Optical chemical sensors for food freshness

Aleksandra LOBNIK, Polonca NEDELJ KO, Matejka TUREL, Miha VRATIČ, Andreja GUTMAHER

18-20 April 2018, MassTwin Antwerpen Workshop
IOS, Institute of Environmental Protection and Sensors

http://www.ios.si/

PATENT:

PATENT:

PATENT:

PATENT:
Sensor Applications

- Optical Chemical and Bio-Sensor Systems

Environment
- Nanosensors for detection of pollutants
  - detection of heavy metals, pesticides, phosphates, antibiotics
  - detection of O₂, CO, CO₂, NH₃, nitrites, organic pollutants

Safety and Protection
- Nanosensors for Safety and protection, e.g.
  - food safety (for detection of freshness, preservatives, pesticides, etc.)
  - protection against UV radiation and toxic substances
  - personal protection (for firemen, soldiers)

Health
- Nanosensors for Health, e.g.
  - detection of emotional states for individuals (e.g., detection of noradrenaline, adrenaline, dopamine, serotonin, cortisol)
  - detection of biogenic amines in saliva/urine (identification of cancer)
DEFINITION OF SENSORS

Chemical Sensors are miniaturized analytical devices that can deliver real-time and on-line information on the presence of specific compounds or ions in complex samples.

Nanosensors – using nanomaterials or nanotechnologies to prepare nano sensor receptors
# Chemosensors and Biosensors

Sensing versus Analyzing: the fundamental differences

<table>
<thead>
<tr>
<th><strong>Chemical Analysis</strong></th>
<th><strong>Chemical Sensing</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>sample treatment possible</td>
<td>sample treatment not desired or even not possible</td>
</tr>
<tr>
<td>result obtained after some time</td>
<td>result obtained on-line ((=) continuously)</td>
</tr>
<tr>
<td>results obtained in laboratory</td>
<td>results obtained in-situ</td>
</tr>
<tr>
<td>pH can be adjusted</td>
<td>pH cannot be adjusted usually</td>
</tr>
<tr>
<td>preconcentration possible</td>
<td>preconcentration not possible</td>
</tr>
<tr>
<td>includes manual operation</td>
<td>usually fully automatted</td>
</tr>
<tr>
<td>chemical &amp; instrumental</td>
<td>instrumental only</td>
</tr>
</tbody>
</table>
## Sensor Types

<table>
<thead>
<tr>
<th>Electrical</th>
<th>Optical</th>
<th>Others</th>
</tr>
</thead>
<tbody>
<tr>
<td>potentiometry</td>
<td>reflectometry</td>
<td>piezo-electric</td>
</tr>
<tr>
<td>coulometry (amperometry)</td>
<td>fluorescence, phosphorescence</td>
<td>quartz micro balance (QMB)</td>
</tr>
<tr>
<td>conductometry</td>
<td>surface plasmon resonance (SPR)</td>
<td>acoustic sensing</td>
</tr>
<tr>
<td>capacitive (impedimetry)</td>
<td>interferometry</td>
<td>calorimetry</td>
</tr>
<tr>
<td>.....</td>
<td>infrared, Raman, evanescent wave, ....</td>
<td>.....</td>
</tr>
</tbody>
</table>
Sensor Technology Involves …

* Spectroscopy, Electrochemistry, etc.
* Polymer Chemistry
* Physical Chemistry
* Organic and Inorganic Materials
* Interface chemistry
* Biochemistry
* Nanotechnology
* Analytical Chemistry
* Computational Chemistry (AANs)
“Optrode” - (from optical electrode) and “optode” (from Greek - the optical way)

- **intrinsic optical property** of the analyte is utilized for its detection

- **indicator (or label) based** sensing is used when the analyte has no intrinsic optical property

- **(FOCSs)** represents a subclass of chemical sensors in which an optical fiber is used as part of the transduction element.
OPTICAL CHEMICAL/BIO-SENSOR system

analyt

chemical, biosignal

receptor

analytical signal
FIBRE OPTICAL CHEMICAL SENSORS

- optical fiber is used to transmit the EM radiation to and from a sensing region
- remote sensing
The major advantages:

- Optodes do not require a reference cell.
- Miniaturization.
- Remote sensing.
- No interferences to strong magnetic fields/pressure.
- Multiple analysis with a single instrument.
Disadvantages:

- Ambient light can interfere.
- Limited long-term stability because of photobleaching or wash-out of the immobilized indicator.
- Mass transfer of the analyte from the sample into indicator phase is necessary in order to obtain a steady-state signal.
- Limited dynamic range.
- Selectivity of indicators and the immobilization techniques are to be improved.
ANALYTICAL ASPECTS OF SENSORS

• sensitivity and selectivity for the analyte
• broad dynamic range
• reversibility
• lack of frequent calibration
• fast response
• small size
Analytical aspects of sensors

• sensitivity in the range of interest
• selectivity for the analyte
• broad dynamic range
• reversibility
• lack of frequent calibration
• fast response
• inertness to sample matrix
• small size
Sensitivity ($\Delta S / \Delta C$); Note: by definition of IUPAC, sensitivity is **not** the limit of detection!

- LOD (usually 3x the noise $N$) $$
- Dynamic range
- Selectivity
- Linearity (linear range)
- Resolution (smallest difference in concentrations that is detectable)
The best chemical sensor ...

... is the pH electrode since it

* measures over 10 log concentration units
* acts fully reversibly
* is very stable over time
* is not expensive
* is sterilizable
* has been optimized over 60 years.

Even though

* it could be smaller
* it could be even more selective
Design of optical chemical sensor
„Indicator chemistry“

Indicator
Polymer matrix
Immobilization

Sensor characteristics
Indicators

Absorbance based:
- Undergo colour change
- Detection by “naked eye
- Detection by colorimetry

Luminescence based:
- Fluorescence
- Phosphorescence
- Chemilumininiscence
- Electroluminiscence
- Detection by:
  - UV lamp
  - Intensity change
  - Lifetime measurements
Fluorescent intensity and lifetime based “Lanthanide chelates”

- narrow, line-like emission peaks
- large Stokes’ shifts (≥ 200 nm)
- long lifetimes (from micro- to several mili-seconds range)
  → lifetime-based assays advantageous over intensity-based: highly immune to photobleaching, changes in fluorophore concentration, turbidity in the sample, optical misalignment, etc.
- long lifetimes enable gated detection mode
  → time-resolved luminescence
Time-resolved luminescence

- pulsed excitation
- background fluorescence (nano seconds)
- measuring window (integration time)
- Ln-complex emission (longer emission of photons; micro- to miliseconds)

After »short-lived« fluorescence has ceased away, only »long-lived « luminescence of Ln-complex is measured.
Polymer carrier

* hydrophobic
  - PVC, PMMA, PE, PS, ...
  - Detection of gases

* hydrophilic
  - Polysaccharides, polyacrilates, polyamines, hydrogels...
  - Ion detection

* hidrophobic-hydrophilic
  - Sol-gel
  - Detection of ions and gases
**Sol-gel process (Silica nanoparticles )**

1. *hydrolysis*

\[
\text{Si(OR)}_4 + \text{H}_2\text{O} \rightarrow \text{HO-Si(OR)}_3 + \text{ROH}
\]

2. *condensation:*

\[
\text{HO-Si(OR)}_3 + \text{HO-Si(OR)}_3 \rightarrow \text{HO-Si(OR)}_2\text{-O-Si(OR)}_2 + \text{HOH}
\]

\[
\text{HO-Si(OR)}_3 + \text{Si(OR)}_4 \rightarrow \text{Si(OR)}_3\text{-O-Si(OR)}_3 + \text{ROH}
\]

<table>
<thead>
<tr>
<th>Monomers</th>
<th>Other metals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si(OR')_4</td>
<td>Zr(OR')_4</td>
</tr>
<tr>
<td>R_1-Si(OR')_3</td>
<td>R_1-Zr(OR')_3</td>
</tr>
<tr>
<td>R_1 R_2-Si(OR')_2</td>
<td>R_1 R_2-Zr(OR')_2</td>
</tr>
<tr>
<td>R: aliphatic, aromatic</td>
<td>Ti, Sn</td>
</tr>
</tbody>
</table>
ADVANTAGES OF USING NANOMATERIALS FOR SENSORS

- Improved sensor characteristic (response time, sensitivity, etc.)
- In-vivo measurements,
- Small sample volumes,
- Multi-analyte sensing
Design of Optical nanosensor

- a, macromolecular nanosensors (dendrimers);
- b, NSs based on polymer materials and sol-gels;
- c, multi-functional core-shell systems;
- d, multi-functional magnetic beads;
- e, NSs based on quantum dots;
- f, NSs based on metal beads

Silica nanoparticles - Sol-gel process

1. *hydrolysis*

\[
\text{Si(OR)}_4 + \text{H}_2\text{O} \rightarrow \text{HO-Si(OR)}_3 + \text{ROH}
\]

2. *condensation*:

\[
\begin{align*}
\text{HO-Si(OR)}_3 + \text{HO-Si(OR)}_3 & \rightarrow \text{HO-Si(OR)}_2\text{-O-Si(OR)}_2 + \text{HOH} \\
\text{HO-Si(OR)}_3 + \text{Si(OR)}_4 & \rightarrow \text{Si(OR)}_3\text{-O-Si(OR)}_3 + \text{ROH}
\end{align*}
\]

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<tr>
<td>Si(OR’)_4</td>
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</tr>
<tr>
<td>R₁-Si(OR’)_3</td>
<td>R₁-Zr(OR’)_3</td>
</tr>
<tr>
<td>R₁ R₂-Si(OR’)_2</td>
<td>R₁ R₂-Zr(OR’)_2</td>
</tr>
<tr>
<td>R: aliphatic, aromatic</td>
<td>Ti, Sn</td>
</tr>
</tbody>
</table>
Applications in Food safety: Food freshness

Food freshness depends on microbiological activity and various Biogenic amines are formed and released.

Amino acid

\[
\text{H}_2\text{N} - \text{CH} - \text{COOH}
\]

\[
\text{decarboxylation}
\]

\[
\text{amino acid}
\]

\[
\rightarrow
\]

\[
\text{biogenic amine}
\]

\[
\text{R} - \text{CH}_2\text{NH}_2 + \text{CO}_2
\]
METHODS FOR BIOGENIC AMINE DETERMINATION

• Instrumental or classical methods
  • High-performance liquid chromatography (HPLC)
  • Thin-layer chromatography (TLC)
  • Gas chromatography (GC)
  • Micellar electrokinetic chromatography (MEKC)

• Other methods
  • electrochemical methods (capillary electrophoresis) (CE)
  • enzymatic methods (biosensors)

• Optical methods
  • Optical chemical sensors (OCS)

Derivational reagents:
  • dansyl chloride,
  • benzoyl chloride,
  • dansyl chloride,
  • fluorescein,
  • 9-fluorenymethyl chloroformate,
  • naphthalene-2,3-dicarboxaldehyde
  • orthophthalaldehyde (OPA)
OPTICAL DETERMINATION OF BA BY O-PHTHALDIALDEHYDE (OPA)

- Spectral properties
- BA determination in real samples
- Response time
- OPA sensitivity to other BA
- The impact of the concentration of OPA
- Influence of pH medium

1. Agmatine
2. Serotonin
3. Dopamine
4. Octopamine,
5. Tyramin
6. Ethanolamine
7. Spermidine
8. Cadaverine
9. methyl-fenilethylamine
10. Noradrenalin
11. Adrenalin
12. Isopentlylamine
13. Melatonin
14. Acetilholin chloride
15. Putrescine
16. Isopropanolamine
17. β-alanine
18. Histamine
19. Cysteamine
20. Spermine
OPTICAL DETERMINATION OF BA IN SOLUTION

Spectral properties

AGMATINE

Response time

Emission spectra of OPA-AgmS product  
$6,0 \times 10^{-7} \text{ M} - 1,0 \times 10^{-4} \text{ M}$

Calibration curve of fluorescent product OPA-AgmS  
$6,0 \times 10^{-7} \text{ M} - 8,0 \times 10^{-6} \text{ M}$  
$\text{LOD} = 2,5 \times 10^{-7} \text{ M}$

$y = 1,53 + 81,47x$
$r = 0,9989$

Analytical letters, 2015, vol. 48, iss. 10, str. 1619-1628
OPTICAL DETERMINATION OF BA BY O-PHTHALDIALDEHYDE (OPA)
Characterization of SiO₂ particles

TEM, SEM, FT-IR, BET, potentiometric titration, zeta potential

TEM (on the left side) and SEM (on the right side) images of SiO₂ particles which they have been prepared based on precursor TEOS with different molar ratios (R): (a) R=4, (b) R=20, (c) R=40, (d) R=80.
# Summary

Comparison of the results based on the optical determination of AgmS in solution with and without SiO$_2$-SH-OPA particles at pH 13

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Optical determination of AgmS with OPA</th>
<th>Optical determination of AgmS with SiO$_2$-SH-OPA particles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluorescent product</td>
<td>OPA-AgmS</td>
<td>SiO$_2$-SH-OPA-AgmS</td>
</tr>
<tr>
<td>Spectral properties ($\lambda_{ex}/\lambda_{em}$)</td>
<td>340 nm / 473 nm</td>
<td>340 nm / 430 nm</td>
</tr>
<tr>
<td>Concentration range</td>
<td>$6.0 \times 10^{-7} \text{ M} - 8.0 \times 10^{-6} \text{ M}$</td>
<td>$1.0 \times 10^{-5} \text{ M} - 1.0 \times 10^{-2} \text{ M}$</td>
</tr>
<tr>
<td>The correlation coefficient $r^2$</td>
<td>0.9989</td>
<td>0.9989</td>
</tr>
<tr>
<td>Linear equation</td>
<td>$y = 1.53 + 81.47x$</td>
<td>$y = 5.43 + 0.71x$</td>
</tr>
<tr>
<td>LOD</td>
<td>$2.5 \times 10^{-2} \text{ M}$</td>
<td>$7.3 \times 10^{-7} \text{ M}$</td>
</tr>
<tr>
<td>Response time</td>
<td>20 min</td>
<td>2 – 3 min</td>
</tr>
<tr>
<td>Buffer</td>
<td>pH 13</td>
<td>pH 13</td>
</tr>
</tbody>
</table>

*Journal of Sol-Gel Science and Technology, 2016, 1-10*
• The sensor is suitable for raw, untreated fish and chicken meat
• Color change is a measure of the usefulness of the meat (see color scale)
• Response time is 30 minutes
• The sensor is useful when blue coloration is reached (spoiled meat) and can be used again if the initial color was yellow
Correlation to the microbiological measurements

- sensor absorption measurements on spectrophotometer (laboratory)
- monitoring of the activity of microbiological parameters - bacteria Pseudomonas spp. (PSDM)
- signal of the sensor in correlation with the increase in the number of bacteria Pseudomonas spp.
OPTICAL DETERMINATION OF BA BASED

Freshness of the food in fridge

In the meet package
Design and characterization of azo (dicyanovinyl) dyes for the colorimetric detection of thiols and biogenic amines detection

Chemical structures of the azobenzene dyes CR-528 and CR-555 before and after the reaction with 2-mercaptoethanol (2-ME).

T. Mastnak, A. Lobnik, Sensors, 2018
Design and characterization of dicyanovinyl reactive dyes for the colorimetric detection of thiols and biogenic amines detection

Absorption spectra of CR-528 ($7.2 \times 10^{-6}$ M; A) and CR-555 ($7.9 \times 10^{-6}$ M; B) in the presence of various concentrations of 2-ME (from $0$ to $4.8 \times 10^{-4}$ M) in ethanol solution.

T. Mastnak, A. Lobnik, Sensors, 2018
Design and characterization of dicyanovinyl reactive dyes for the colorimetric detection of thiols and biogenic amines detection

Spectrophotometric titrations (calibration curves) for sulfur-based analytes (NaHS, 2-ME; A, B) and for amine-based (BA) analytes (spermine, spermidine, ethanolamine; C, D); n = 3.

T.Mastnak, A. Lobnik, Sensors, 2018
# Design and characterization of dicyanovinyl reactive dyes for the colorimetric detection of thiols and biogenic amines detection

<table>
<thead>
<tr>
<th>Indicator</th>
<th>Analyte</th>
<th>Working Range (molL(^{-1}))</th>
<th>Response time (min)</th>
<th>Remark</th>
<th>[Ref.]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CR-528</td>
<td>Spermine</td>
<td>(3 \times 10^{-6} - 1.2 \times 10^{-4})</td>
<td>30</td>
<td>A, ethanol solution</td>
<td>our work</td>
</tr>
<tr>
<td></td>
<td>Spermidine</td>
<td>(3 \times 10^{-6} - 1.2 \times 10^{-4})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ethanolamine</td>
<td>(5 \times 10^{-5} - 1 \times 10^{-3})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>NaSH</td>
<td>(2 \times 10^{-4} - 3 \times 10^{-2})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2-ME</td>
<td>(3 \times 10^{-3} - 3 \times 10^{-1})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CR-555</td>
<td>Spermine</td>
<td>(2 \times 10^{-6} - 2 \times 10^{-5})</td>
<td>30</td>
<td>A, ethanol solution</td>
<td>our work</td>
</tr>
<tr>
<td></td>
<td>Spermidine</td>
<td>(5 \times 10^{-6} - 2.5 \times 10^{-5})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ethanolamine</td>
<td>(2 \times 10^{-5} - 3.1 \times 10^{-4})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>NaSH</td>
<td>(1.2 \times 10^{-5} - 2.5 \times 10^{-4})</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2-ME</td>
<td>(1.5 \times 10^{-4} - 1 \times 10^{-2})</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Organophosphate (pesticide) fluorescence sensor based on thin film and SiO$_2$ NP
A. Lobnik, EU patent, USA and Russian patent

<table>
<thead>
<tr>
<th>Sensor configuration</th>
<th>$t_{95}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thin-film</td>
<td>600 s</td>
</tr>
<tr>
<td>Silica NP</td>
<td>12 s</td>
</tr>
</tbody>
</table>
Comparison of OP sensor characteristics
(A. Lobnik, Š. Korent Urek, EU, USA, Russia patents)

<table>
<thead>
<tr>
<th></th>
<th>Dye-doped thin films</th>
<th>Dye-doped nanoparticles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Limit of detection (mol/L)</td>
<td>6.7×10^{-7}</td>
<td>0.17×10^{-9}</td>
</tr>
<tr>
<td>Working range (mol/L)</td>
<td>6.9×10^{-7} – 6.9×10^{-3}</td>
<td>0.17×10^{-9} – 2.3×10^{-7}</td>
</tr>
<tr>
<td>Response time (s)</td>
<td>600</td>
<td>12</td>
</tr>
</tbody>
</table>
Luminiscence measurements of OP
Biosensing layer for OP determination

(N. Francic, A. Lobnik, E. Efremenko, Bioscience and Technology, 2012)

• **His$_6$-OPH (EC 3.1.8.1.)** – organophosphorous hydrolase
  - Enzyme hydrolyzing a broad spectrum of organophosphorous compounds (OPCs) containing P–O, P–F and P–S bonds in the triesters of orthophosphoric acid
  - Metalloenzyme: cofactors are Co$^{2+}$ and other bivalent ions
    - Optimal activity:
      - $T = 45 - 53$ °C (pH 10.5)
      - pH between 10 in 11.5
    - High specific activity: ~ 5000 U/mg

• **hexahistidine (His$_6$) tag fused to OPH** → improving the catalytic efficiency, especially towards P–S-containing substrates, and the stability under alkaline hydrolysis conditions compared to native OPH
Comparison of two types of biocatalyst films TEOS/GPTMS (R=188, P=5:1) and TMOS/MTMOS (R=148, P=1:2) for a) repeated use in the detoxification of POX. Conditions: 0.675 mM paraoxon, temperature 25 °C, 50-mM Na-carbonate buffer (pH 9.5); and b) stability of SiO₂ thin films with entrapped His₆-OPH

Anal Bioanal Chem (2011) 401:2631–2638
Silica particles with immobilized His$_6$-OPH for POX determination/detoxification

TEM micrographs of silica particles. (A-B), SEM microgram (C), and particle size distribution (D) of MPS 5 particles.
Silica particles with immobilized His$_6$-OPH for POX determination/detoxification

Fig. 4: Cycles of usage (a) and stability (b) of silica particles with immobilized His$_6$-OPH.
Mesoporous TiO$_2$ thin films as efficient enzyme carriers for paraoxon determination/detoxification

Schematic representation of the preparation route of His$_6$-OPH-conjugated mesoporous titania thin films trough CDI mediated reaction.
Mesoporous TiO$_2$ thin films as efficient enzyme carriers for paraoxon determination/detoxification

Figure Cycles of usage for covalently attached His$_6$-OPH, TiF-10 and TiF-bim (black and grey squares), and adsorbed His$_6$-OPH, TiF-10, TiF-10 and TiF-bim (black and grey circles). Measurements were performed with selected 50 mm$^2$ bio-functionalized mesoporous titania thin-films with covalently attached His$_6$-OPH at 20 °C and pH 10.5 (CB, 50 mM). Substrate: 0.3 mM paraoxon.

Figure Stability of titania bio-sensing film (TiF-9) with covalently attached enzyme several days after film preparation.

Analyst, 2014, 139, 3127–3136
Design and investigation of optical properties of $N$-(Rhodamine-B)-lactam-ethylenediamine (RhB-EDA) fluorescent probe
(E. Soršak, A. Lobnik, Sensors 2018)

The linear range for Ag$^{+}$ ions' detection is from $0.43 \cdot 10^{-3}$ to $10^{-6}$ M.
Applications in water: Determination of phosphate (P) using europium-tetracycline complex (EuTc)


\[ y = A + B \times \log(c_P \times 10^6) \]

<table>
<thead>
<tr>
<th></th>
<th>0 µs lag.t.</th>
<th>60 µs lag.t</th>
</tr>
</thead>
<tbody>
<tr>
<td>40 µs int.t.</td>
<td>1,033 ± 0,018</td>
<td>0,955 ± 0,081</td>
</tr>
<tr>
<td>100 µs int.t.</td>
<td>-0,224 ± 0,007</td>
<td>-0,586 ± 0,091</td>
</tr>
</tbody>
</table>

\( r^2 \) = 0,993, 0,954

Ex filter = 405±30 nm, Em filter = 612±10 nm
Determination of copper (Cu) using terbium-ligand complex (TbL₂)

Spectral properties of TbL₂ in water solution

lag time = 50 µs, integration time = 1000

F/F₀ = A + B × log(c₉Cu × 10⁹)

r² = 0.996

10-300 nmol/L Cu²⁺
LOD = 10 nmol/L
A new circular economy concept: from textile waste towards chemical and textile industries feedstock

Prof. dr. Aleksandra Lobnik, UMARI, IOS, Ltd.
Resynetex Co-coordinator, Technical Manager
COORDINATOR: SOEX
CO-COORDINATOR: IOS

20 project partners from 10 different EU members: industrial associations, enterprises, small and medium-sized enterprises, research institutions.

Together, we create an effective model for the whole value chain.
The RESYNTEX project is considering and demonstrating the whole value chain starting from:

- Citizen behaviour study and change
- Data aggregation and traceability
- New business models
- LCA/LCC
- Communication and promotion

New collection approaches for unwearable mixed textile waste
Automatic sorting and pre-cleaning of textile blends
Cascading separation of textile components

- From proteins to sustainable resins and panels
- From cellulose (sugars) in solution to ethanol conversion
- From polyamide (oligomers) to chemical products
- From polyester (monomers) to PET production

New governance and methods for successful symbiosis of unwearable textile waste

New opportunities for industrial symbiosis and replication in Europe
Improving legislation and standards for efficient industrial symbiosis

Liquid and solid waste management and treatment

Demonstration of semi works pilot plant facility (500 t/y) in industrial environment
RESYNTEX PROJECT WORK STRUCTURE

What are the Afams of the Resyntex project

As a result, **economic advantages** can be provided besides prevention of industrial environmental problems

<table>
<thead>
<tr>
<th>Feedstock from textile waste</th>
<th>Obtained chemical products from symbiosis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Protein hydrolysates</td>
<td>Adhesives used for the manufacturing of wood-based panels <em>(CHIMAR)</em></td>
</tr>
<tr>
<td>Depolymerized cellulose (sugars in solution)</td>
<td>Cellulosic ethanol manufacturing based on the PROESA® technology <em>(CTX1)</em></td>
</tr>
<tr>
<td>Polyamide oligomers (mainly polyamide 6 and 66)</td>
<td>Hydrogenolysis/hydrolysis of PA6 and PA6,6 leading to hexanoic acid, N-pentylamine (also known as amylamine), aminohexanoic acid, caprolactam and other important chemical intermediates, <em>(Arkema)</em></td>
</tr>
<tr>
<td>PET monomers (terephthalic acid, ethylene glycol)</td>
<td>Reaction of alcohols and PA6,6 leading e.g. to diesters (e.g. as solvents) and hexamethylenediamine <em>(Arkema)</em></td>
</tr>
<tr>
<td></td>
<td>Plastic bottles constituted of PET from the reaction of terephthalic acid (PTA) and ethylene glycol (EG) <em>(CTX1)</em></td>
</tr>
</tbody>
</table>
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