Waste water treatment by advanced oxidation processes (solar photocatalysis in degradation of industrial contaminants)

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Plataforma Solar de Almería , TABERNAS-Almería

SPAIN

Outlook

- **Introduction**
  - **Photocatalysis**
  - **Photo-Fenton**
- **Photoreactors**
  - **Compound Parabolic Collectors**
  - **State of the art**
- **Applications**
  - **Pesticides**
  - **OMW**
  - **Pharmaceutical WW**

Introduction

Biodegradable substances:

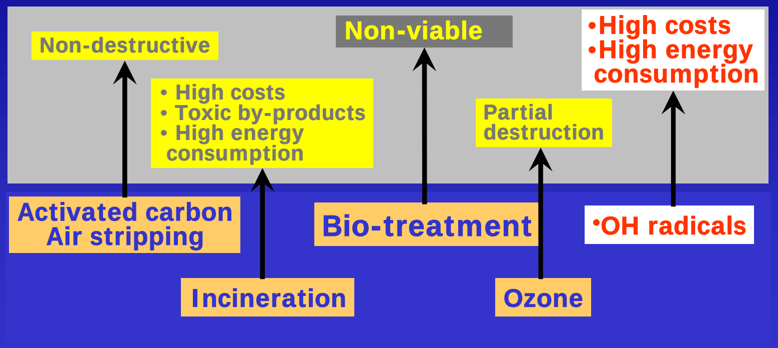
- Biofilter treatment / activated sludge treatment

Non-biodegradable substances can show

- Non-toxic / inert behaviour
- Acute toxicity
- Chronic toxicity

Alternative treatment

Introduction



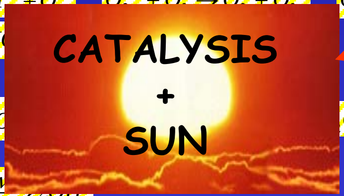
Introduction

- H_2O_2/Fe^{2+} **(Fenton):** $Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^\bullet$
- H_2O_2/Fe^{2+} **(**Fe^{3+}**) / UV (Photo-Fenton):** $Fe^{3+} \xrightarrow{h\nu} Fe^{2+} + HO^\bullet$
- $TiO_2/h\nu/O_2$ **(Photocatalysis):** $TiO_2 \xrightarrow{h\nu} e^- + h^+$

  $h^+ + H_2O \rightarrow {}^\bullet OH + H^+$
- O_3/H_2O_2: $H_2O_2 \xleftrightarrow{H^+} HO^- + O_3 \rightarrow O_2 + HO_2^-$ $HO_2^- + O_3 \rightarrow HO_2^\bullet + O_3^{\bullet -}$

  $HO_2^\bullet \Leftrightarrow H^+ + O^{\bullet -}$ ... $O^{\bullet -} + O_3 \rightarrow O_2 + O^{\bullet -}$ ... $O_3^{\bullet -} + H^+ \rightarrow HO_3^\bullet$

  $HO_3^\bullet \rightarrow H$ ...
- O_3/UV: $O_3 \xrightarrow{h\nu} O$ ... O_2 $H_2O_2 \xrightarrow{h\nu} 2HO^\bullet$
- H_2O_2/UV: $H_2O_2 \xrightarrow{h\nu} 2OH$

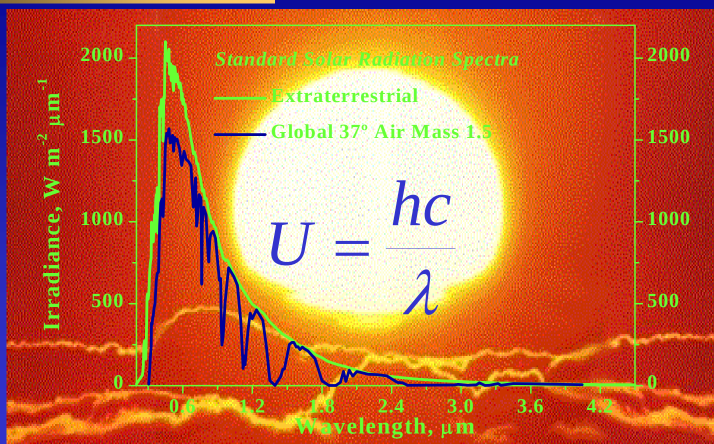


Introduction

Photochemical AOPs

AOP	key reactions	wavelength
UV/ H_2O_2	$H_2O_2 + h\nu \rightarrow 2\ OH^\bullet$	$\lambda < 300$ nm
UV/ O_3	$O_3 + h\nu \rightarrow O_2 + O\ (^1D)$ $O\ (^1D) + H_2O \rightarrow 2\ OH^\bullet$	$\lambda < 310$ nm
UV/H_2O_2/ O_3	$O_3 + H_2O_2 + h\nu \rightarrow O_2 + OH^\bullet + OH_2^\bullet$	$\lambda < 310$ nm
UV/ TiO_2	$TiO_2 + h\nu \rightarrow TiO_2\ (e^- + h^+)$ $TiO_2(h^+) + OH^-_{ad} \rightarrow TiO_2 + OH_{ad}^\bullet$	$\lambda < 390$ nm
photo-Fenton	$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + OH^\bullet + OH^-$ $Fe^{3+} + H_2O + h\nu \rightarrow Fe^{2+} + H^+ + OH^\bullet$	$\lambda < 580$ nm

Photocatalysis

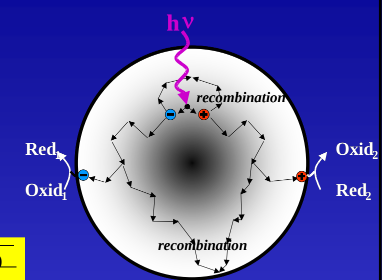


Photocatalysis

$$\lambda_G = \frac{hc}{E_G}$$

"band gap" energy is the energetic separation between a semiconductor valence and conduction band

Material	"Band gap" (eV)	λ for e-/h+ formation (nm)
CdO	2.1	590
CdS	2.5	497
Fe_2O_3	2.2	565
GaP	2.3	540
SnO_2	3.9	318
TiO_2	3.0	390
WO_3	2.8	443
ZnO	3.2	390
ZnS	3.7	336

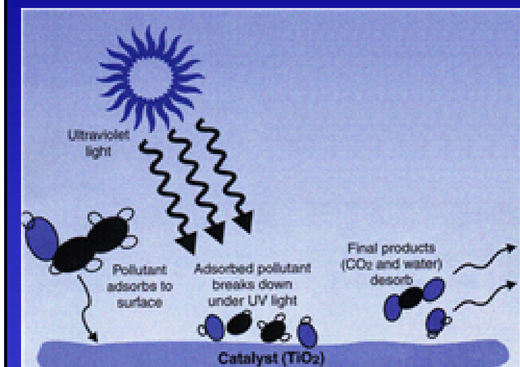


$$C + h\upsilon \rightarrow C(e^- + h^+)$$

$$h^+ + H_2O \rightarrow {}^{\bullet}OH + H^+$$

$$e^- + O_2 \rightarrow O_2^{\bullet -}$$

Photocatalysis



- The process takes place at ambient temperature.
- Oxidation of the substances into CO_2 is complete.
- The oxygen necessary for the reaction is obtained from the atmosphere.
- The catalyst is cheap, innocuous and can be reused.
- The catalyst can be attached to different types of inert matrices.

Photocatalysis

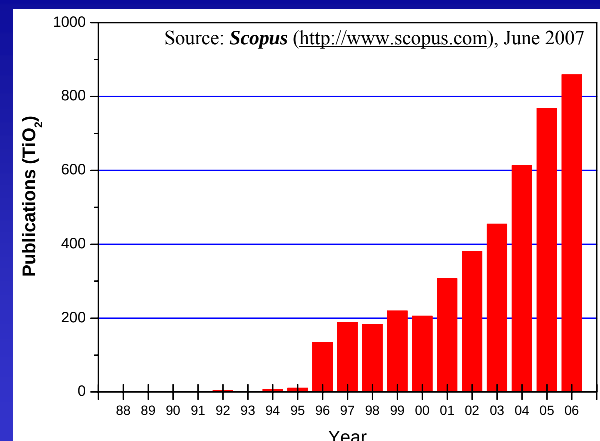


Photo-Fenton

Photo-Fenton method

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + {}^{\bullet}OH$$

$$Fe^{3+} + H_2O \xrightarrow{h\nu} Fe^{2+} + H^+ + {}^{\bullet}OH$$

Advantages

- High reaction rates
- Cheap, non-toxic reagents (Fe, H_2O_2, acid, base)

Disadvantages

- pH adjustment necessary
- Iron removal necessary

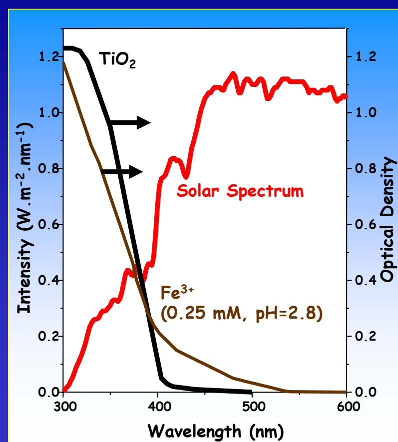


Photo-Fenton

Reactions Fe^{2+}, Fe^{3+} and H_2O_2 in water

$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + OH^{\bullet}$	$k = 53 - 76\ M^{-1}\ s^{-1}$
$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^-$	$k = 2.6 - 5.8 \cdot 10^8\ M^{-1}\ s^{-1}$
$Fe^{2+} + HO_2^{\bullet} \rightarrow Fe^{3+} + HO_2^-$	$k = 0.75 - 1.5 \cdot 10^6\ M^{-1}\ s^{-1}$
$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2^{\bullet} + H^+$	$k = 1 - 2 \cdot 10^{-2}\ M^{-1}\ s^{-1}$
$Fe^{3+} + HO_2^{\bullet} \rightarrow Fe^{2+} + O_2 + H^+$	$k = 0.33 - 2.1 \cdot 10^6\ M^{-1}\ s^{-1}$
$Fe^{3+} + O_2^{\bullet -} \rightarrow Fe^{2+} + O_2$	$k = 0.05 - 1.9 \cdot 10^9\ M^{-1}\ s^{-1}$
$OH^{\bullet} + H_2O_2 \rightarrow H_2O + HO_2^{\bullet}$	$k = 1.7 - 4.5 \cdot 10^7\ M^{-1}\ s^{-1}$

Radical reactions

$2OH^{\bullet} \rightarrow H_2O_2$	$k = 5 - 8\ 10^9\ M^{-1}\ s^{-1}$
$2HO_2^{\bullet} \rightarrow H_2O_2 + O_2$	$k = 0.8 - 2.2\ 10^6\ M^{-1}\ s^{-1}$
$HO_2^{\bullet} + OH^{\bullet} \rightarrow H_2O + O_2$	$k = 1.4\ 10^{10}\ M^{-1}\ s^{-1}$

Equilibriums

$H_2O_2 \rightleftharpoons HO_2^- + H^+$	$K = 2.63\ 10^{-12}\ M$
$[Fe]^{3+} + H_2O_2 \rightleftharpoons [Fe(HO_2)]^{2+} + H^+$	$K = 3.1\ 10^{-3}\ M$
$[Fe(OH)]^{2+} + H_2O_2 \rightleftharpoons [Fe(OH)(HO_2)]^{+} + H^+$	$K = 2\ 10^{-4}\ M$
$HO_2^{\bullet} \rightleftharpoons O_2^{\bullet -} + H^+$	$K = 3.55\ 10^{-5}\ M$
$OH^{\bullet} \rightleftharpoons O^{\bullet -} + H^+$	$K = 1.02\ 10^{-12}\ M$
$HO_2^{\bullet} + H^+ \rightleftharpoons H_2O_2^{\bullet +}$	$K = 3.16 – 3.98\ 10^{-12}\ M$

Photo-Fenton

Fenton reactions with organics

$$R^\bullet + Fe^{3+} \rightarrow R^+ + Fe^{2+}$$
$$R^\bullet + Fe^{2+} \rightarrow R^- + Fe^{3+}$$
$$R^\bullet + R^\bullet \rightarrow R - R$$
$$R^\bullet + HO_2^\bullet \rightarrow RO_2H$$
$$Fe^{2+} + RO_2H \rightarrow Fe^{3+} + OH^- + OR^\bullet$$
$$R^\bullet + O_2 \rightarrow RO_2^\bullet$$
$$RO_2^\bullet + H_2O \rightarrow ROH + HO_2^\bullet$$

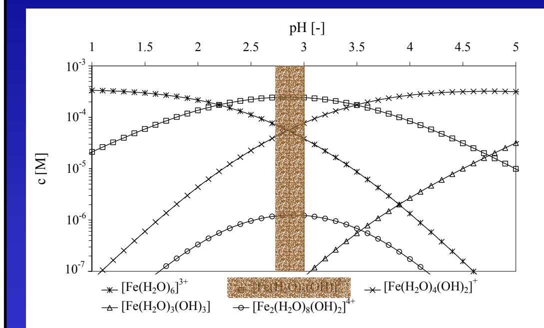
$$OH^\bullet + RH \rightarrow R^\bullet + H_2O$$
$$R - CH = CH_2 + OH^\bullet \rightarrow R - C^\bullet H - CH_2OH$$
$$OH^\bullet + RX \rightarrow RX^{\bullet +} + OH^-$$

Persistent complexes with Mono- y Dicarboxylic acids (L).
Reaction is stopped before complete mineralisation

$$Fe^{3+} + n\ L \rightarrow [FeL_n]^{x+} \xrightarrow{H_2O_2,\ dark} \text{no further reaction}$$

Photo-Fenton

"Photo-Fenton"



$$[Fe^{3+}L] + h\nu \longrightarrow [Fe^{3+}L]^* \longrightarrow Fe^{2+} + L^\bullet$$
$$[Fe(H_2O)]^{3+} + h\nu \longrightarrow Fe^{2+} + OH^\bullet + H^+$$
$$[Fe(OH)]^{2+} + h\nu \longrightarrow Fe^{2+} + OH^\bullet$$
$$[Fe(OOC - R)]^{2+} + h\nu \rightarrow Fe^{2+} + CO_2 + R^\bullet$$

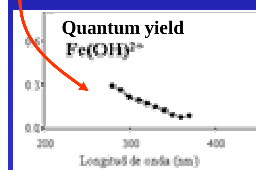
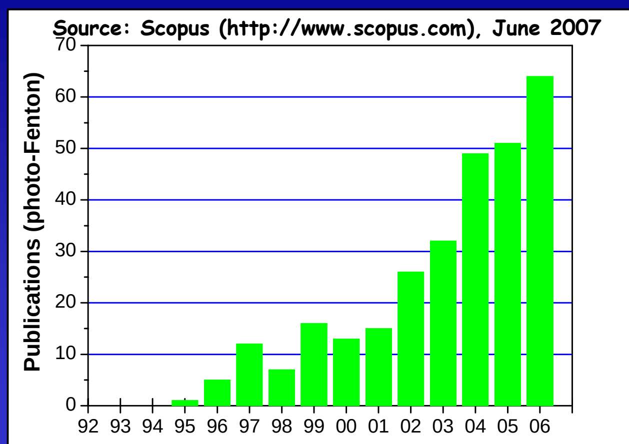


Photo-Fenton



Outlook

Introduction

- **Photocatalysis**
- **Photo-Fenton**

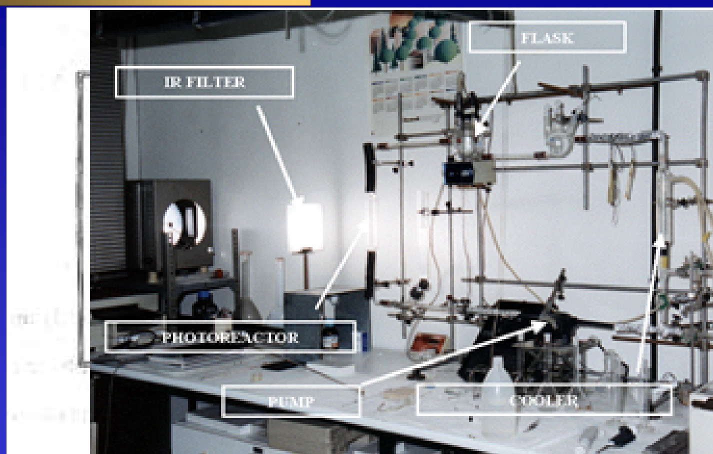
Photoreactors

- **Compound Parabolic Collectors**
- **State of the art**

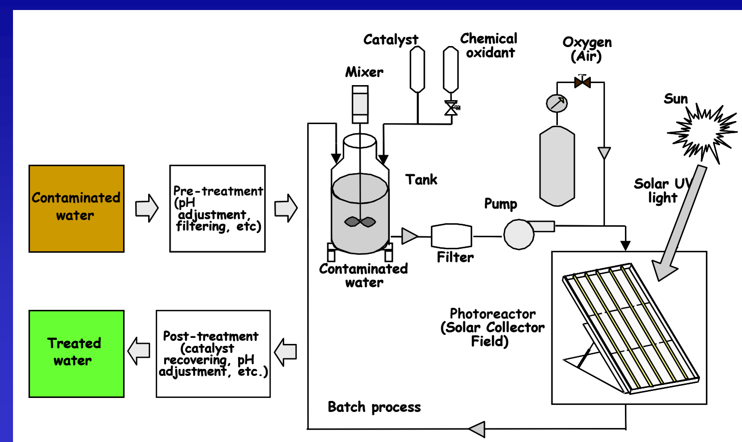
Applications

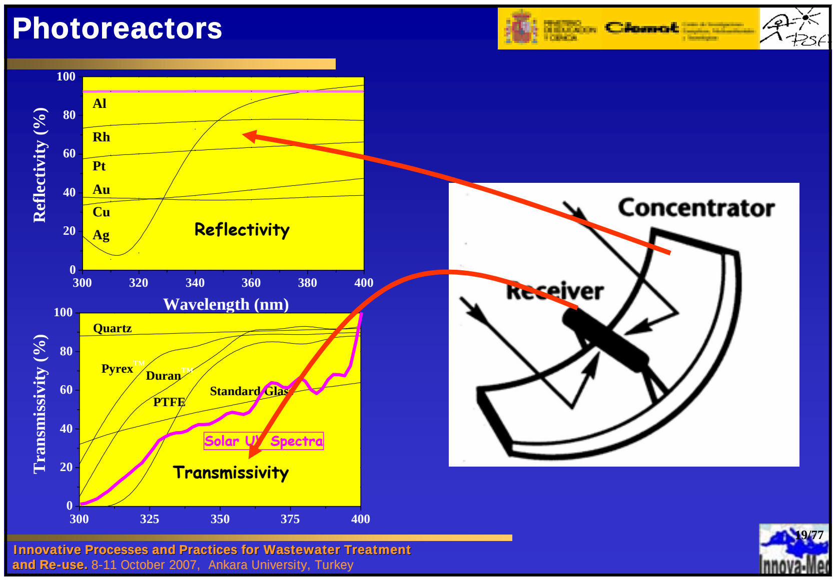
- **Pesticides**
- **OMW**
- **Pharmaceutical WW**

Photoreactors



Photoreactors




Photoreactors
Reflectivity (%)
Al
Rh
Pt
Au
Cu
Ag
Reflectivity
Wavelength (nm)
Transmissivity (%)
Quartz
Pyrex
Duran
PTFE
Standard Glas
Solar UV Spectra
Transmissivity
Concentrator
Receiver
Innovative Processes and Practices for Wastewater Treatment
and Re-use. 8-11 October 2007, Ankara University, Turkey
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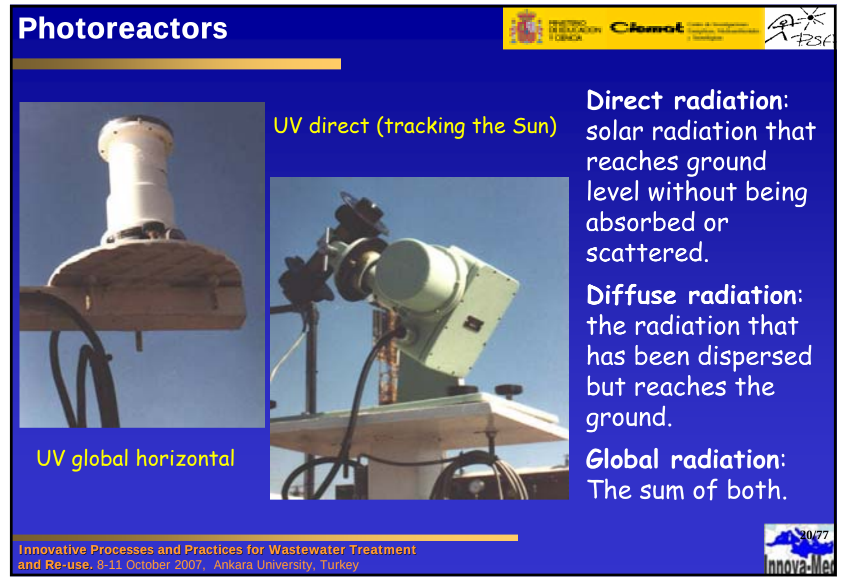
Photoreactors
UV direct (tracking the Sun)
UV global horizontal
Direct radiation: solar radiation that reaches ground level without being absorbed or scattered.
Diffuse radiation: the radiation that has been dispersed but reaches the ground.
Global radiation: The sum of both.
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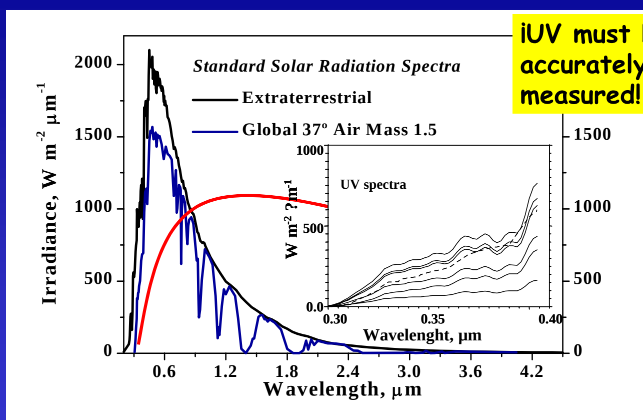
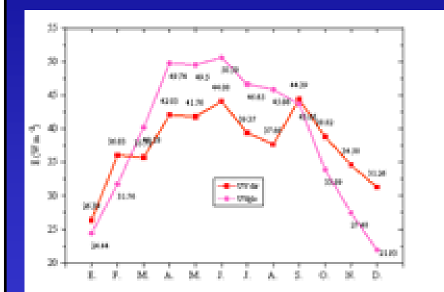
Photoreactors

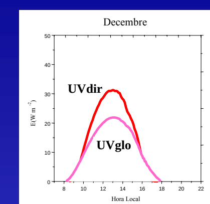


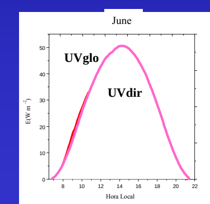
¡UV must be accurately measured!

Photoreactors

Máximum UV (λ<400 nm) sunny days (37°, N)







State of the art

Sandia National Labs (Albuquerque, USA) developed in 1989 the first solar facility for water detoxification at pre-industrial level based on 1-axis Parabolic Trough Collectors (PTC). CIEMAT, in 1990, erected the second at *Plataforma Solar de Almería* (Spain), using 2-axis PTCs.

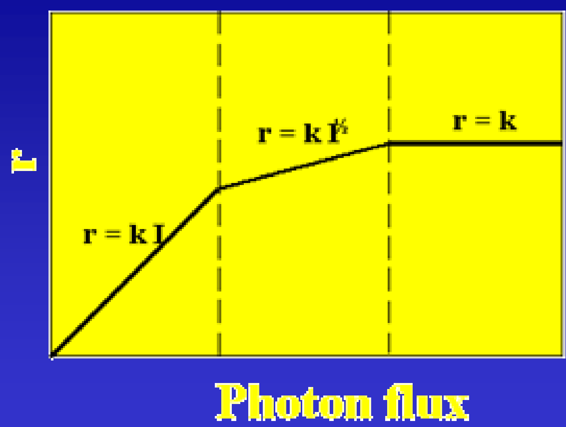
These pilot plants were the first step in the development of the solar technology.

State of the art

In the early nineties, the National Renewable Energy Laboratory, Sandia National Laboratories and the Lawrence Livermore National Laboratory addressed the "Livermore experiment" (USA). A Solar Detox Plant was installed using one-axis PTCs to treat TCE-groundwater contaminated during the Second World War.

This experiment constituted the first on-site test. Tests were successful but the economic figures not!

State of the art

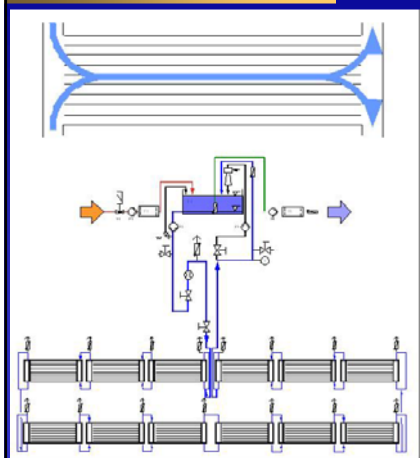


State of the art

One-sun (non-concentrating) collectors are cheaper than PTCs. An extensive effort in the design of small non-tracking collectors, has resulted in the testing of several different non-concentrating solar reactors.

The design of a robust one-sun photoreactor is not trivial: weather-resistant, chemically inert and ultraviolet-transmissive. Also, flow in non-concentrating systems is usually laminar.

State of the art



DSSR-Pilotplant II

The Planning Concept:

Illuminated Reactor Area A = 30 m^2

Flow Re 5000 (turbulent)

Volumetric Flow Rate V_{tot} = 12 m^3 /h

Pressure Drop = 0.5 bar

Treatment Capacity 0.9 m^3/d

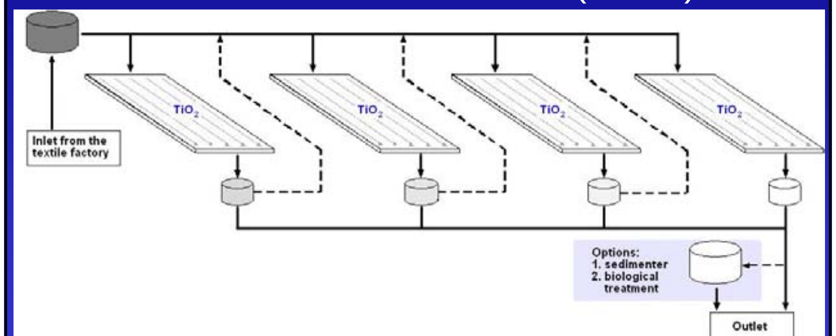
State of the art

DSSR-Pilotplant II in Wolfsburg

Source: Prof. D. Bahnemann, Universität Hannover

State of the art

Thin-Film Fixed Bed Reactor (TFFBR)



Source: Prof. D. Bahnemann, Universität Hannover

State of the art

Thin-Film Fixed Bed Reactor (TFFBR)

1995 in Almeria/Spain

2003 in Tunis/Tunisia

Source: Prof. D. Bahnemann, Universität Hannover

State of the art

PARABOLIC CONCENTRATORS

MAIN ADVANTAGES	MAIN DISADVANTAGES
Turbulent flow	Only Direct radiation
No vaporization of compounds	High cost (Sun Tracking)
	Low optical efficiency
	Low Quantum efficiency (with TiO_2)
	Overheating

NON CONCENTRATING PHOTOREACTORS

MAIN ADVANTAGES	MAIN DISADVANTAGES
Direct & Diffuse radiation	Laminar flow (low mass transfer)
No heating	Vaporization of reactants
Low cost	Reactants contamination
High optical efficiency	

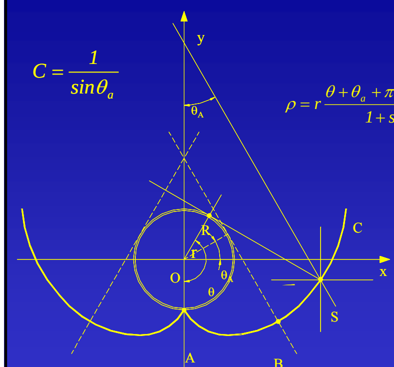
Compound Parabolic Collectors

1 Sun COMPOUND PARABOLIC COLLECTORS

- ✓ Turbulent flow conditions
- ✓ No vaporization of volatile compounds
- ✓ No tracking
- ✓ No Overheating
- ✓ Direct and Diffuse radiation
- ✓ Low cost
- ✓ Weatherproof (no contamination)

Compound Parabolic Collectors

$$C = \frac{1}{\sin\theta_a}$$



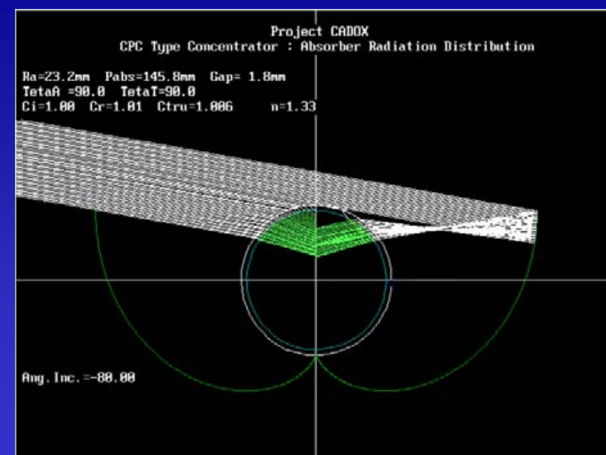
$\rho = r\theta \quad for \quad |\theta| \le \theta_a + \pi/2$ Part A-B

$\rho = r\dfrac{\theta + \theta_a + \pi/2 - \cos(\theta - \theta_a)}{1 + \sin(\theta - \theta_a)} \quad for \quad \theta_a + \dfrac{\pi}{2} \le |\theta| \le \dfrac{3\pi}{2} - \theta_a$ Part B-C

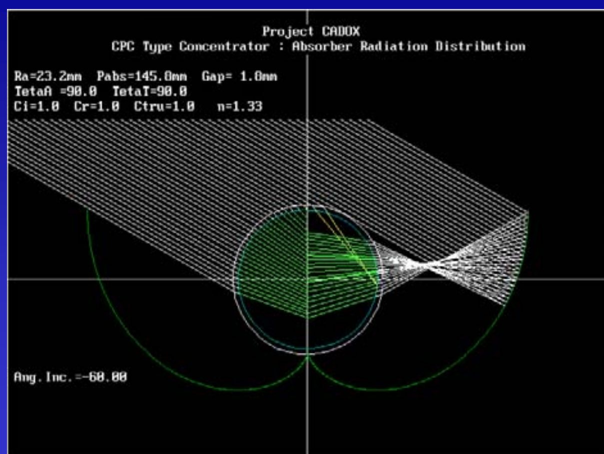
If $\theta_a = 90$ ⇨ $C = 1$

One Sun CPC collector manufacturing: θ_a = 90° ⇒ all direct and diffuse solar photons can be collected and used (diffuse UV radiation is a very important fraction of total solar UV)

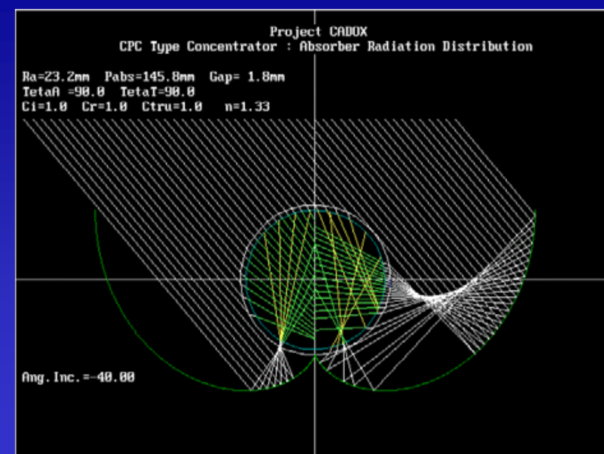
Compound Parabolic Collectors



Compound Parabolic Collectors



Compound Parabolic Collectors



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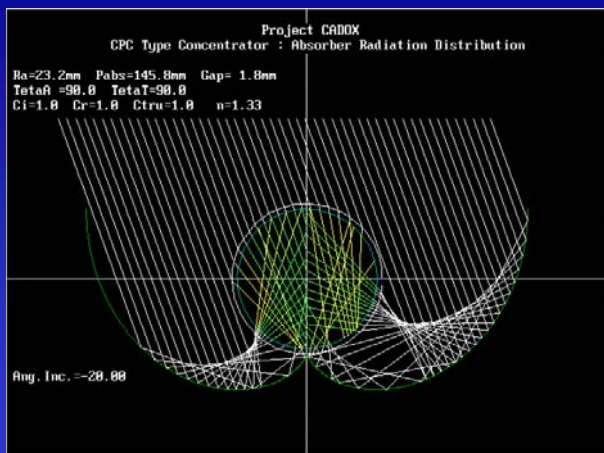
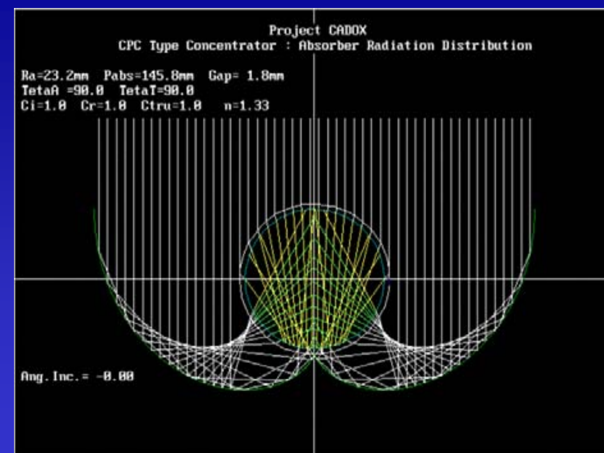
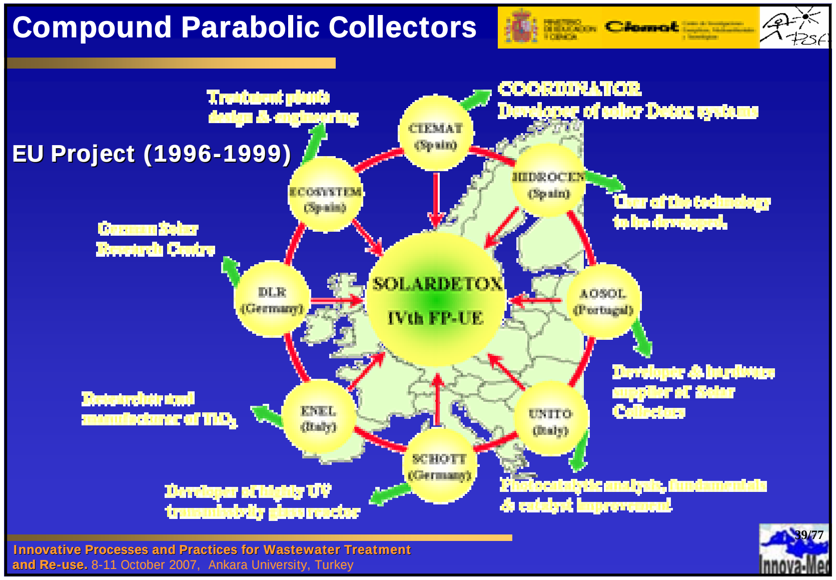


Compound Parabolic Collectors




Compound Parabolic Collectors
EU Project (1996-1999)
Treatment plants design & engineering
COORDINATOR
Developer of solar Detox systems
CIEMAT (Spain)
ECOSYSTEM (Spain)
HIDROCEN (Spain)
User of the technology to be developed.
German Solar Research Centre
DLR (Germany)
SOLARDETOX
IVth FP-UE
AOSOL (Portugal)
Developer & hardware supplier of Solar Collectors
Researcher and manufacturer of TiO_2
ENEL (Italy)
UNITO (Italy)
SCHOTT (Germany)
Developer of highly UV transmissivity glass reactor
Photocatalytic analysis, fundamentals & catalyst improvement
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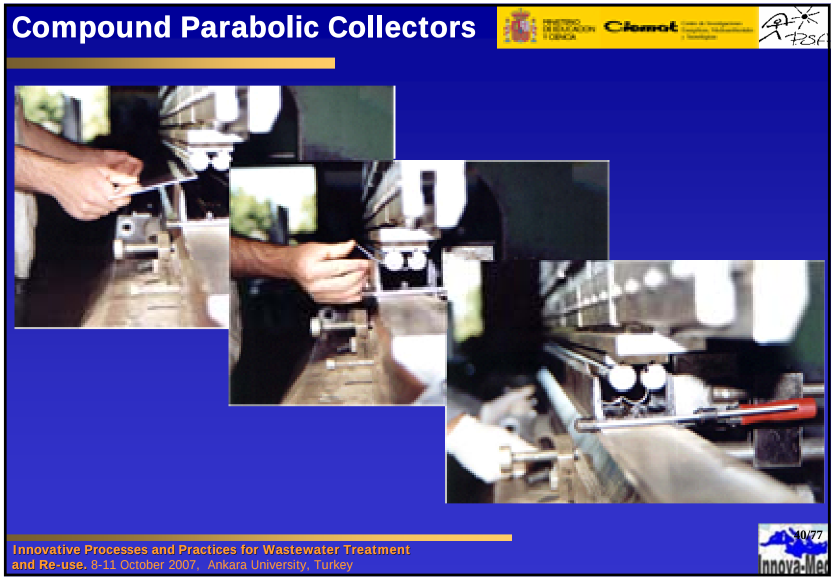
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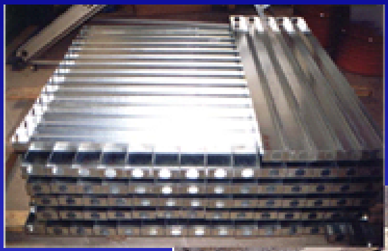
Compound Parabolic Collectors



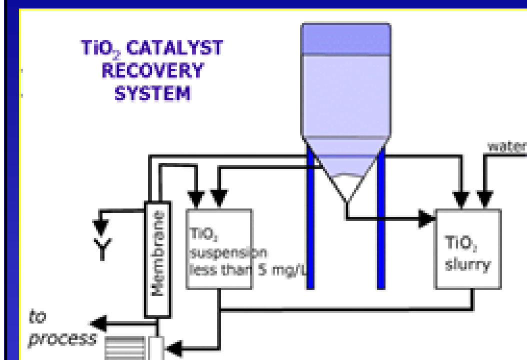
Compound Parabolic Collectors

A very simple one-sun CPC collector was designed, constructed and tested to optimize the manufacturing process (modularity), on site installation (minimum interconnecting pieces and not illuminating zones) and cost saving

Additional system advantages:

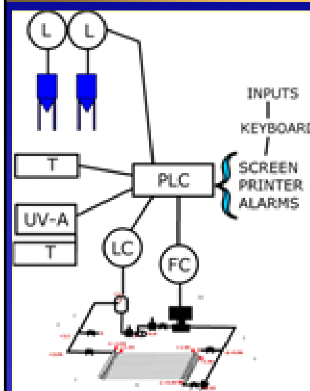
- Easy manufacturing
- Low investment cost
- Simple operation and supervision
- Low maintenance requirements
- No sun tracking devices are needed
- UV diffuse radiation can be profited

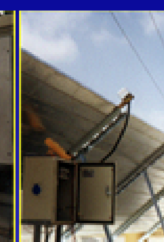
Compound Parabolic Collectors



Slurry systems are the most efficient, resulting an important reduction in the final treatment cost. A process for catalyst recuperation has been patented. EP-1-101-737-A1 (2001).

Compound Parabolic Collectors

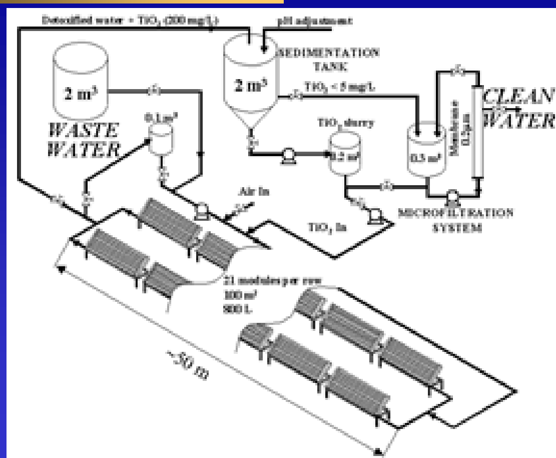




The plant is designed with full automatic systems. A Programmable Logic Controller receives all plant data signals (flow-rate, tanks level, temp, solar UV-A irradiation, etc) and control pumps and system valves.

Process evolution is monitored through the measuring and integration of UV light up to a fixed level.

Compound Parabolic Collectors



Compound Parabolic Collectors

The SOLARDETOX Consortium (Brite-Euram III Program, Contract No. BRPR-CT97-0424) has installed during 1999 the first European Solar Detoxification Plant. Main plant characteristics are:

- **CPC surface: 100 m^2**
- **Treatment volume: 800 L.**
- **Batch Operation**
- **Automatic operation**
- **cost of the plant: 100000 €**

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- Photo-Fenton

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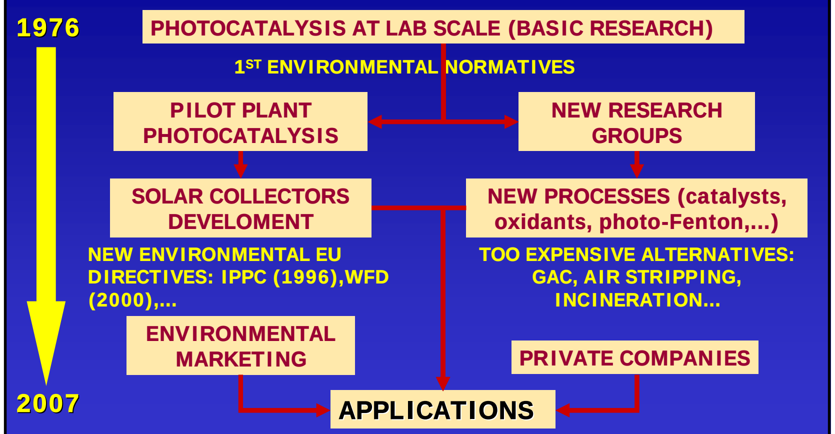
- State of the art

➡ Applications
- Pesticides
- OMW
- Pharmaceutical WW

Applications



Applications

✓ It has demonstrated that the solar photocatalytic technology is sufficiently developed for industrial use. A European industrial consortium has been created to the design and set-up of turnkey SOLARDETOX plants.

✓ The technology developed can be used, without modification, to address solar Photo-Fenton and TiO_2 degradation process.

APPLICATIONS

- Organics concentration ≤ hundreds of mg L-1.
- Low-medium flow (< 10 m3/h).
- Contaminants present within complex mixtures of organics.
- Contaminants with no easy treatment by conventional technologies.

Applications

– Phenols, nitrophenols and halophenols.

– Pharmaceutical compounds (antibiotics, disinfectants...).

– Water disinfection.

– Gasoline additives (MTBE, ETBE,..).

– Chlorinated hydrocarbons (solvents, VOCs, etc).

– Residues from textile industry (dyes).

– Agrochemical wastes (pesticides).

Pesticides

Small amounts of pesticide remaining in the empty containers (approx. 70 units/ha).

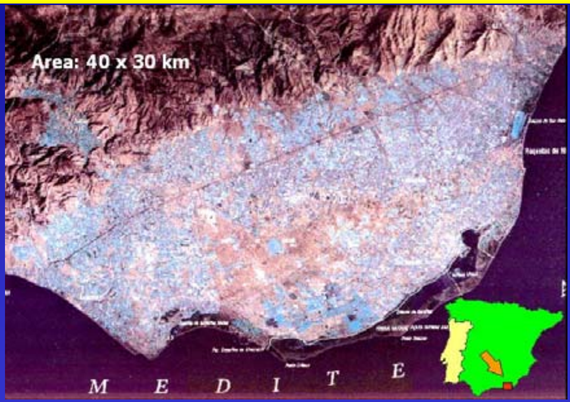
Pesticides

QUITE WELL SUITED TO SOLAR PHOTOCATALYTIC TREATMENT:

1. The initial pesticide concentration can be controlled as a function, so the most appropriate concentration for optimum photocatalytic efficiency can be chosen.
2. Toxicity is extreme, low-volume and in a well-defined location.
3. Such point sources of pollution may be ideally treated in small-scale treatment units.
4. Intensive agriculture in greenhouses is usually concentrated in sunny countries.

Pesticides

The intensive agriculture activity is a very important economical sector in Almería. There are more than 350 km² of greenhouses.



Pesticides

These greenhouses yearly consume 5.200 tons of phytosanitary products (1.5 million of bottles; 1.9 L average volume).
A process has been designed to recycle the plastic of these bottles. The recycling process needs a washing of the plastic. This produces a water with hundreds of mg/L of persistent toxic compounds.

Proposed Solution : *Solar Photocatalytic Treatment*

Pesticides

Plant design data:

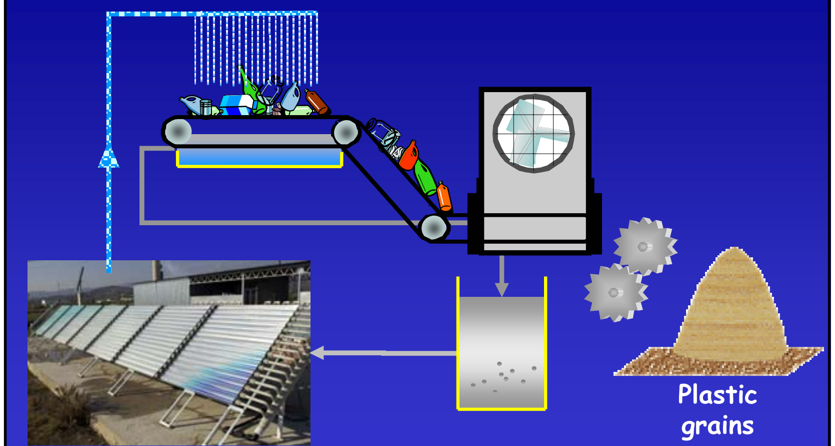
a) Total yearly volume of water to be treated (V_T): **1875 m³**

b) Yearly operating hours of solar facility (T_S): **3000 h**

c) Yearly average global UV irradiation (UV_G), sunrise to sunset: **18.6 W_{UV} m^{-2}**

d) Average solar energy needed to degrade the contaminants (Q_{UV}): **12 kJ_{UV} L^{-1}**

Pesticides



Pesticides

Pesticides

$$A_r = \frac{Q_{UV}\, V_t}{T_s\, UV_G} = \frac{12\,x\,10^3\; x\,1875\,x\,10^3}{3000\;x\,3600\;x\,18.6}\left[\frac{J\;L^{-1}\;L}{s\;W\;m^{-2}}\right] = 112\;m^2$$

Final selected plant dimensioning (solar collector area) was: **150 m²**

Solar field figures:

a) Individual CPC modules formed by **20 parallel tubes** (surface: **2.7** m^2/module)

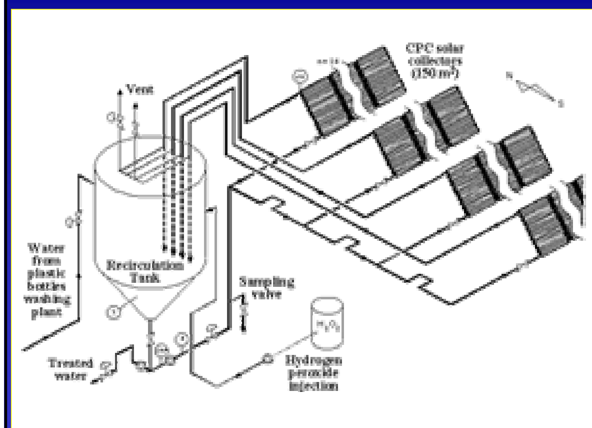
b) 4 parallel rows with **14 modules** each mounted on a 37º-tilted platform (local latitude)

c) total collectors surface: **150 m²**

d) Total photoreactor volume: **1061 L**

e) Total volume per batch: **1500** to **2000 L**

Pesticides



Operating procedure:

a) The system is run in **batch mode** using a **2000 L** recirculation tank

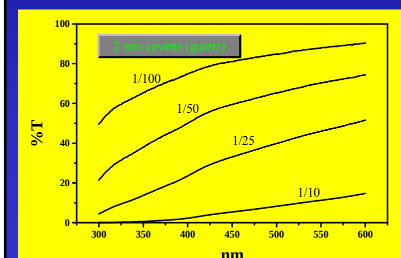
b) The **4 rows** are connected in parallel (independently operated) and the **14 modules** of each row in series

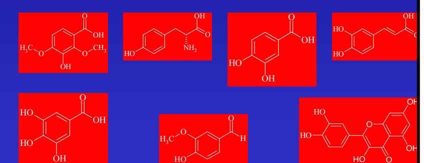
c) After treatment water is **returned** to the washing system and the tank is **refilled** with new contaminated water

OMW

Typical composition (only main parameters) of OMW:

COD:	80000 mg/L
Dissolved Phenols:	4500 mg/L
TOC:	35000 mg/L
pH:	5.1
Relevant anions:	phosphate 700 mg/L, chloride 500 mg/L,sulphate 100 mg/L, nitrite 6 mg/L.

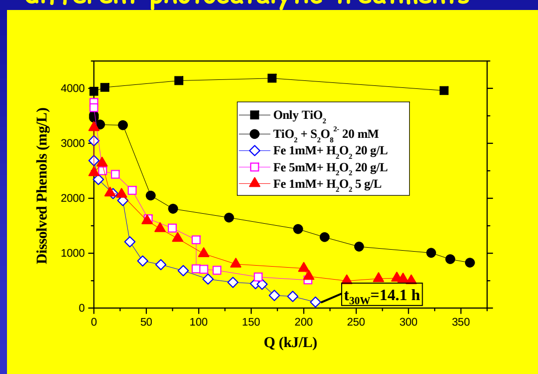




Transmission spectra of OMW through 2, mm pathlength at different dilution ratios.

OMW

OMW Dissolved Phenols behaviour with different photocatalytic treatments



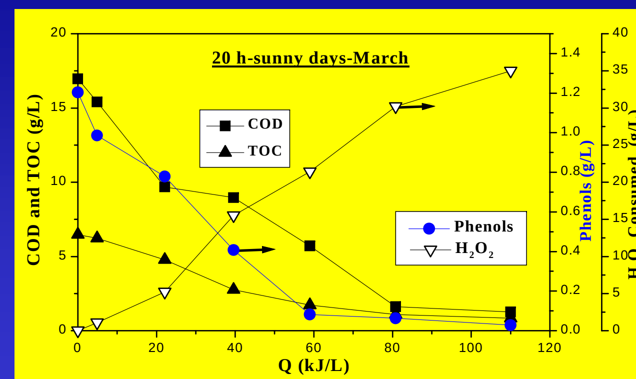
OMW

OMW

Installed and tested in a Olive Mill at Kivery (ARGOS, Greece).

OMW

Treatment of OMW in the FFR. Fe = 5mM



OMW

TOC:	16000 ppm	8000 ppm	0 ppm
Tot.Phenols:	3200 mg/L	1600 mg/L	0 mg/L

OMW

Pot experiment performed in greenhouse because of:

-Protection from environment (wind, rain, insects)

-Controled irrigation

-Additional light

OMW

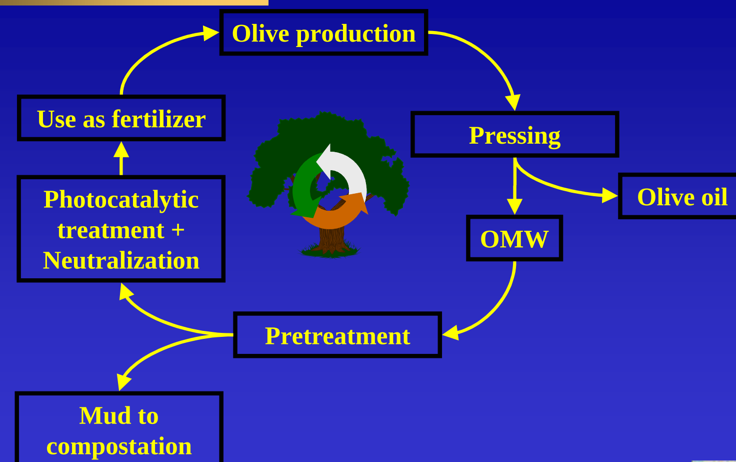
Substrate:

Earth
- Bacteria ⇒ conversion of TOC
- Unknown disposal of nutrients

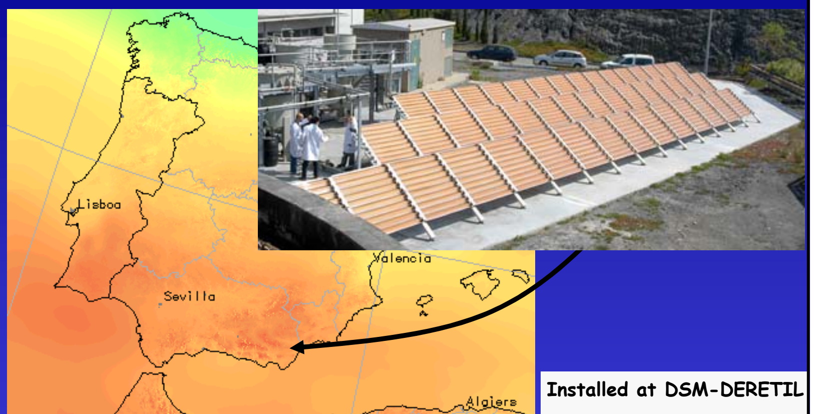
Perlita
- Aluminium silicate, porosity of 95%
- Inert substrate

Pots:
- 12 L volume, PVC, fair grey

OMW



Pharmaceutical WW

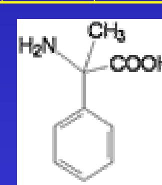


Installed at DSM-DERETIL
Villaricos (ALMERIA)

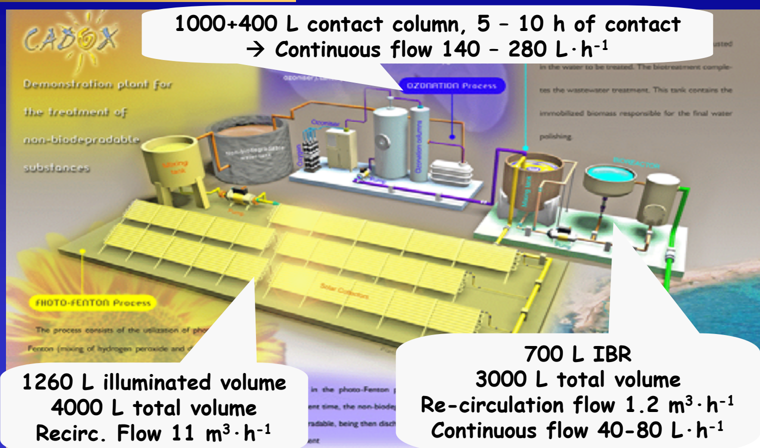
Pharmaceutical WW

Composition of wastewater (seawater) containing Femac (α-methylphenilglycine, $C_9H_{11}NO_2$)

	mg L^{-1}		mg L^{-1}		mg L^{-1}
Femac	500-600	Susp. solids	20-100	COD	1500-1800
TOC	400-500	NH_4^+	0-40	NO_3^-	200-600



Pharmaceutical WW

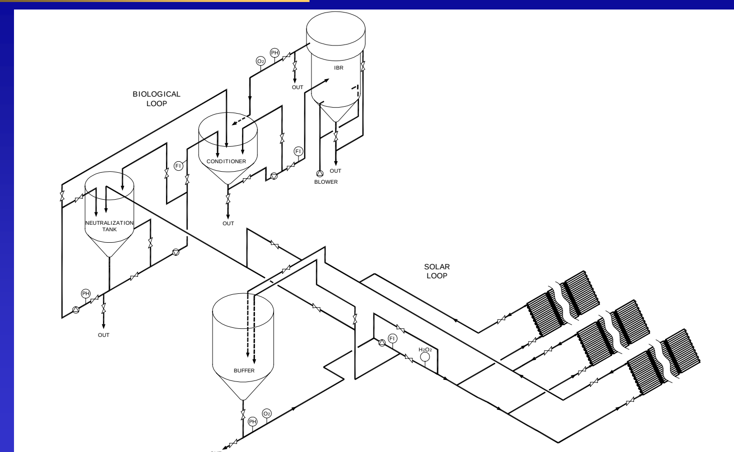


1000+400 L contact column, 5 – 10 h of contact
→ Continuous flow 140 – 280 $L \cdot h^{-1}$

1260 L illuminated volume
4000 L total volume
Recirc. Flow 11 $m^3 \cdot h^{-1}$

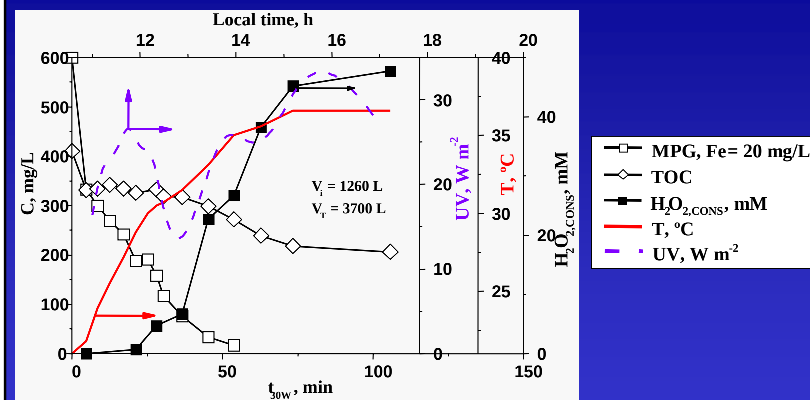
700 L IBR
3000 L total volume
Re-circulation flow 1.2 $m^3 \cdot h^{-1}$
Continuous flow 40-80 $L \cdot h^{-1}$

Pharmaceutical WW



Pharmaceutical WW

Pharmaceutical WW



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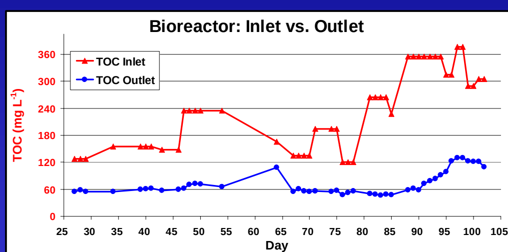

IBR during 75 days of continuous mode operation. Continuous operation showed the stability and permanent activity of the immobilised biomass with an influent, which originates from the AOP pre-treatment (photo-Fenton)

Innovative Processes and Practices for Wastewater Treatment and Re-use. 8-11 October 2007, Ankara University, Turkey

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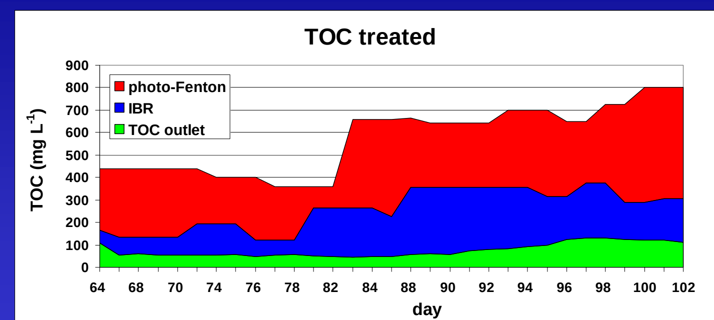
Pharmaceutical WW

Overview coupling Photo-Fenton/Biotreatment



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Pharmaceutical WW

Total cost per m³ of treated effluents containing 1 kg/m³ of Femac (i.e. 700 mg TOC/L) for different scenarios. Depreciation: 10 years

	photo-Fenton/ Biol (Demo plant) 2300 m^3 year		photo-Fenton/Biol (1000 m^2 CPC) 23000 m^3 year		photo-Fenton/Biol (10000 m^2 CPC) 230000 m^3 year	
	€/m^3	%	€/m^3	%	€/m^3	%
Reagents	4.26	14.6	4.26	40.9	4.26	58.8
Electric power	0.43	1.5	0.21	2.0	0.21	2.9
Manpower	17.11	58.6	2.67	25.6	0.36	5.0
Capital costs (solar field)	1.96	6.7	1.52	14.6	1.52	21.0
Capital costs (others)	5.43	18.6	1.75	16.8	0.89	12.3
Total (€ m^{-3})	**29.2**		**10.4**		**7.2**	

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SPECIAL ISSUES ABOUT SOLAR PHOTOCATALYSIS

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