

Sludge recovery from industrial wastewater treatment

M. T. Santos¹, P. Lopes¹

¹ Área Departamental de Engenharia Química, Instituto Superior de Engenharia de Lisboa - ISEL, Instituto Politécnico de Lisboa

- **Introduction – Industrial sludges**
- **Goals**
- **Materials and Methods**
- **Results and discussion**
- **Conclusions**
- **Future perspectives**



Main Industries in Portugal

Food

Textiles

Chemicals

Paper

Wood and Cork



Industrial wastewater (IWW)

Composition – depends on the type of industrial activity.

Industrial sludges

With proper treatment are
valuable material resources



**sustainable circular
economy**

How to treat industrial wastewater?

Based on **processes and operations** units more suited to its characteristics.

➤ Industrial WWTP

Liquid Phase Treatment



Aeration tank



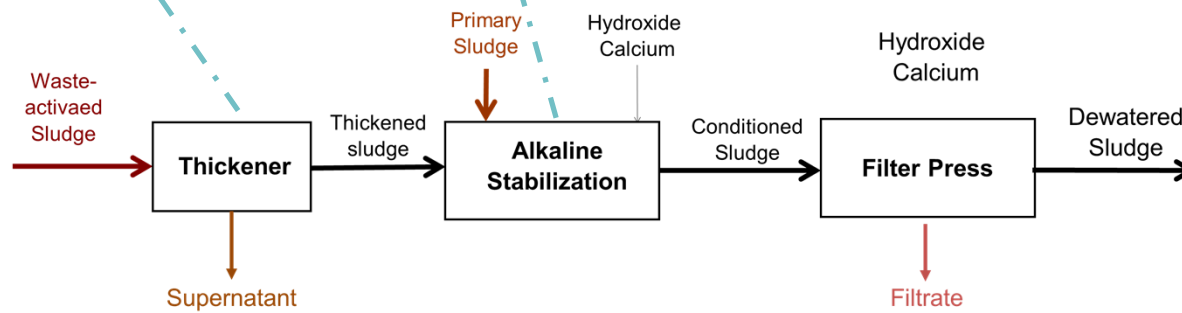
Sedimentation tank



Sludges

How to treat industrial wastewater?

Solid Phase Treatment



Composting or Landfill



Sludges

How to treat or disposal industrial Sludges?

co-composting with MSW – problems due to the sludges composition.

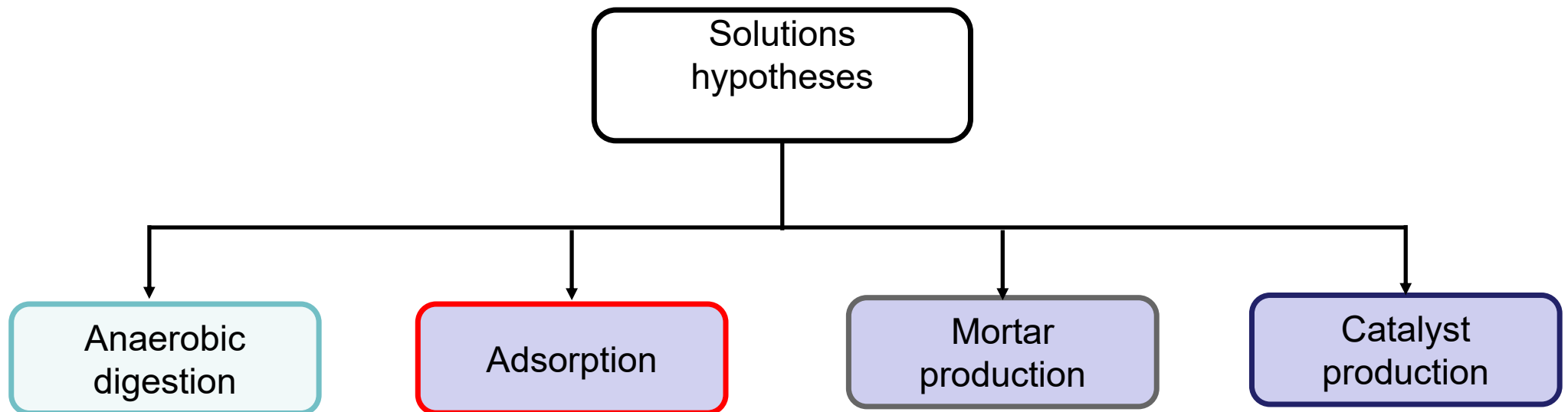


700 ton/year industrial sludges –
Composting 35€/ton e 395€/transport
(31,800€/ year)

Industrial Sludges Composition

Parameter	Units	value
Dry Matter	%m/m	68.2
Organic matter	%m/m	56.2
pH	Escala de Sorensen	12.6
Calcium	g Ca/kg	210
Phosphorus	g P/kg	0.22
Potassium	g K/kg	0.23
Magnesium	g Mg/kg	1.81
zinc	g Zn/kg	0.06

- Identify and analyze treatment/valorization options for these wastes, giving priority to their recovery to the detriment of disposal.



Anaerobic digestion

- ❖ Potential Metanogenic Assay (32 d)
- ❖ 2 substrate mixtures:
 - Reactors A - 80% MS + 20 % IS
 - Reactors B - 90% MS + 10% IS
- ❖ Inoculum – digested sludges from WWTP

MS – Mixed sludge from Municipapl WWTP
IS – Industrial sludge

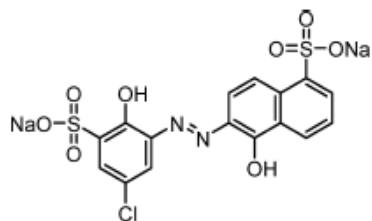
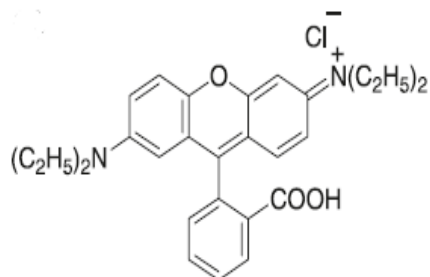
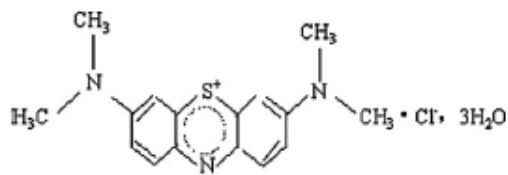
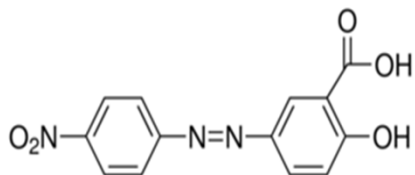
Composition of mixtures for reactors AD

Item	Reactors A (g)	Reactors B (g)	Reactors I (g)
MS	4.85	5.45	-
IS	0.09	0.05	-
Inoculum	19.05	19.05	19.05
water	175.45	175.45	180.95

Anaerobic
digestion

- ❖ Quantitative and qualitative analysis of Biogas
- ❖ Specific methane production





Adsorption

- ❖ Adsorbent (75% ADS e 25 % IS)
- ❖ 4 Dyes:
 - Rhodamine-B - $C_{28}H_{31}N_2O_3C$
 - Mordant Orange 1 - $C_{13}H_9N_3O_5$
 - Methylene Blue - $C_{16}H_{18}N_3SCl$
 - Mordant Blue 9 - $C_{16}H_9ClN_2Na_2O_8S_2$
- ❖ 6 concentrations and Useful of 30 mL
- ❖ Adsorbent masses (0,5, 1, 1,5, 2 e 3 g)
- ❖ Tests of 1h30min with measurements of $\frac{1}{2}$ in $\frac{1}{2}$ h

Mortar production

- ❖ Method: NP EN 191-1: 2006
- ❖ 5 mortar mixtures:
 - 1 reference (cement only)
 - 1%, 2.5%, 5% e 10% Sludge
- ❖ Test time 28 d



Composition	Reference	Sludge (1%)	Sludge (2.5%)	Sludge (5%)	Sludge (10%)
Cement (g)	450.02	445.52	438.71	427.51	405.03
Sand (g)	1,350	1,350	1,350	1,350	1,350
Sludge (g)	-	4.51	11.32	22,51	45.02
Water (g)	225.02	225.01	225.03	225,03	225.02

Mortar
production

- ❖ Bending strength tests
- ❖ Compressive strength tests



Biofuel production - heterogeneous catalysis

- ❖ Used food oils
- ❖ Pre-treatment drying - 70 °C
- ❖ Calcination pretreatment 850 °C
- ❖ Convert calcium (sludge) to calcium oxide.





Biofuel production -
heterogeneous
catalysis

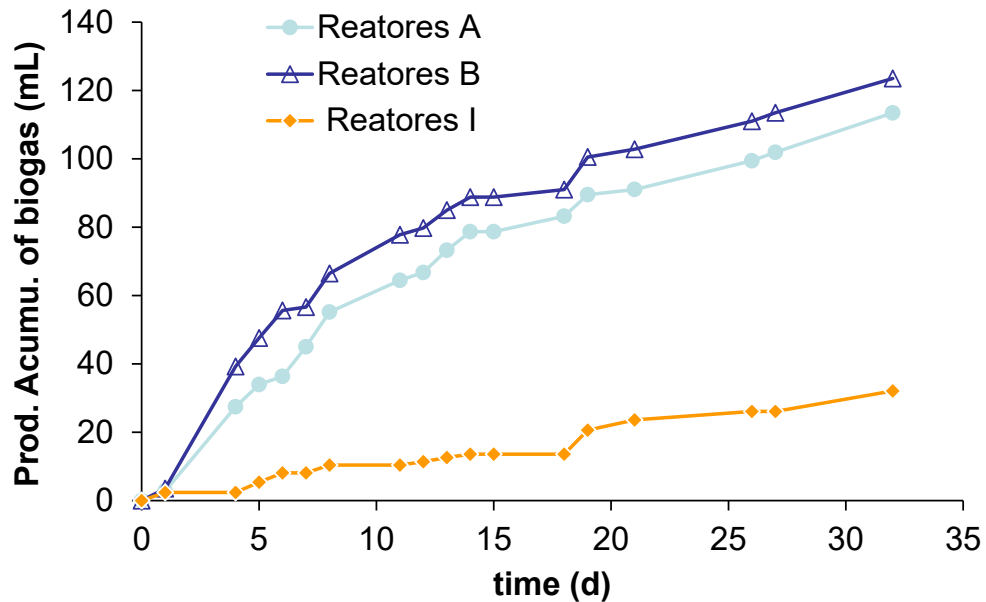
- ❖ FTIR-ATR
- ❖ Acidity index
- ❖ Density



Anaerobic Digestion Assay

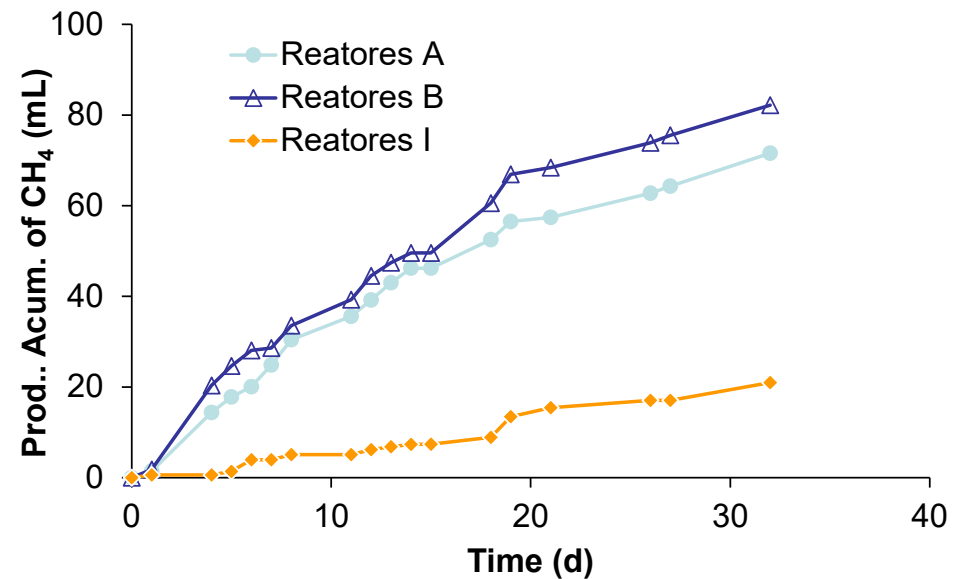
➤ Reactors A

- **21%** - SV removal
- **CH₄ - 55 to 60%**



➤ Reactors B

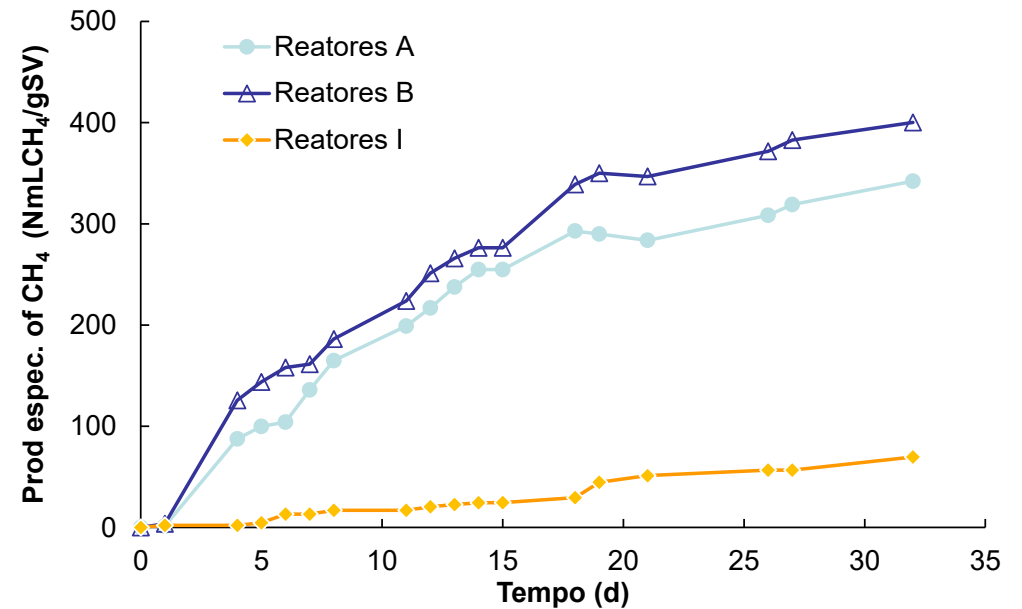
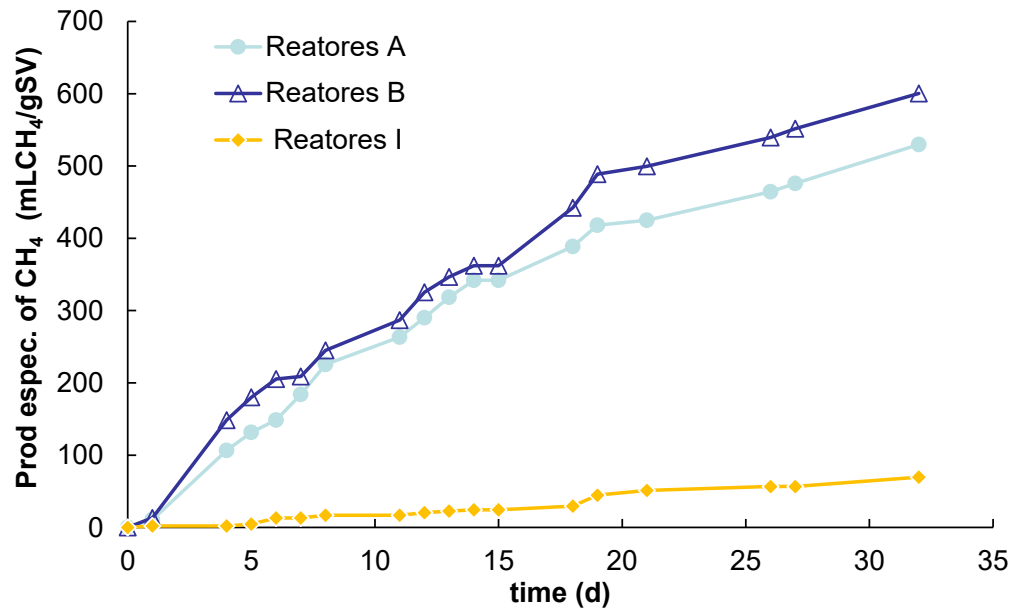
- **42%** SV removal
- **CH₄ - 60 to 65%**



➤ Methane production

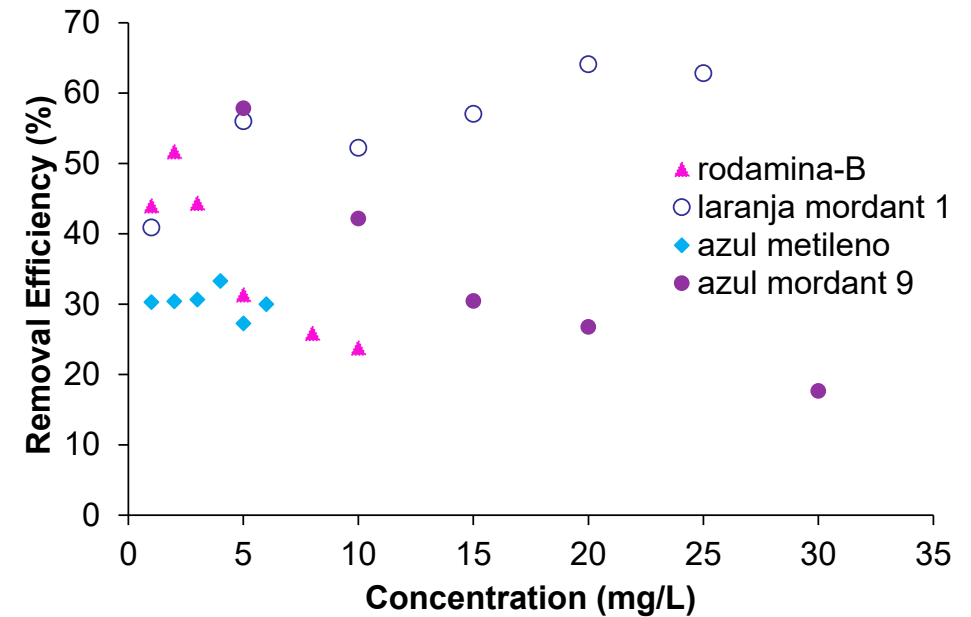
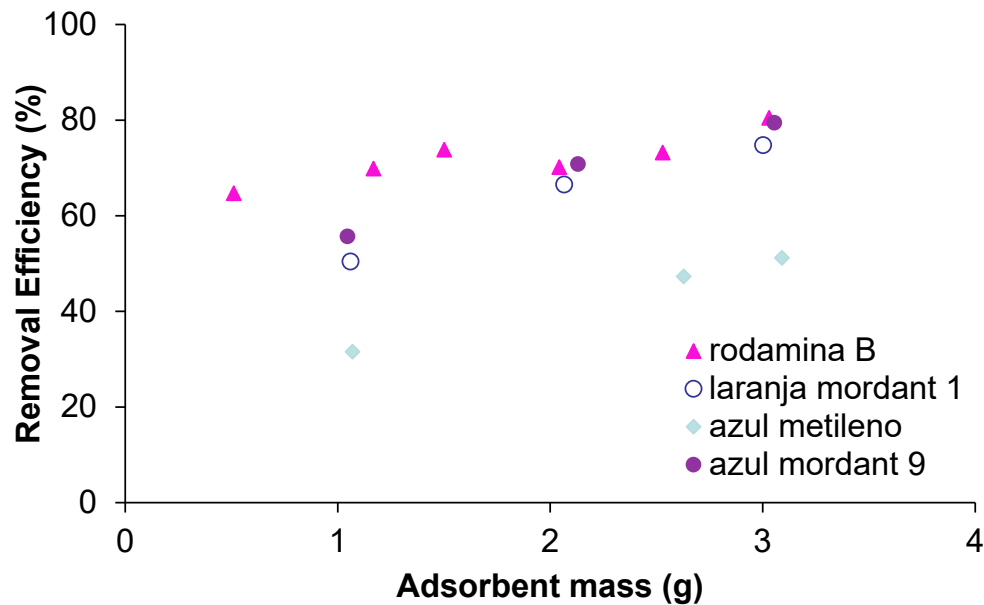
- ❖ Test Conditions – 1 atm and 35°C
- ❖ Conditions PTN – 1 atm and 0°C

Reactors	Prod. acumu. of biogas (mL)	Prod. acumu. of CH ₄ (mL)	Prod. Espec. of CH ₄ (mL CH ₄ /gVS)	Prod espec. of CH ₄ (N.mL CH ₄ /gVS)
A	113	71	529	342
B	123	82	600	400



Adsorption tests

- It was obtained $R^2 \geq 0,989$
- Removal efficiency between **70 to 80%**



- **Isothermics** of adsorption tests for each of the dyes

Linearized equation of Langmuir:

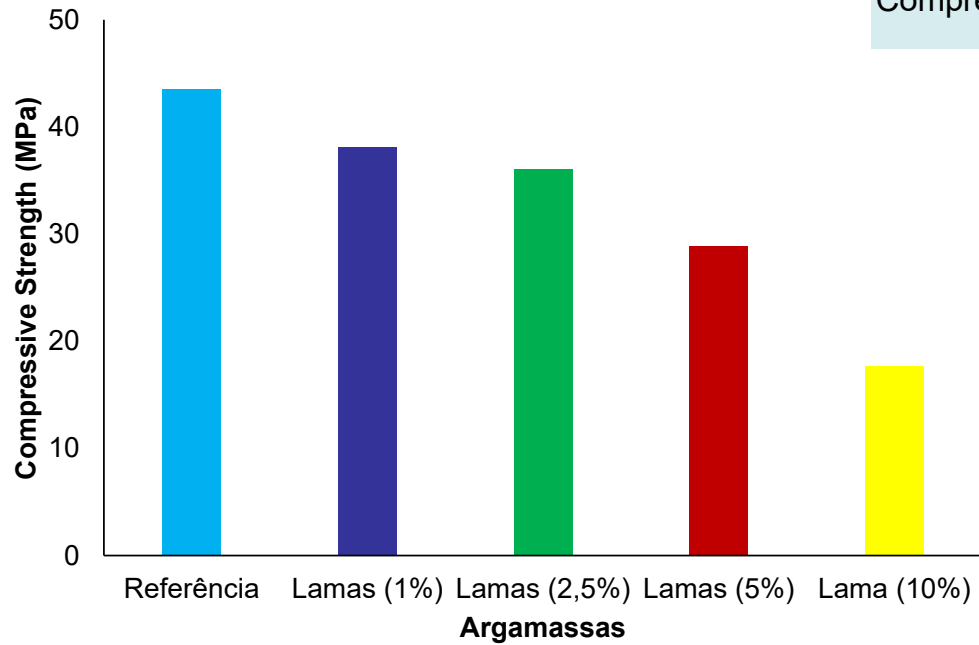
$$\frac{C_e}{q_e} = \frac{1}{q_{\max} k_L} + \frac{1}{q_{\max}} C_e$$

Linearized equation of Freundlich:

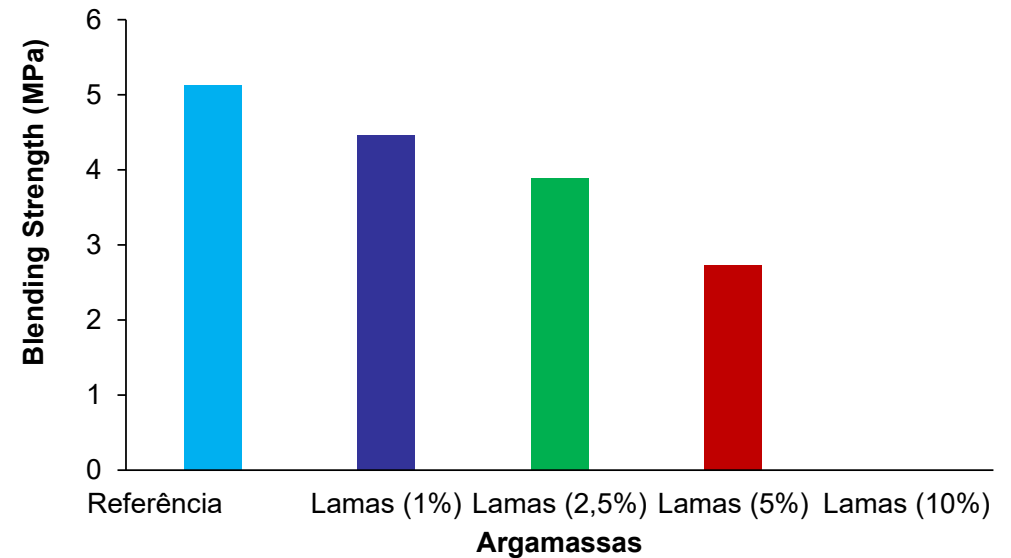
$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$$

- The isothermal that best fits – **Langmuir Isothermic**

- $q_{\max} = 0,780$ mg/g - Mordant Blue 9
- $q_{\max} = 0,274$ mg/g - Rhodamine-B
- $q_{\max} = 0,494$ mg/g - Mordante Oranje 1
- $q_{\max} = 0,010$ mg/g - Methylene Blue

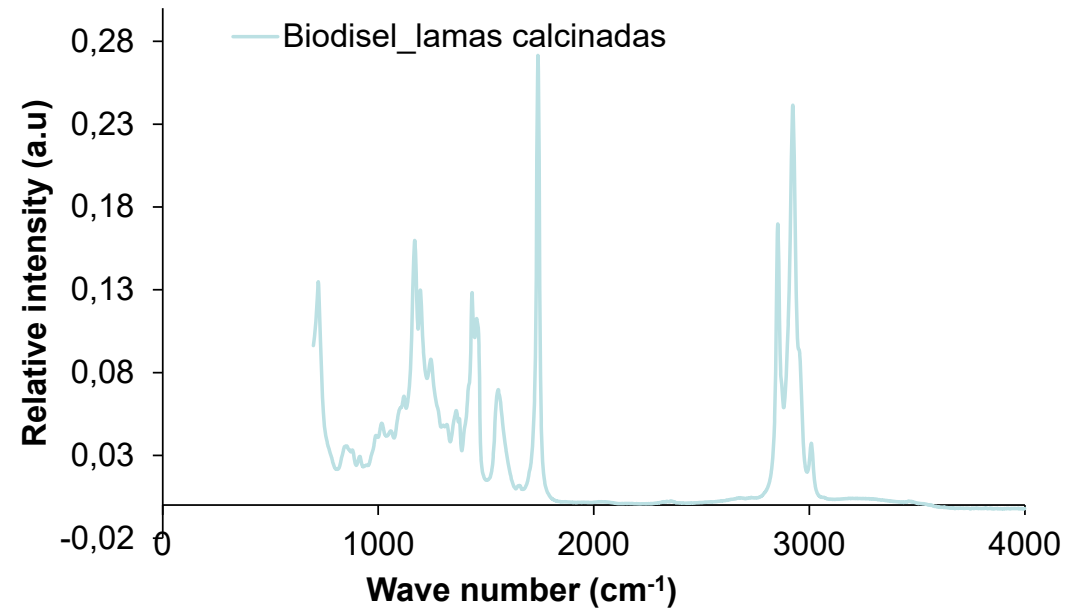
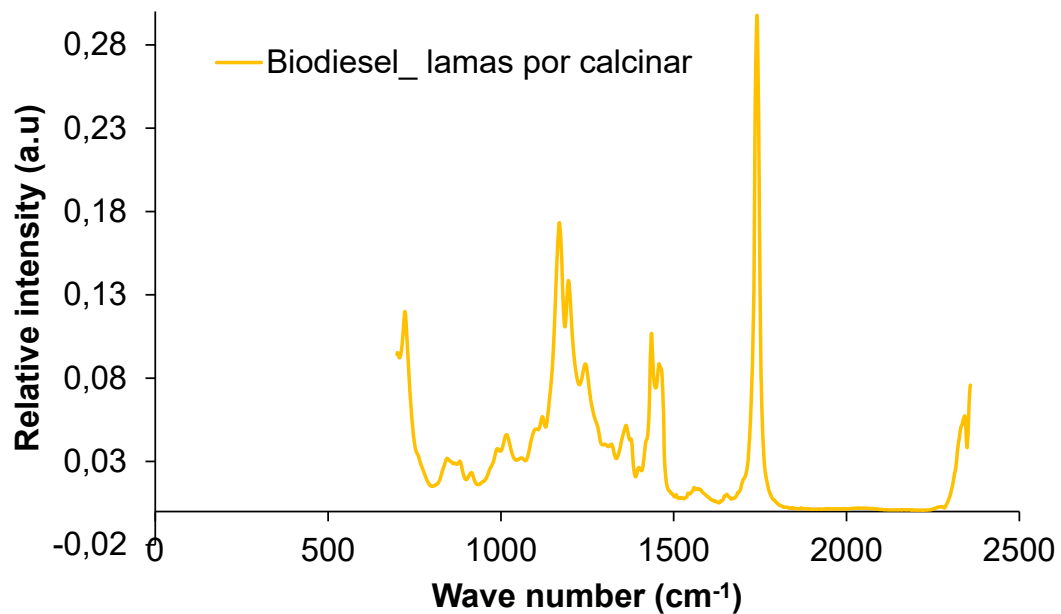


Strength	Reference	Sludge (1%)	Sludge (2.5%)	Sludges (5%)	Sludge (10%)
Blending (MPa)	5.13	4,46	3,89	2,73	-
Compressive (MPa)	43.54	38.14	36.04	28.90	17.12



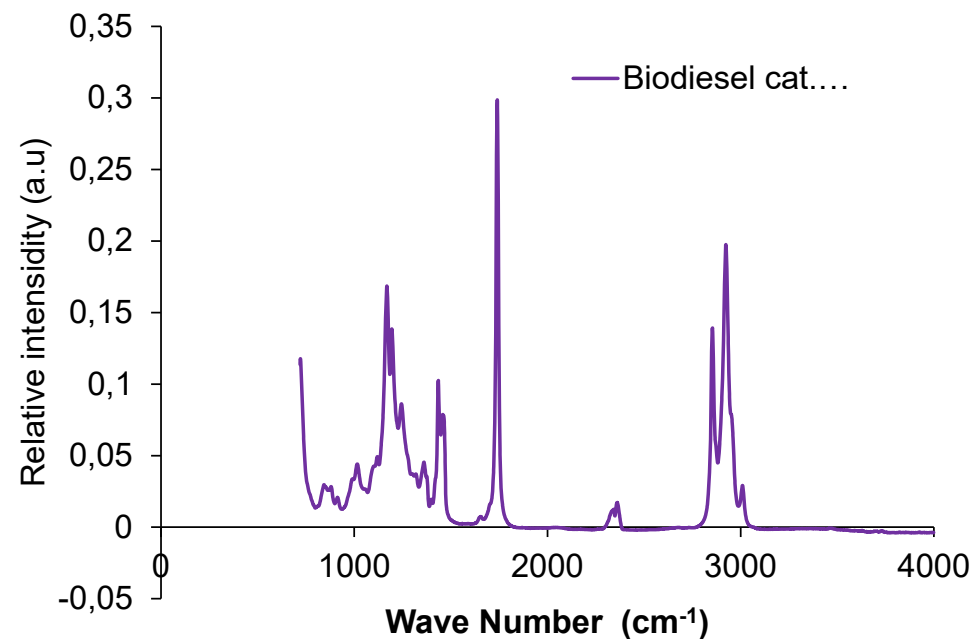
Catalyst production tests for biofuel production

- ❖ Conversion of 90%
- ❖ Acidity index : **0.17 e 0.18**



➤ Functional groups (FTIR-ATR)

Chemical bonding	Function	Wave Number (cm ⁻¹)
-O-H	Alcohol, phenol, enol, carboxylic acid	3650 – 3200
-R ₂ NH	Secondary Aminas	3400 – 3140
-NH ₂	Primary amines	3400 – 3350
-C-H	Alkanes	2962 – 2853
-C-H	Alkenes	3095 – 3010
-CO-C-H	Aldehydes	2900 – 2800
-C≡C-, C≡N	Alcinos and Nitrites	2500 – 2000
R ₂ C=O	Carbonates	1630 – 1850
H ₂ C=CH ₂	Alkenes	1680 – 1650
-C=C-	Aromatics	1450 – 1500



- It is possible to apply **anaerobic co-digestion** with energy recovery. Specific methane production **0.400 L/gSV added**, 4 kWh/kg SV corresponding to **2 kWh/kg industrial sludge**.
- It is possible to produce **adsorbents for removal of water dyes**. With blue mordant 9, a maximum removal of 80% - $q_{max} = 0.780$ mg/g was obtained .
- The **production of mortar** - Visual analysis similar aspect between the specimens with the sludge and the reference. The **bending and compression strengths are lower than** the reference resistance but present acceptable values.
- **Catalyst production - biofuel production was achieved 90% conversions and acidity indexes < 0.5.**

➤ **Cement production:**

- Conduct pilot-scale trials

➤ **Anaerobic digestion:**

- IS with fractions >20%
- different substrates.

➤ **Adsorption:**

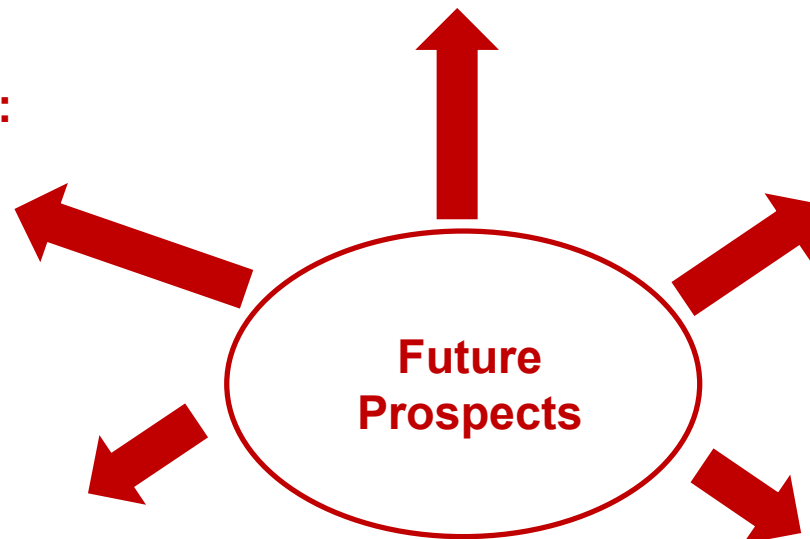
- Apply the adsorbent – different pollutants.

➤ **Mortar production:**

- Curing times less than 28 d
- Replace the sand fraction.

➤ **Production of Heterogeneous Catalysts:**

- Test the number of uses.



Acknowledgements:

We thank to the Chemical Industry for the sludge and data.

email: tsantos@deq.isel.ipl.pt

Thank you for your attention

Questions ?

