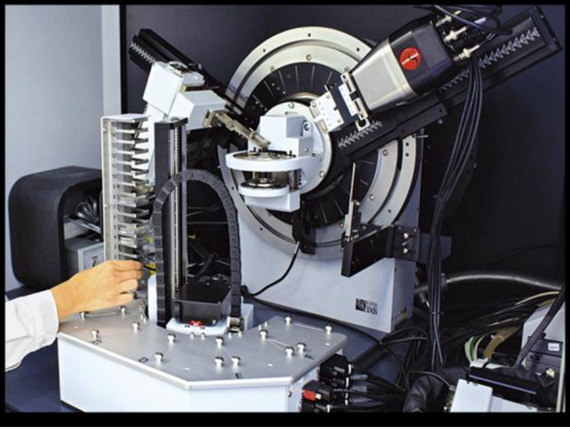


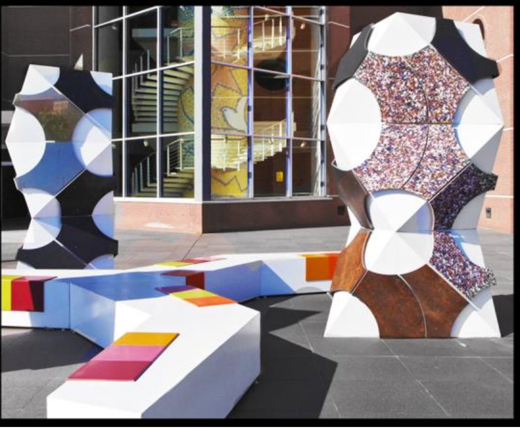

ESPECTROMET – Development of new methodologies for the characterisation of materials by spectroscopic techniques

IMDEEA/2017/119

February 2018

UNIÓN EUROPEA
Fondo Europeo de Desarrollo Regional
Una manera de hacer Europa

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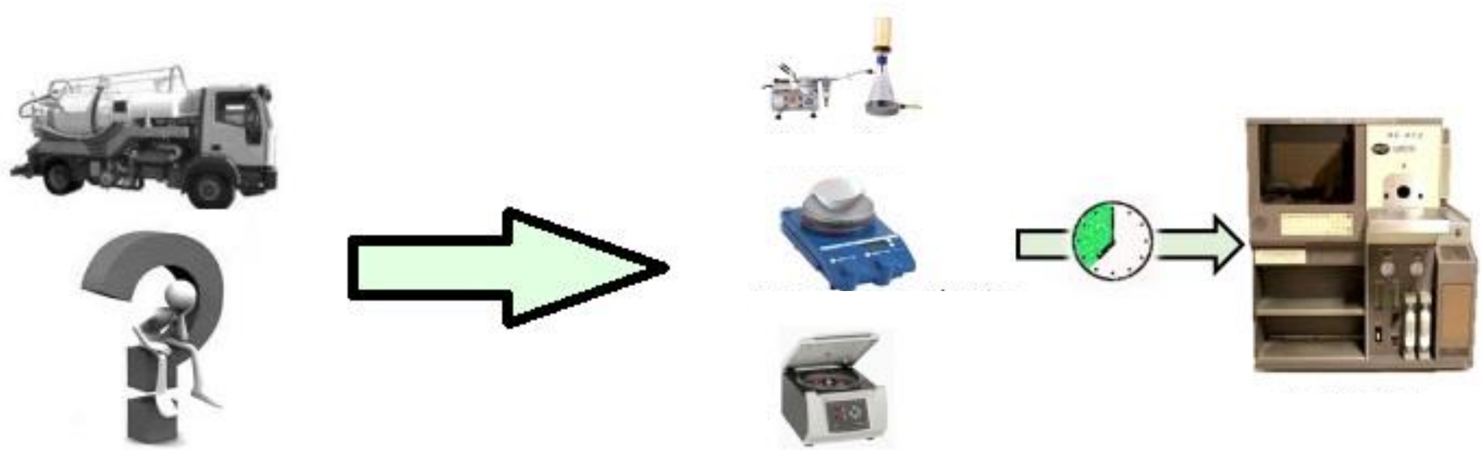
ORGANIC CARBON IN INDUSTRIAL WASTEWATERS

STUDY OF SULPHUR EMISSIONS TO RESOLVE CERAMIC DEFECTS

STUDY OF COMPOSITES FOR ADITIVE MANUFACTURING

WORKING LINE

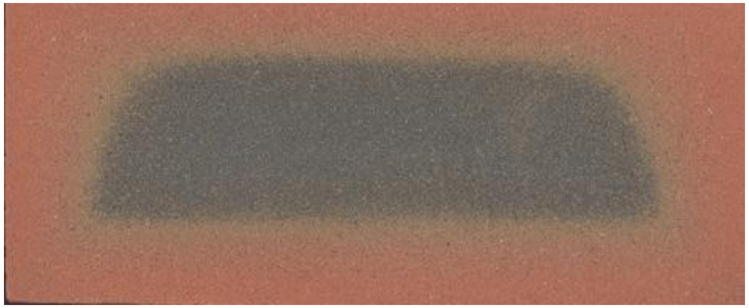
Development of a methodology to control in real time the organic carbon content in industrial wastewaters and sewages

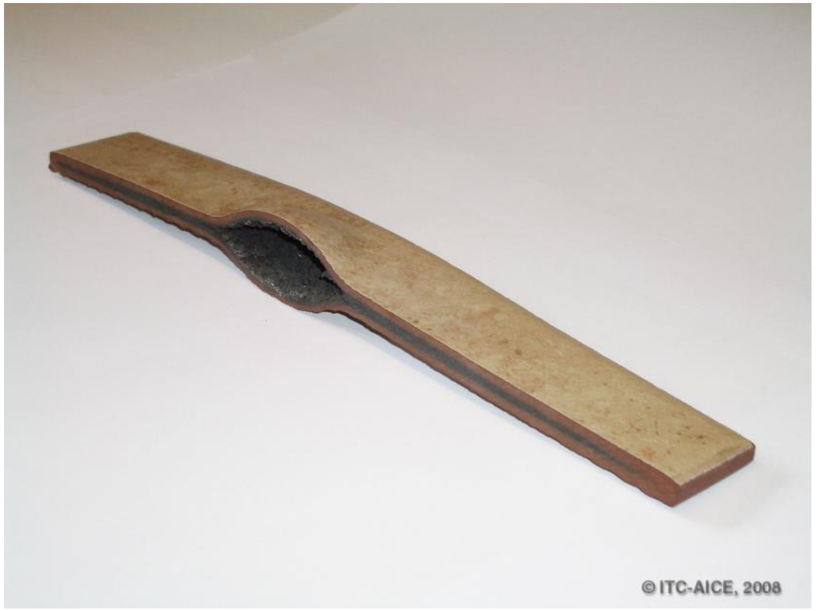


Organic carbon content

1. Problems

Black core





GAZULLA, M.F.; ORTS, M.J.; GOZALBO, A.; AMORÓS, J.L. Determinación del contenido en materia orgánica en arcillas por culombimetría. *XVII REUNION CIENTIFICA DE LA SOCIEDAD ESPAÑOLA DE ARCILLAS (SEA 2002)*. Elche y Castellon, 27 - 30 Noviembre, 2002.

2. Objective

- Find a parameter easy and fast to determine, related with the amount of organic matter present in industrial wastewaters and sewages

- Studied parameters:

    - Conductivity
    - Chemical oxygen demand (COD)

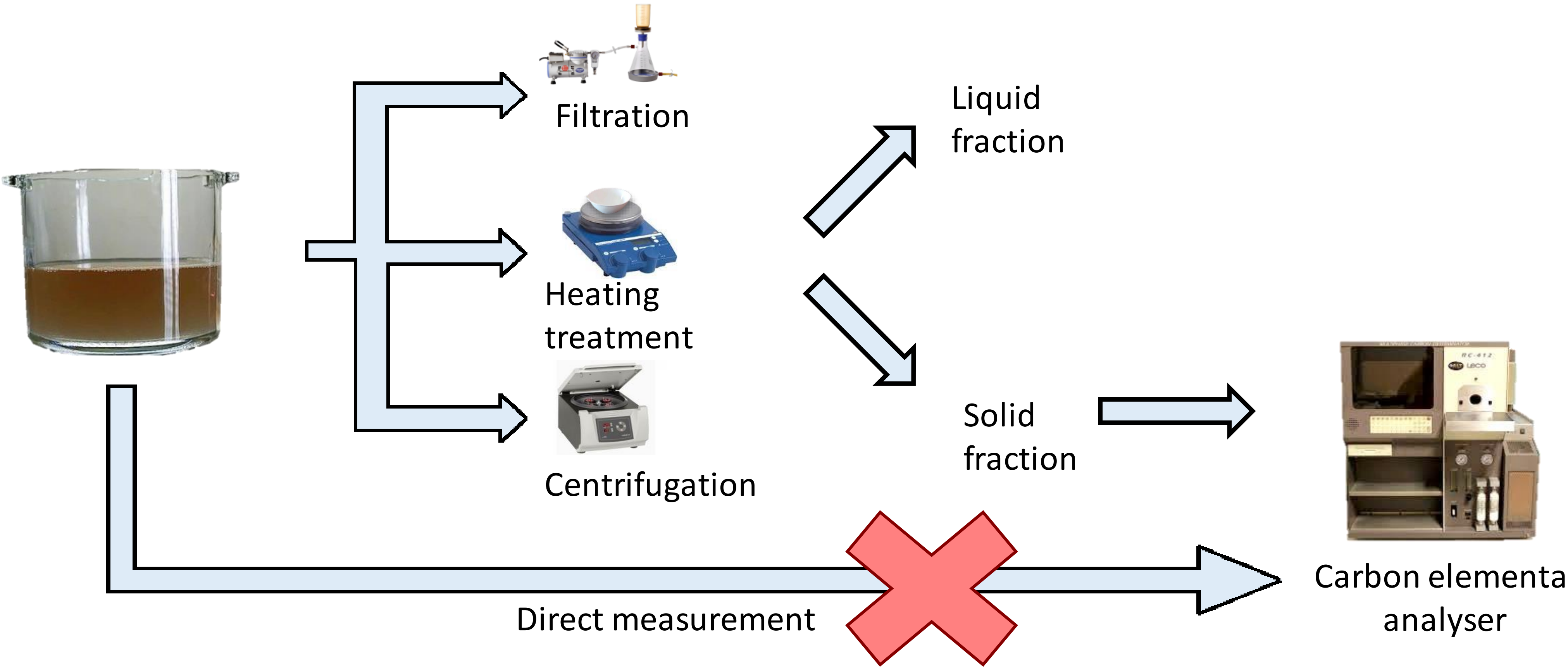
4. Experimental

Tests conducted

- Solid content
- Conductivity
- COD
- Carbon at 490ºC
- Chloride content

4. Experimental

Organic Carbon ($C_{490ºC}$)

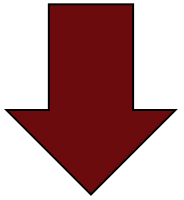


4. Experimental

How can C_{org} ($C_{490ºC}$) be fast determined?

A realtionship between the organic carbon content and COD value was found for different type of waters[1]:

$$COD = n \cdot (1{,}87 \cdot C_{org} - 17) \qquad n = 2\text{-}3$$

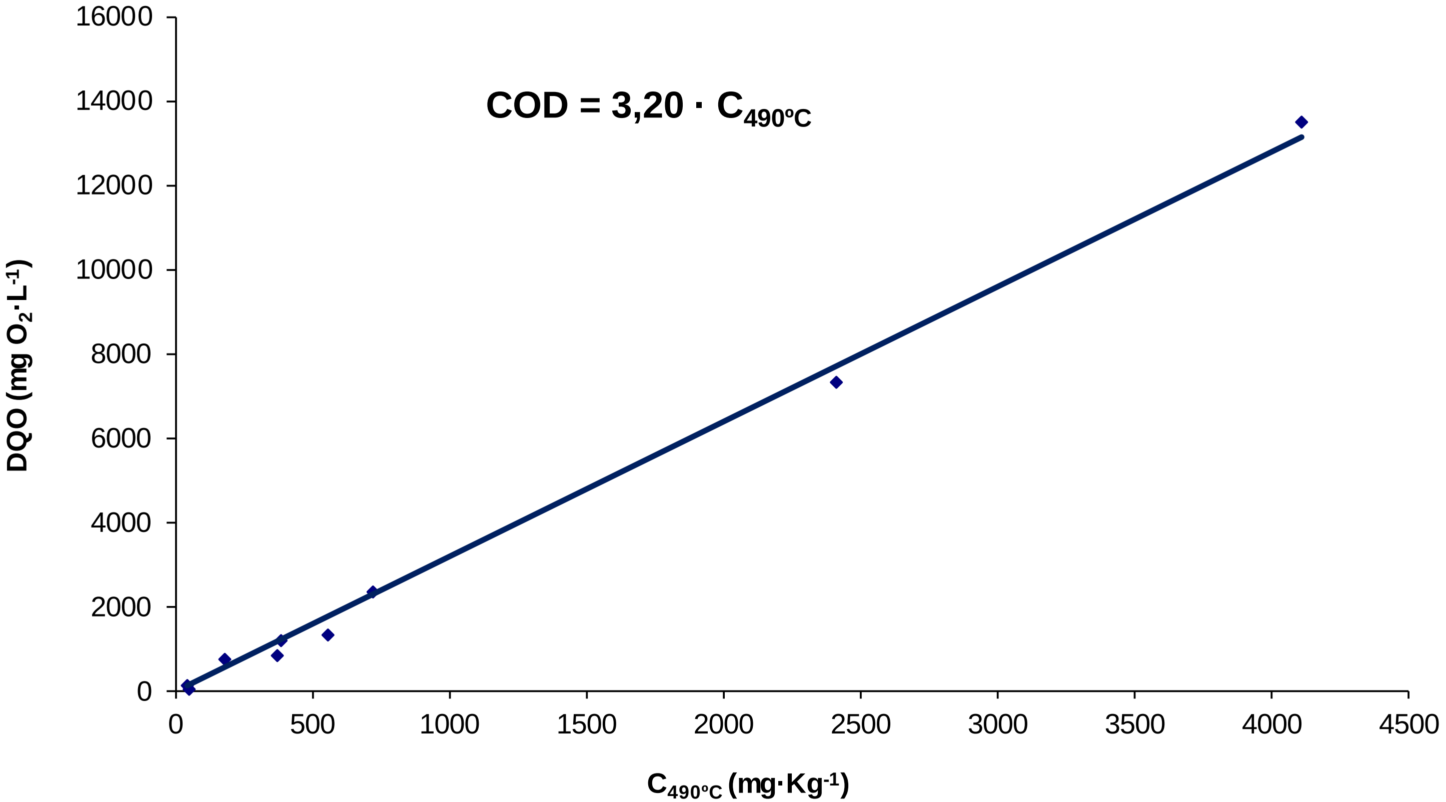


COD ????

[1] Medida de la Contaminación Orgánica.
http://cidta.usal.es/cursos/EDAR/modulos/Edar/unidades/LIBROS/logo/pdf/Medida_contaminacion_organica.pdf

5. Results

Relationship $C_{490ºC}$ - COD



$C_{490ºC}$ (mg · Kg^{-1})	COD (mg O_2 ·L^{-1})
383	1200
41	114
50	60
4110	13485
720	2368
370	829
180	742
2415	7328
560	1348

5. Results

Relationship found in the literature[1]:

$$COD = n \cdot (1{,}87 \cdot C_{org} - 17) \qquad n = 2\text{-}3$$

Relationship obtained in ITC with industrial wastewaters:

$$DQO = 3{,}20 \cdot C_{490ºC}$$

[1] Medida de la Contaminación Orgánica. http://cidta.usal.es/cursos/EDAR/modulos/Edar/unidades/LIBROS/logo/pdf/Medida_contaminacion_organica.pdf

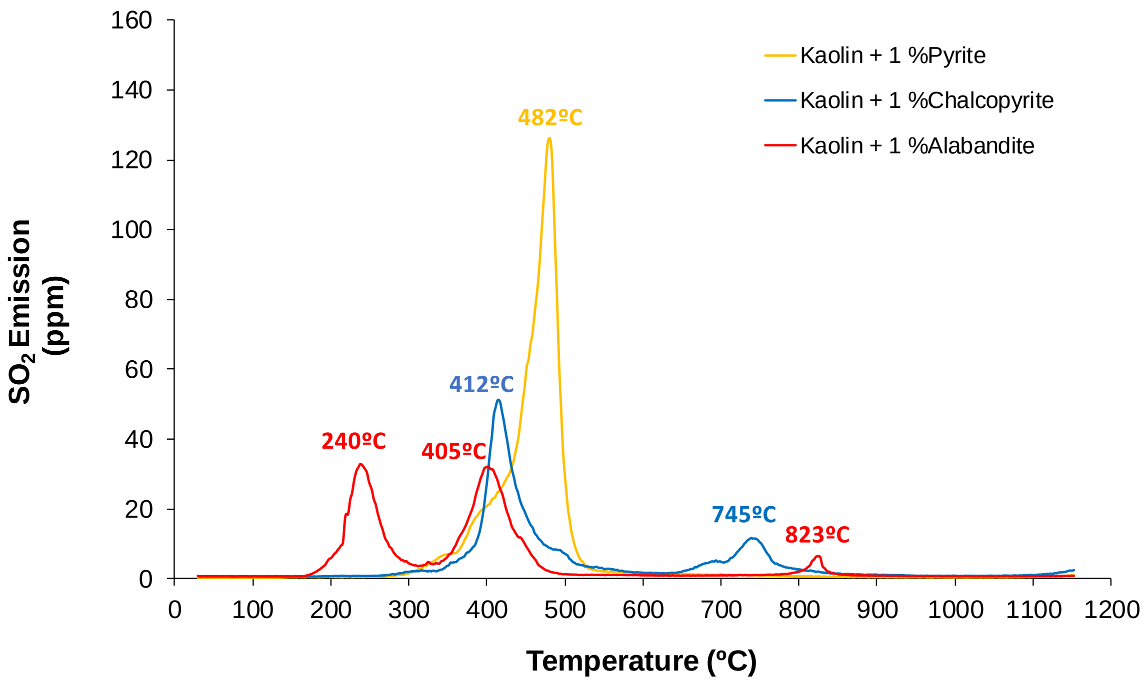
6. Conclusions

1. There is a direct relationship between COD value and the organic matter, therefore the determination of COD can be used as real-time control method for the determination of organic carbon in industrial wastewaters.
2. The time of the assay has been reduced to 30 minutes of digestión plus the time necessary for the titration.
3. There is no need to use neither Ag_2SO_4 nor $HgSO_4$ in COD determination in residual wastewaters and sewages.
4. There is not a direct relationship between the conductivity and the organic carbon.

STUDY OF SULPHUR EMISSIONS TO RESOLVE CERAMIC DEFECTS

OBJECTIVE

- Study of the kinetics of oxidation and decomposition of sulphides in raw materials for the improvement of production processes associated to ceramic defects (**Evolved Gas Analysis Technique**).



PYRITE

CHALCOPYRITE

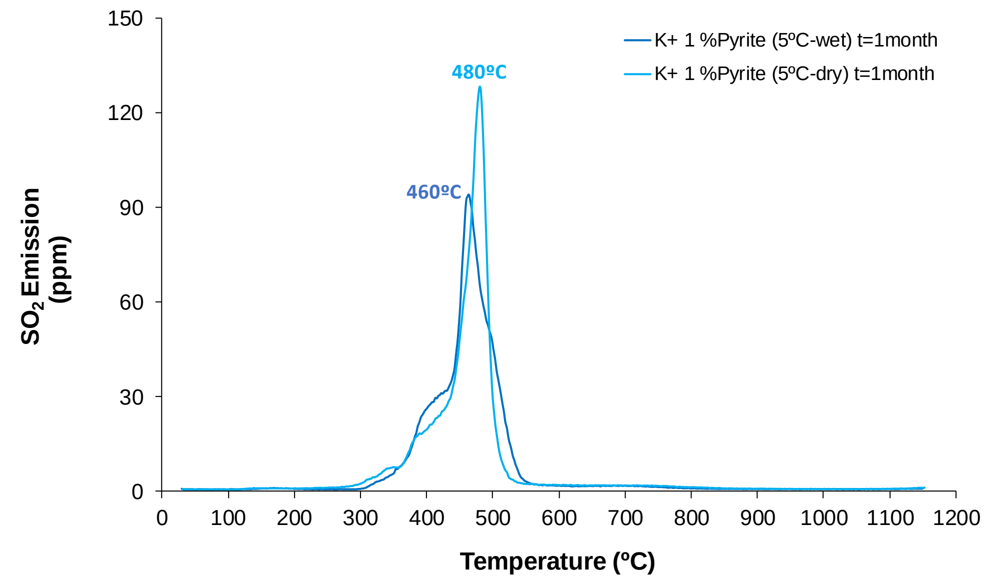
ALABANDITE

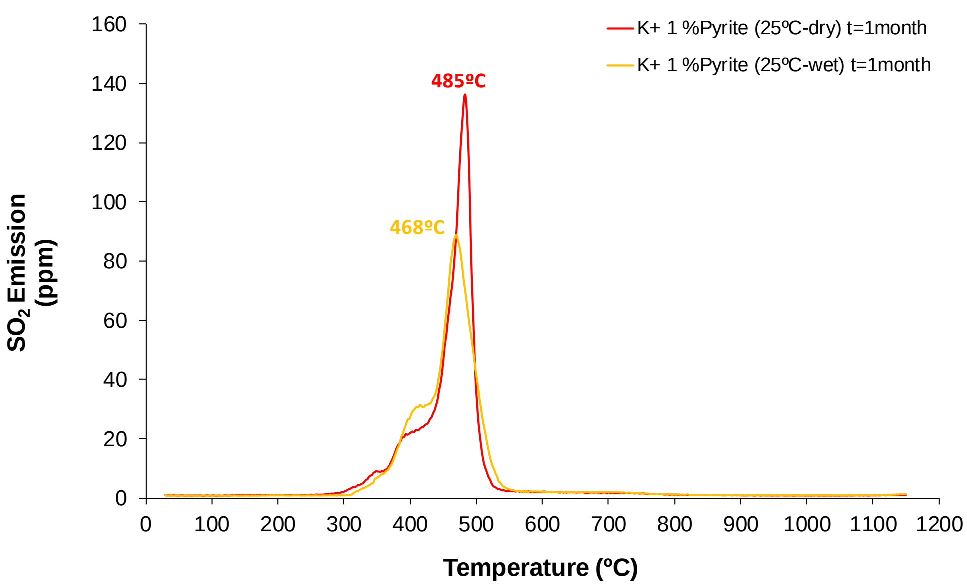
Temperature (25ºC)	•Dry environment •Wet environment
Temperature (45ºC)	•Dry environment •Wet environment
Temperature (5ºC)	•Dry environment •Wet environment

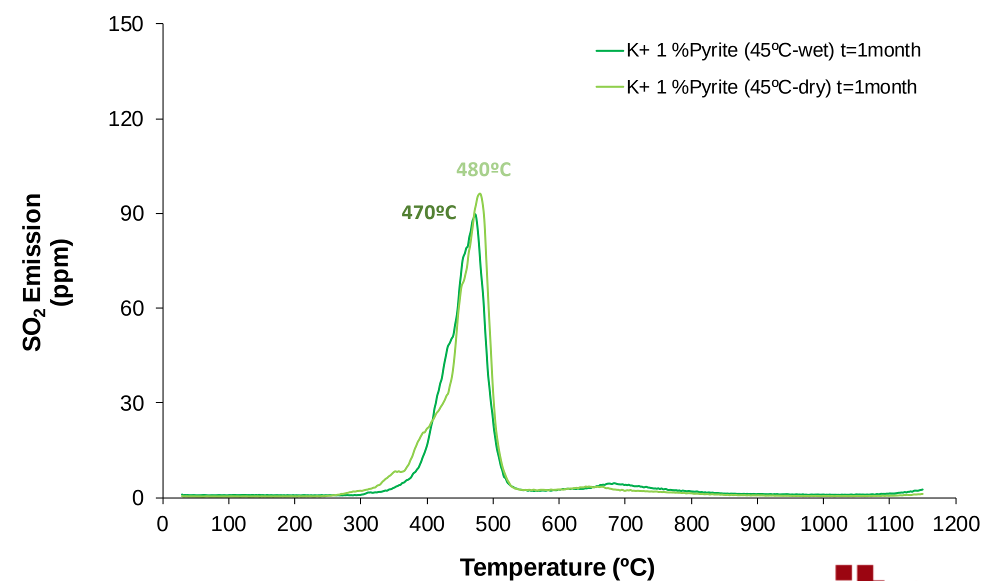
STUDY OF SULPHUR EMISSIONS TO RESOLVE CERAMIC DEFECTS

RESULTS: 1 MONTH PYRITE

$$FeS_2 + \frac{7}{2}O_2 + H_2O \rightarrow Fe^{2+} + 2SO_4^{2-} + 2H^+$$

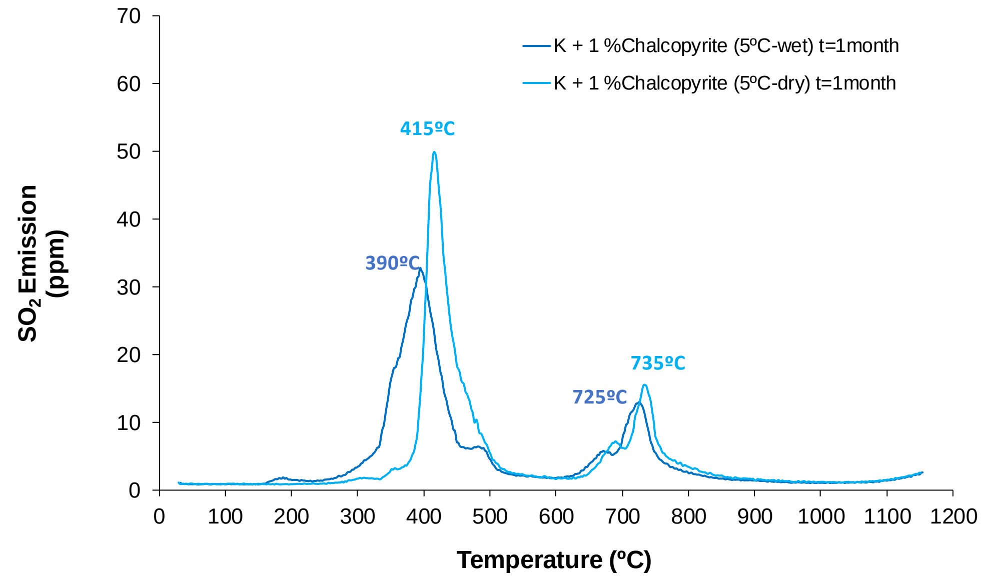


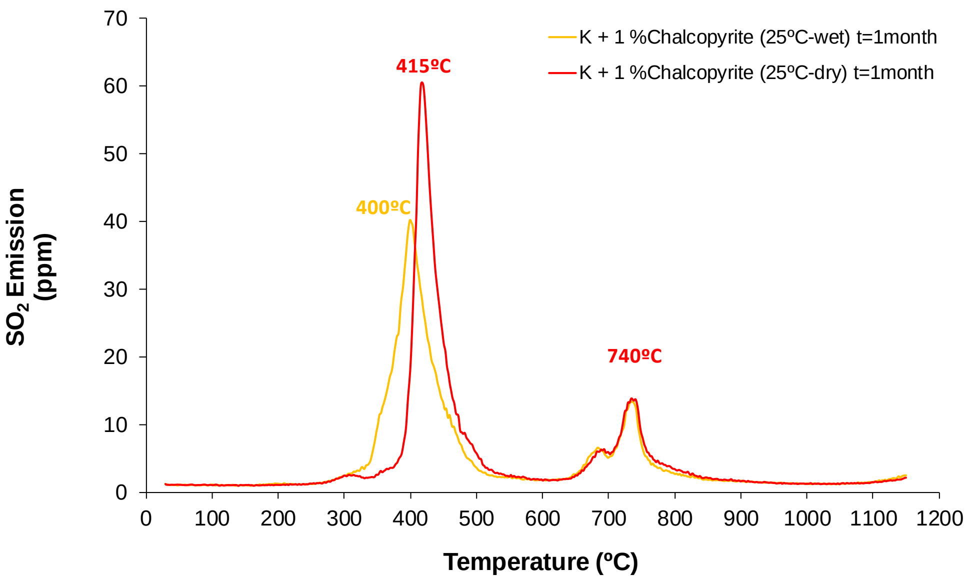


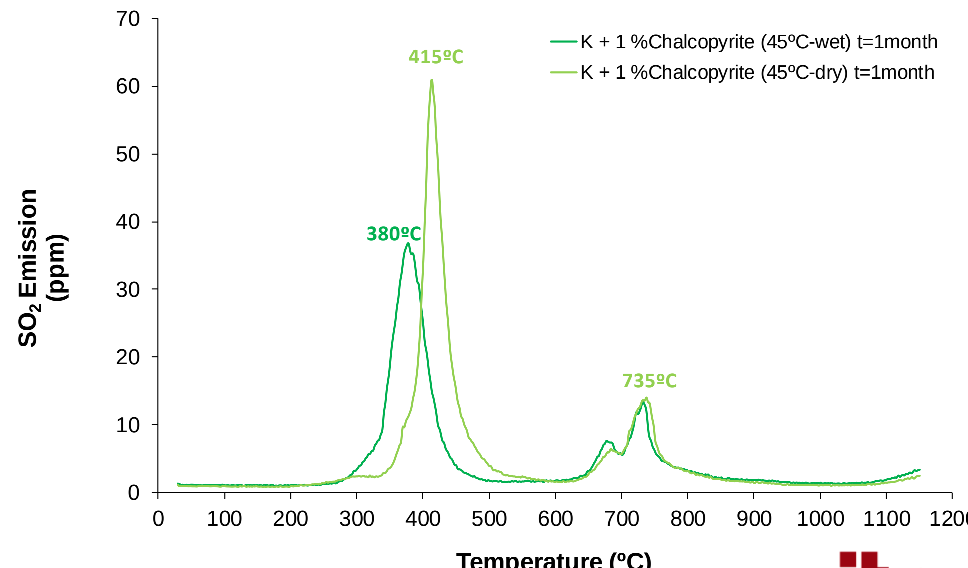


RESULTS: 1 MONTH CHALCOPYRITE

$$CuFeS_2 + 4O_2 \rightarrow CuSO_4 + FeSO_4$$

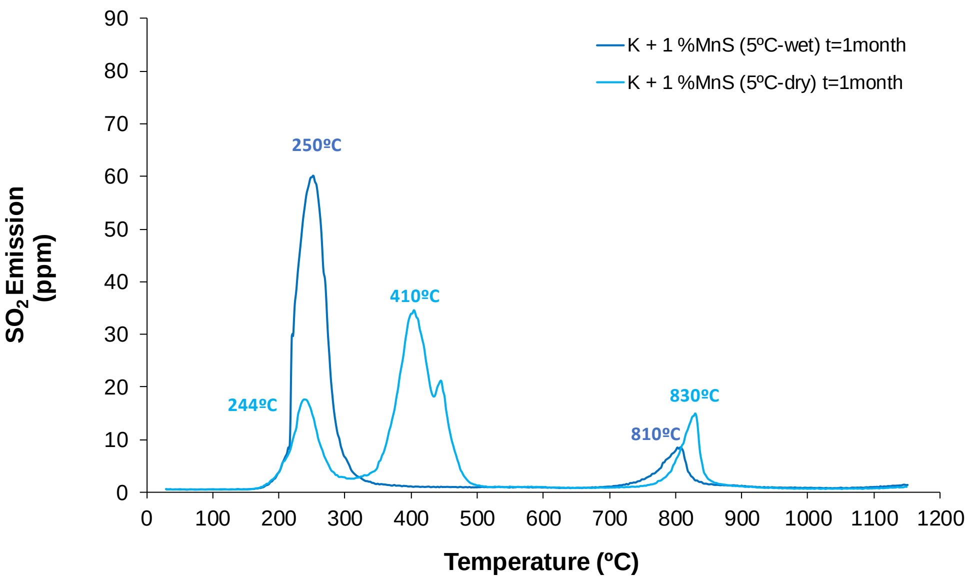


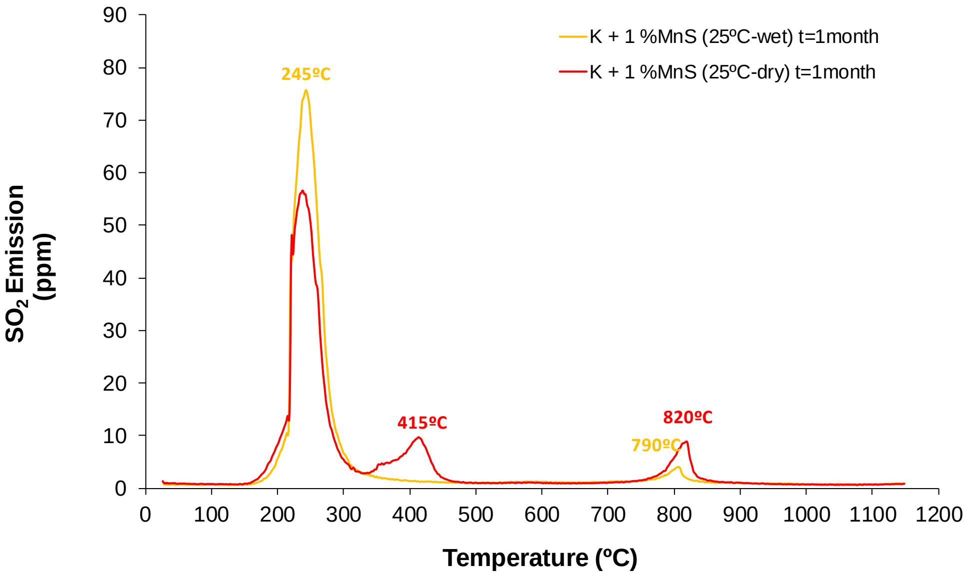


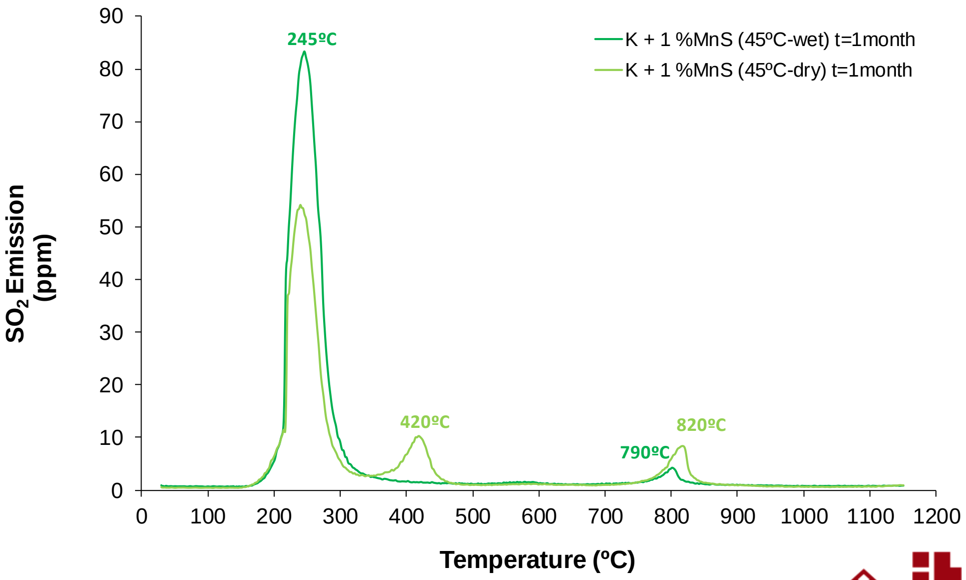


STUDY OF SULPHUR EMISSIONS TO RESOLVE CERAMIC DEFECTS

RESULTS: 1 MONTH ALABANDITE

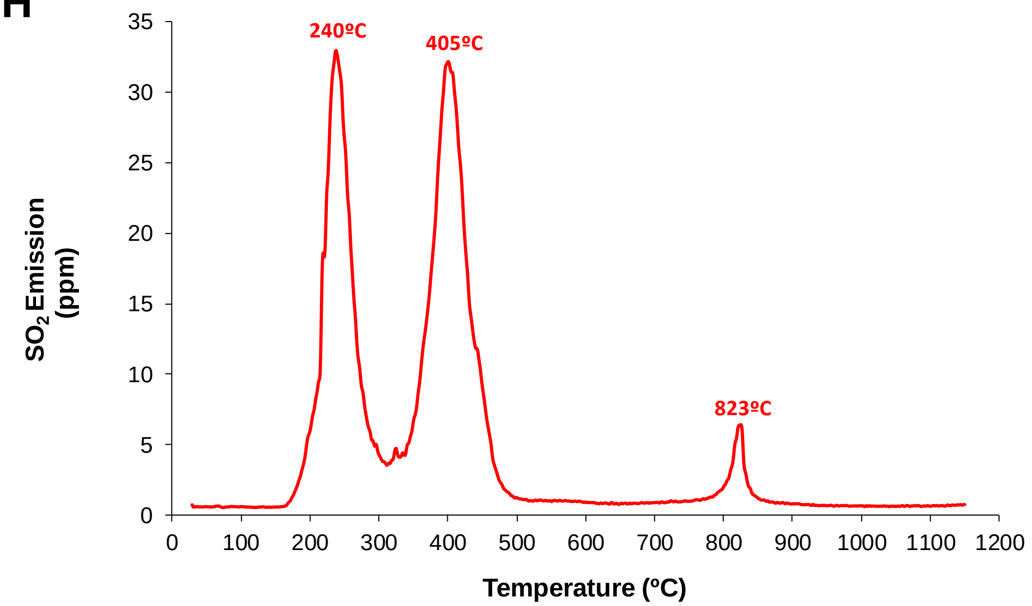






STUDY OF SULPHUR EMISSIONS TO RESOLVE CERAMIC DEFECTS

RESULTS: 1 MONTH ALABANDITE



As is corroborated by thermal analysis:

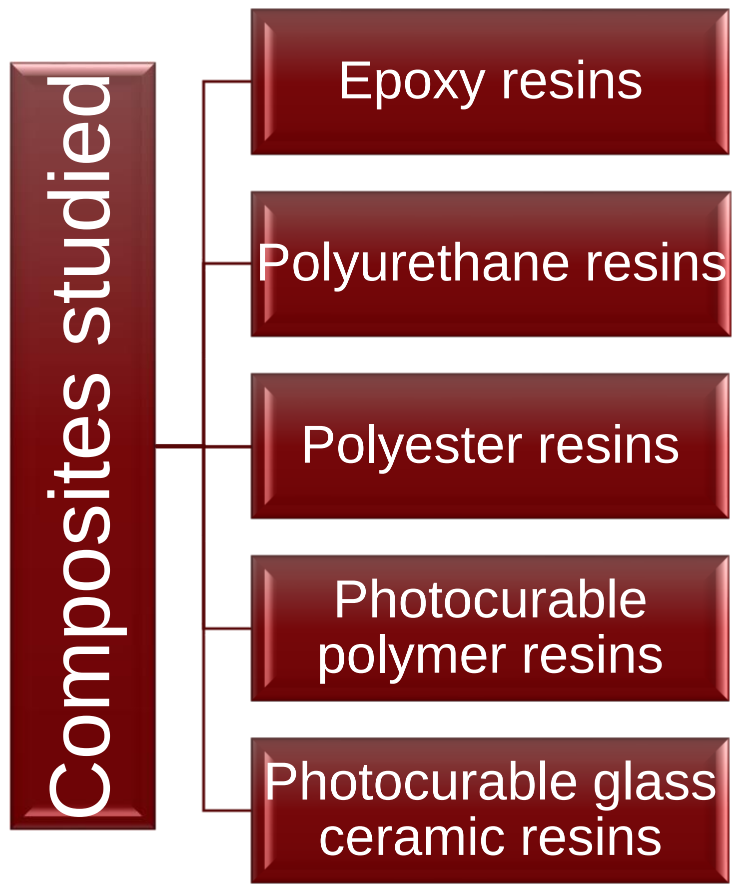
- First reaction in alabandine is explained by the oxidation of MnS partly into MnO and SO_2.
- Second exothermic reaction represents oxidation of MnO (or partly MnS) into Mn_2O_3 and partial formation of $MnSO_4$.
- Third reaction is caused by the transformation of Mn_2O_3 into Mn_3O_4 and decomposition of $MnSO_4$.

CONCLUSIONS

1. Significant differences have been observed in the decomposition of sulphides stored in humid environments for a month, causing changes in the emission of SO_2. This fact is not observed in dry environment.
2. The EGA technique is suitable for the study of impurities of sulphurous nature. It allows study of decompositions and oxidations with temperature with a very low detection limit.
3. In this work, the technique makes it possible to distinguish sulphurous impurities presents in a raw material: pyrite, chalcopyrite and alabandite.

OBJECTIVE

- Obtaining a characterization method to efficiently control and manage the use of composites in 3D printing.



STUDY OF COMPOSITES FOR ADDITIVE MANUFACTURING

Characterization techniques

- IR spectroscopy
- Elemental Analysis (C,H,N,S)
- DSC (Differental Scanning Calorimetry)
- TGA (Thermogavimetric Analysis)

EXPERIMENTAL

Selection of composites

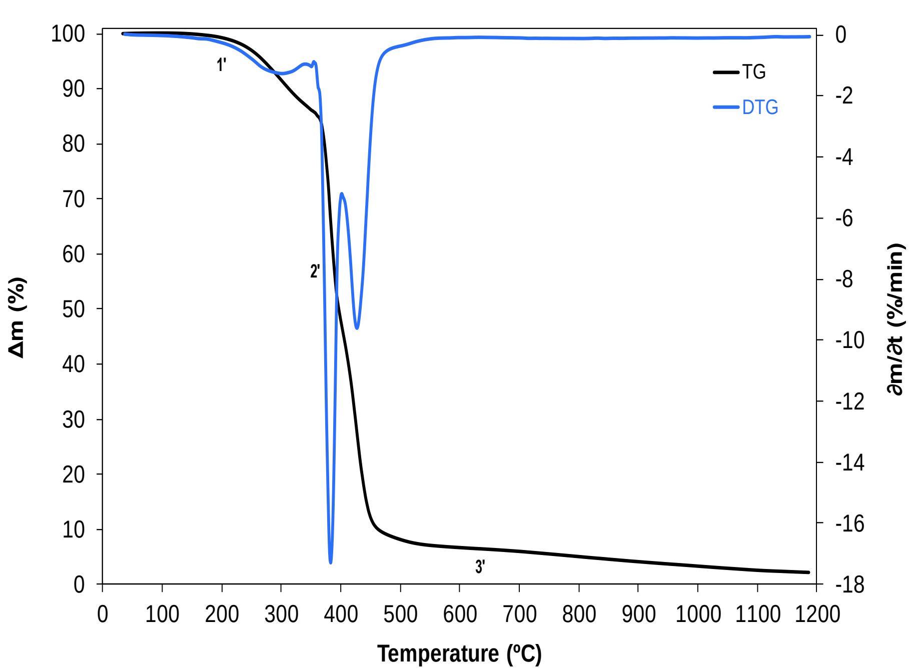
Characterization of composites

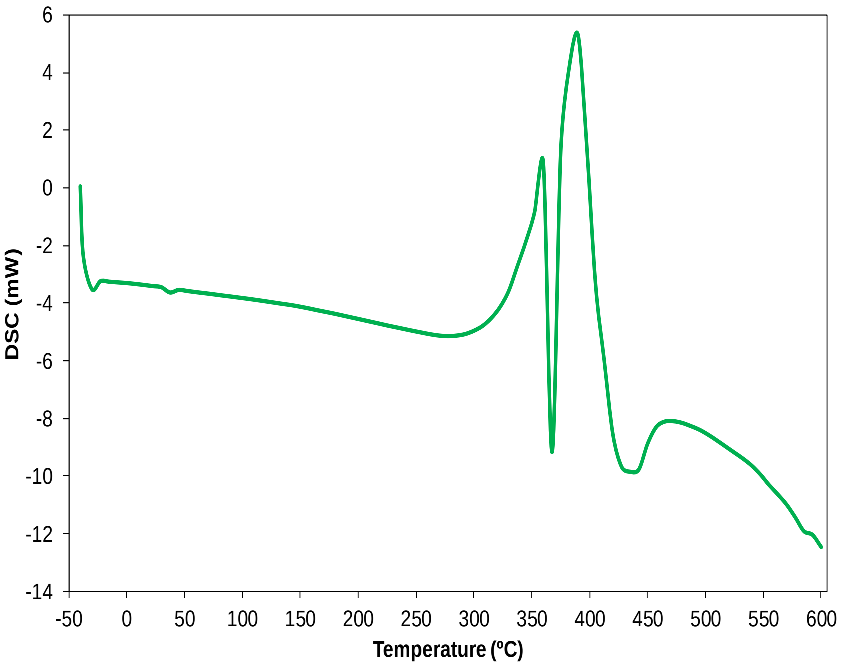
- IR spectroscopy, Elemental Analysis, DSC, TGA

Study of composites curing process

- Study of the behavior of composites during the curing process in function of time and temperature

RESULTS: CHARACTERIZATION OF AN EPOXY RESIN

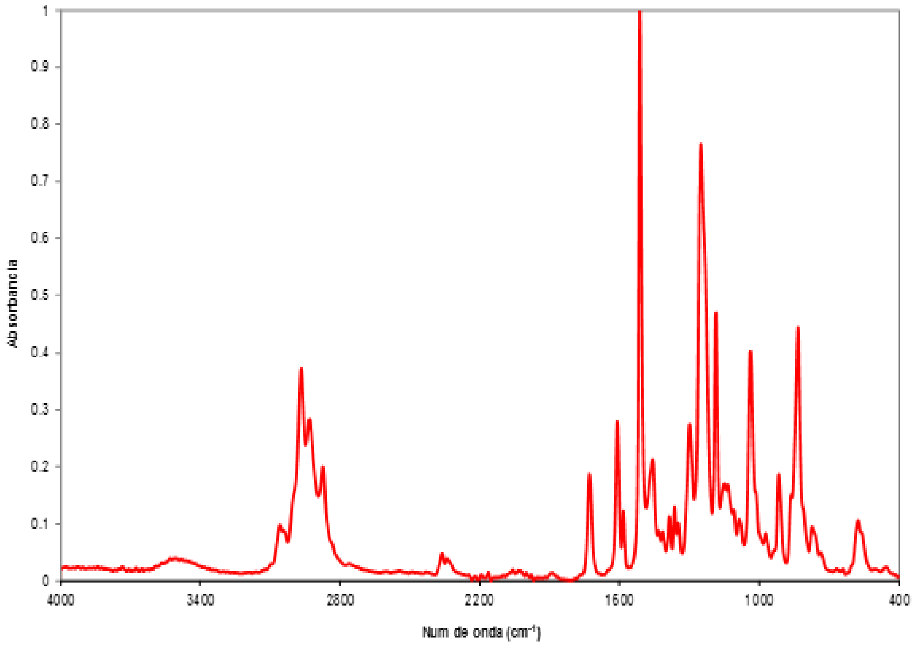


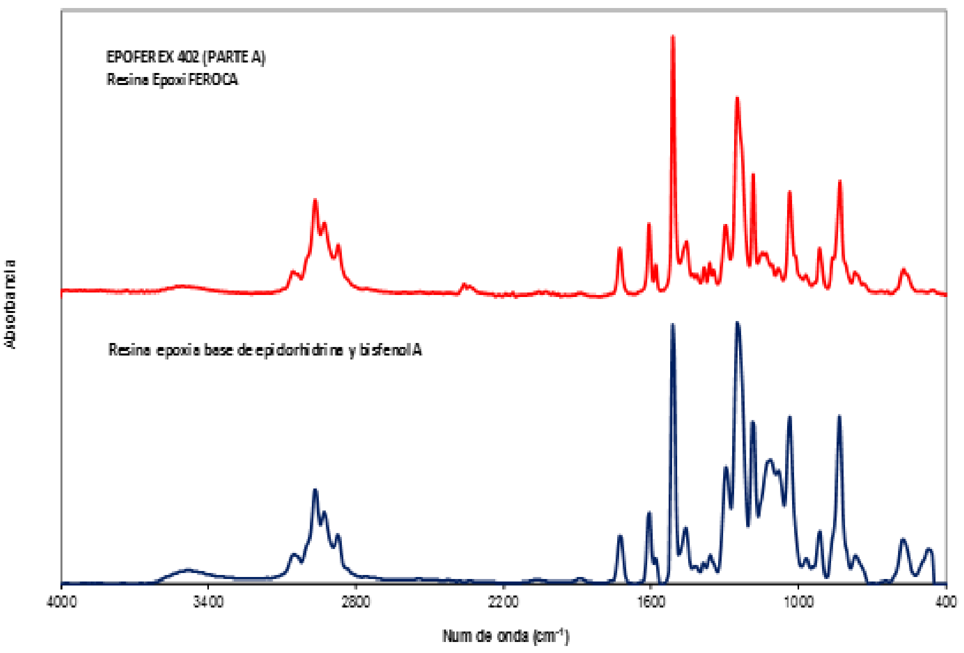


TGA ➡ Information about the compositional analysis of the material and its stability.

DSC ➡ Information about "Glass Transition" (Tg) and the curing process.

RESULTS: CHARACTERIZATION OF AN EPOXY RESIN

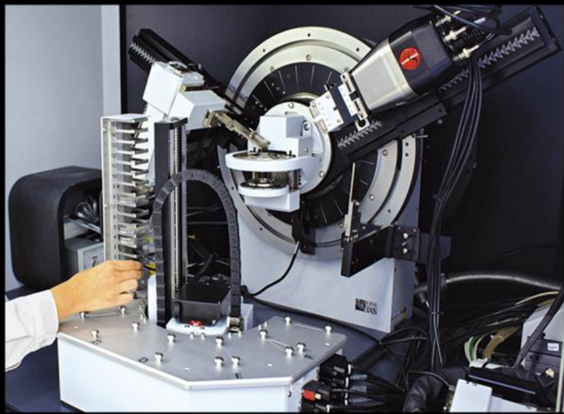




IR ➡ Quantitative and qualitative information about composition of the material ➡ Epichlorohydrin + Bisphenol A

CONCLUSIONS

1. TGA analysis provide information about compositional analysis and thermal and oxidative stabilities of studied materials.
2. DSC provides information about “Glass Transition” (Tg), onset and completion of cure, maximum rate of cure and percent of cure.
3. IR spectroscopy provides qualitative and quantitative information about the composition of the materials and allows the monitoring of curing reaction.
4. All of these characterization techniques will allow us to optimize the composites curing process, and, in turn, manage them in a more efficient way.




Thank you for your attention

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