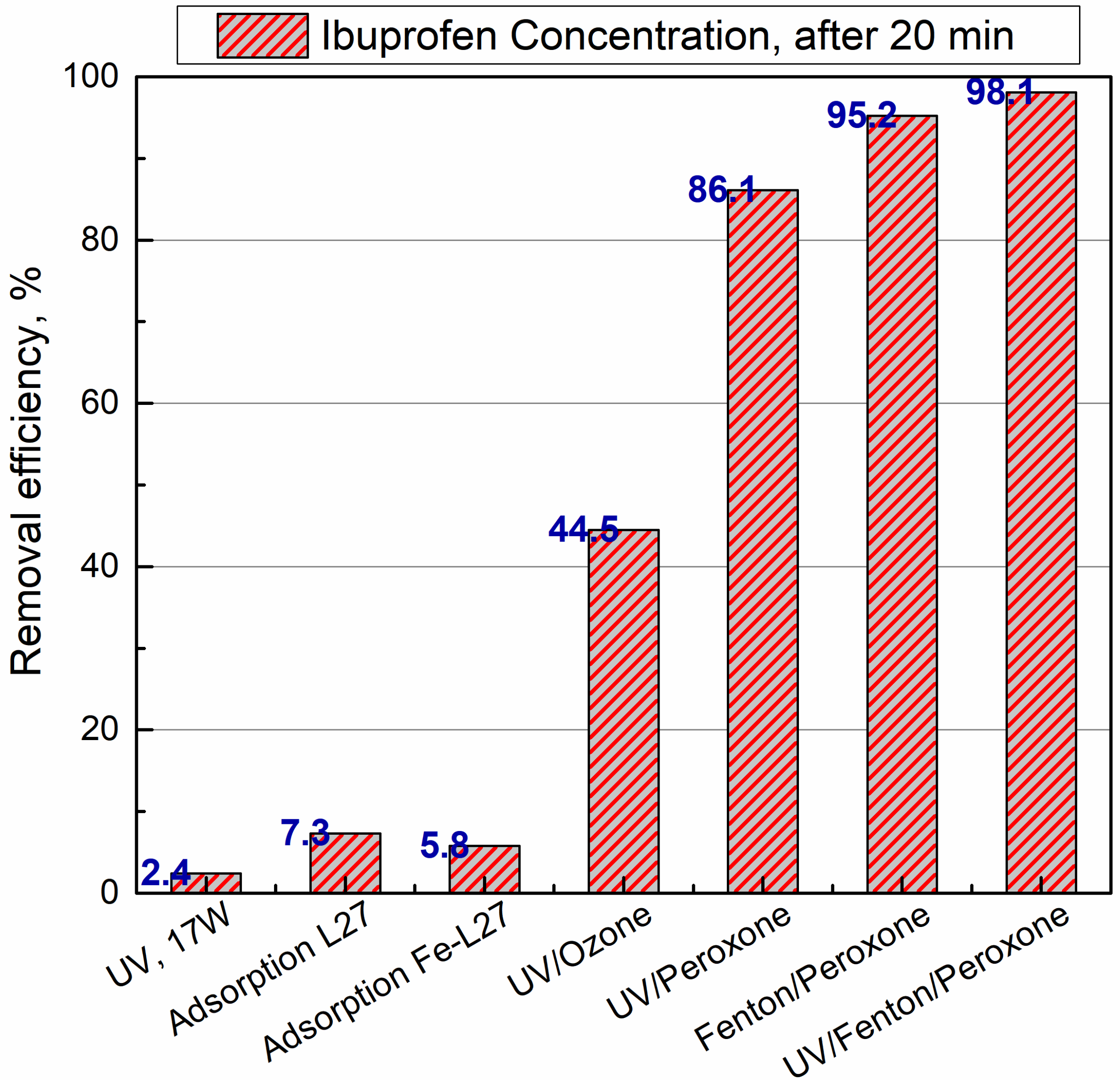
Ibuprofen degradation tests in presence of Fenton-like catalysts prepared with GAC support of different pH_{PZC}. Inset plot: pseudo-first order kinetics

S21 provided the lowest degradation rate (complete removal efficiency in 30 min).

X17 - complete IBP removal efficiency after 25 min.

L27 presents considerable acidic groups and has a well-developed porosity structure – complete IBP removal efficiency after 15 min.

Treatment tests
- in relation to IBP concentration -



pH 7

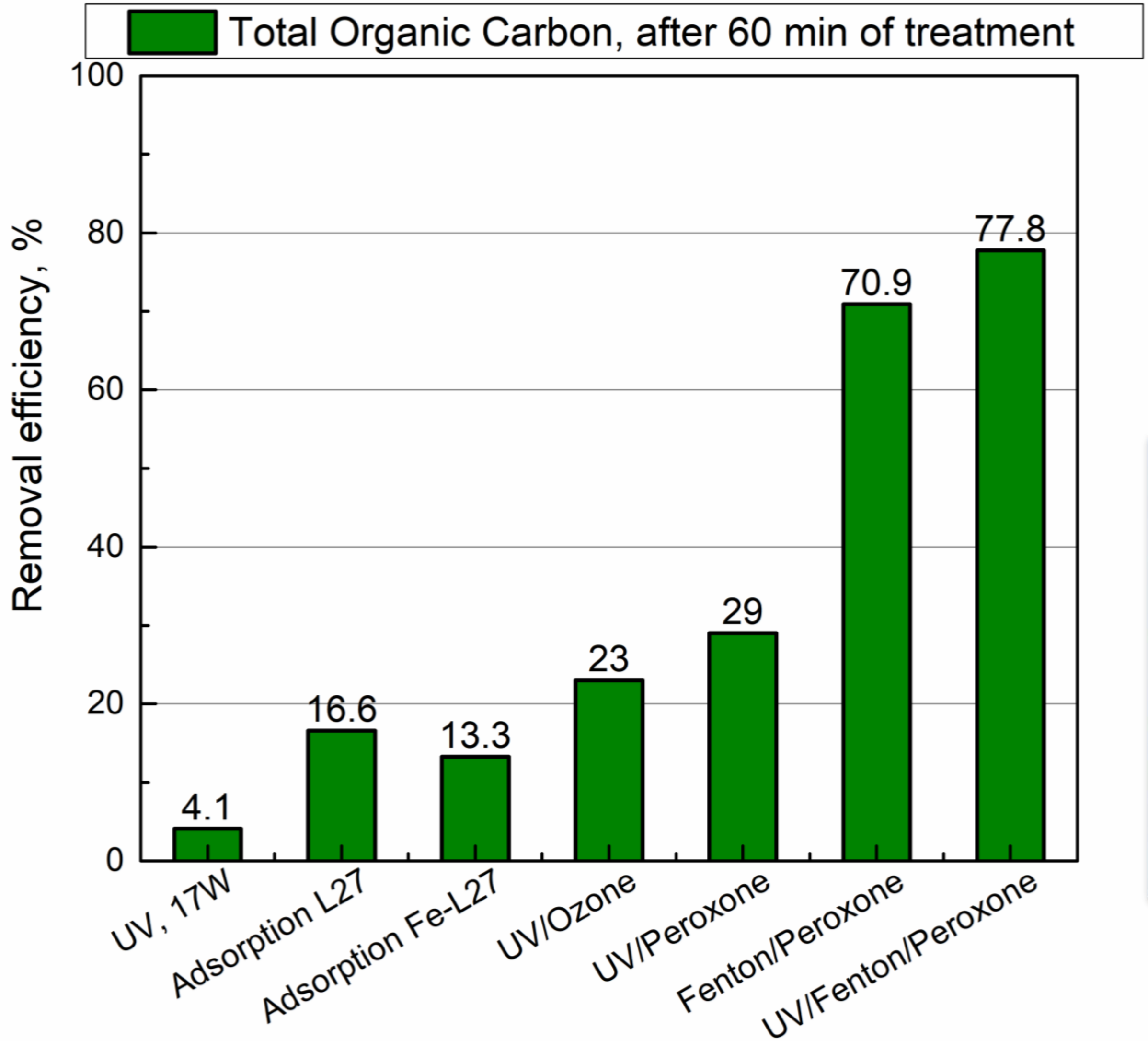
C_i = 200 mg/L

Peroxone and Fenton-Peroxone provide high removal efficiencies in relation to CIBP after only 20 min of treatment.

After 2h, even Ozonation results in >99% RE in relation to C_{IBP}.

Fe impregnation of AC has low influence on the adsorption capacity

Treatment tests
- in relation to the mineralization degree -



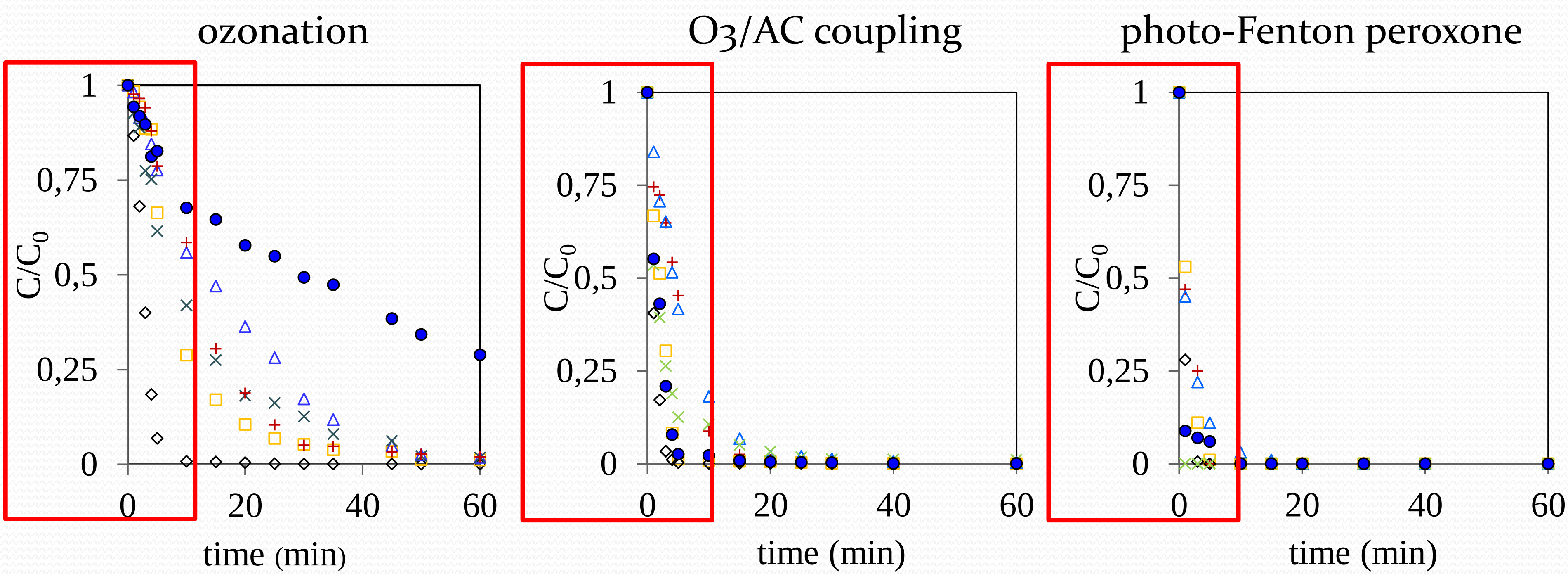
pH 7

C_i = 200 mg/L

In terms of mineralization, Fenton-based processes make the difference.

Ozonation and Peroxone processes degrade the molecule to a rather large degradation product.

Efficiency of Ozonation, AC/Ozonation coupling, and photo-Fenton peroxone process



terbutaline

metoprolol

fluoxetine

sulfamethoxazole

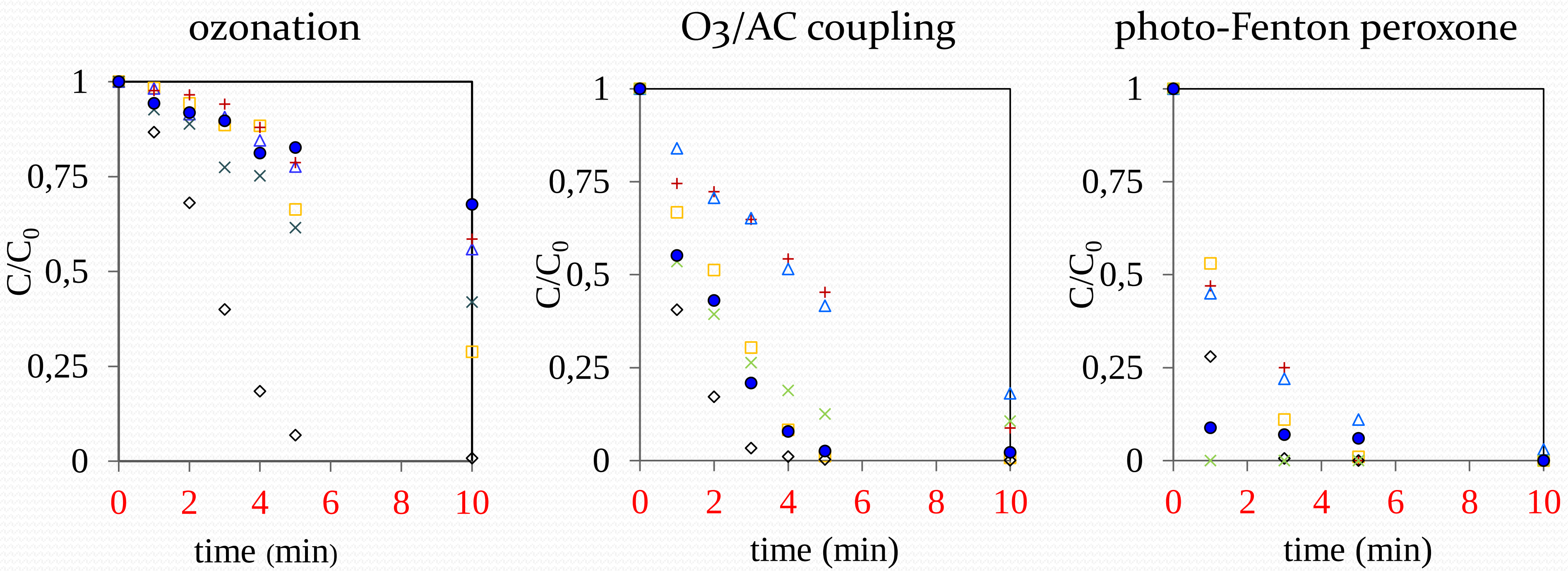
carbamazepine

ketoprofen

pH 3

C_i = 18 mg/L

Efficiency of Ozonation, AC/Ozonation coupling, and photo-Fenton peroxone process

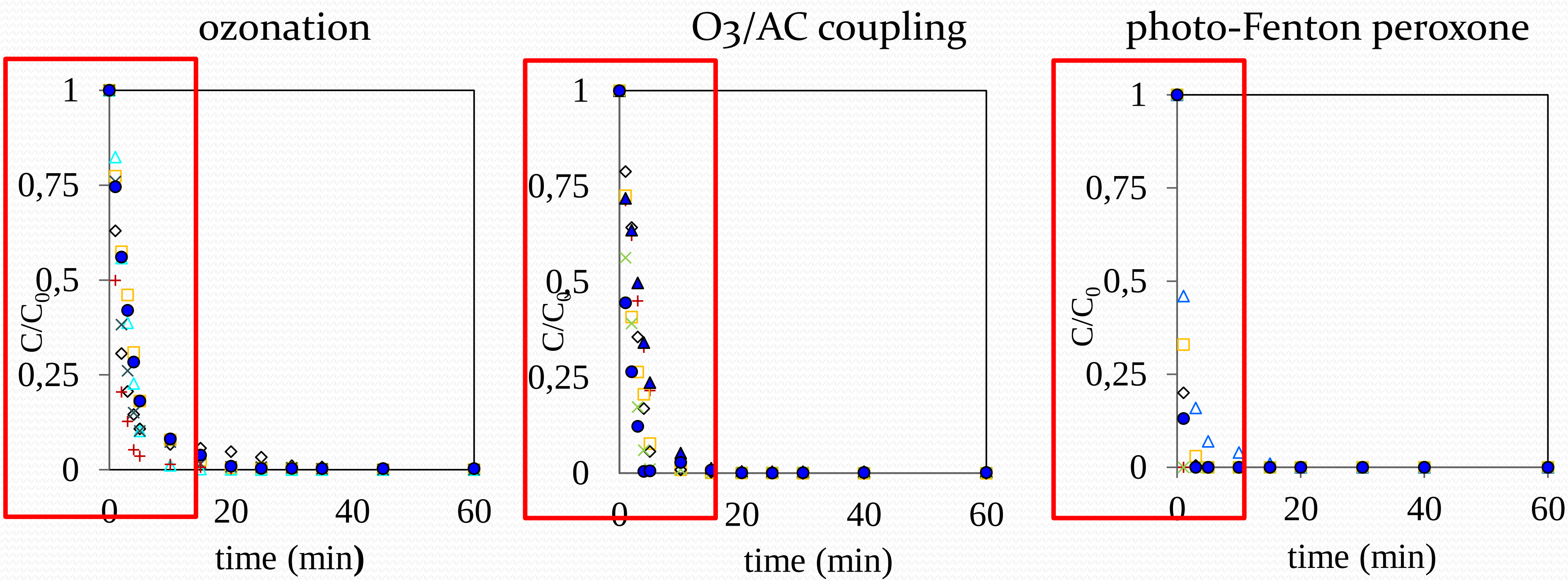


terbutaline
metoprolol
fluoxetine
sulfamethoxazole
carbamazepine
ketoprofen

pH 3

C_i = 18 mg/L

Efficiency of Ozonation and AC/Ozonation Coupling



terbutaline

metoprolol

fluoxetine

sulfamethoxazole

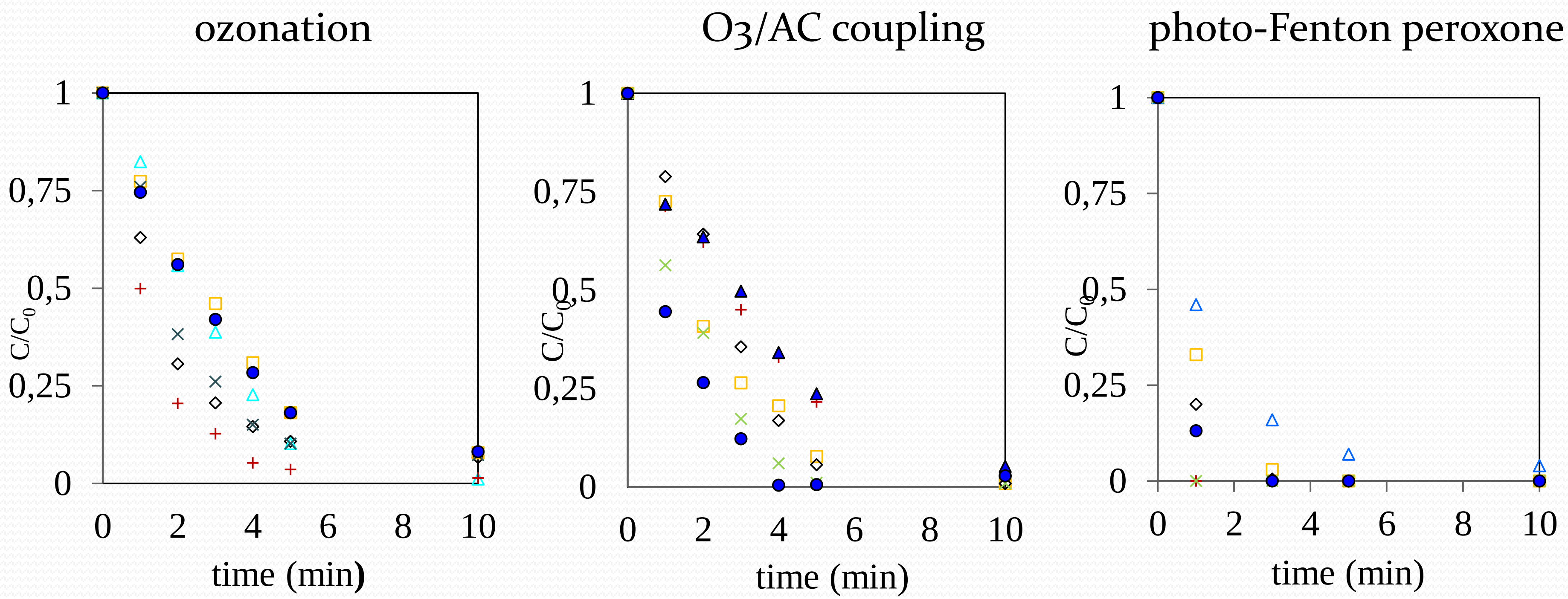
carbamazepine

ketoprofen

pH 7

C_i = 18 mg/L

Efficiency of Ozonation and AC/Ozonation Coupling



terbutaline

metoprolol

fluoxetine

sulfamethoxazole

carbamazepine

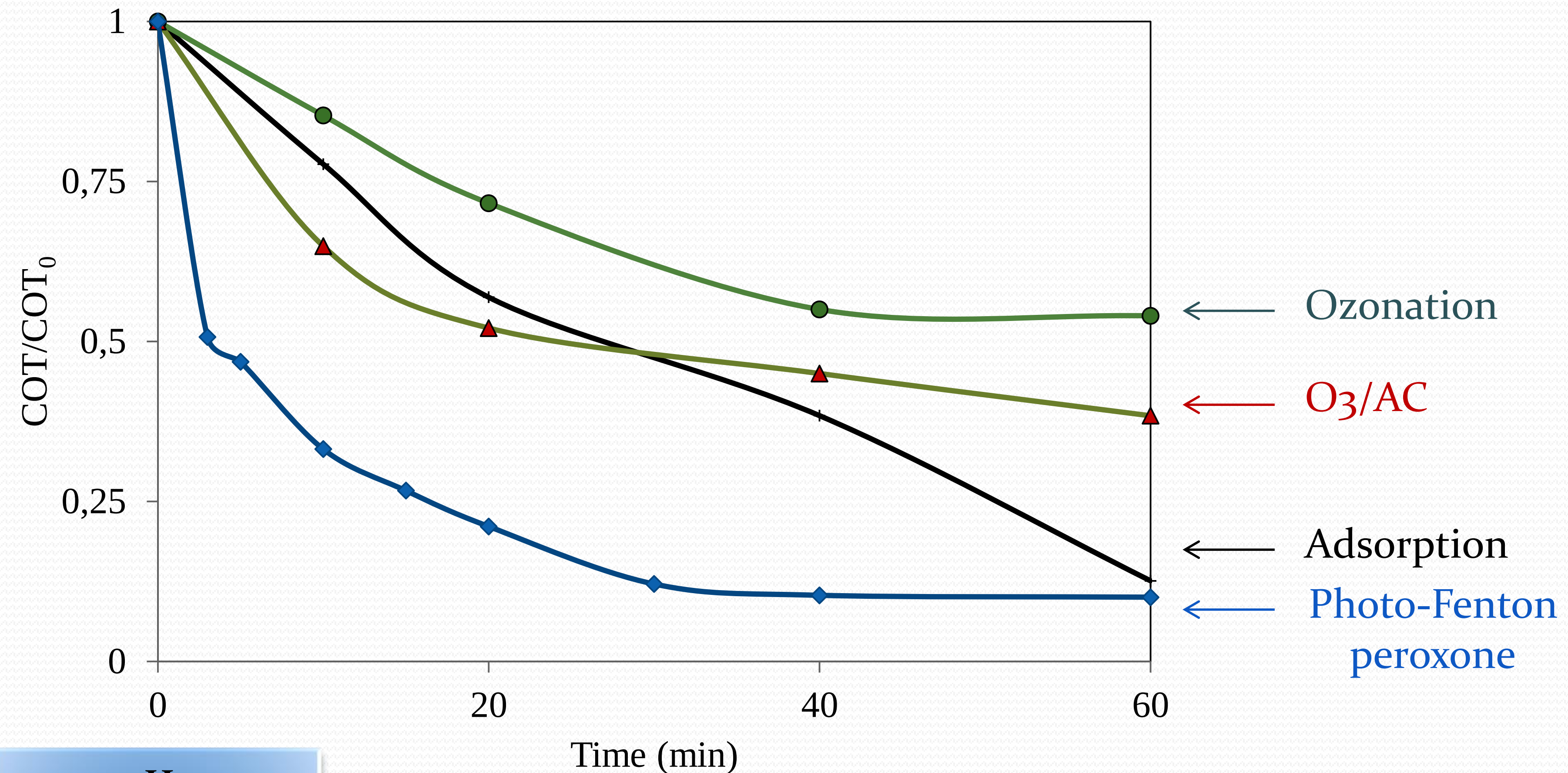
ketoprofen

pH 7

C_i = 18 mg/L

Efficiency of Ozonation and AC/Ozonation Coupling

TOC evolution



pH 7

C_i = 18 mg/L

Photo-Fenton peroxone : Mineralization >90 %

Conclusions

- ✓ It was found that the chemical properties of AC surface are critical in the selection of the proper matrices of Fenton-like catalysts.

- ✓ Among the investigated ACs, the acid surfaced AC provides the optimal micromedium to conduct the heterogeneous Fenton.

- ✓ The integration of the Fenton process by using Fe-impregnated ACs results in remarkable enhancement of the mineralization process.

- ✓ Heterogeneous Fenton based on Fe-impregnated ACs provides good removal efficiencies even at neutral pH of the treated aqueous solution.

- ✓ The use of a low pressure UV-C lamp of only 17W (UV-C Output: 5,7 W at 254 nm) enhances the Fenton-peroxone process.

Ongoing work

- ✓ Elaboration of mesoporous and acid-surfaced ACs from agricultural waste as support for Fe.
- ✓ Collaboration study with ICOA Orléans on the degradation mechanisms of Ibuprofen and Diclofenac. Identification of the by-products generated during the integrated treatment process.
- ✓ Collaboration study with CEMHTI Orléans on the toxicity of the treated solutions.
- ✓ Optimization of the parameters (Fe-AC dose, H_2O_2 dose, O_3 mass flow rate) of the integrated treatment technique in terms of removal efficiency, degree of mineralization and toxicity.

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Thank you for your attention!

Acknowledgments:

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