Consiglio Nazionale delle Ricerche

Istituto per l'Energetica e le Interfasi

Characterization of silica and titania nanoparticles synthesized in a spray flame reactor

F. Cignoli, S. Maffi, C. Bellomunno, S. De Iuliis, G. Zizak

CNR-IENI, U.O. Milano, via Cozzi 53, 20125 Milano, Italy

zizak@ieni.cnr.it

Introduction (1)

Nanostructured materials represent nowadays a wide, and probably still largely unexplored, field of potential applications. In fact, this is a research topic in high and rapid development, both at a basic level and under the point of view of possible practical applications, leaving large space for a thorough scientific analysis, which requires with no doubt long time for ultimate conclusions.

This paper deals with the preliminary work performed in the field of FSP (Flame Spray Pyrolysis) synthesis for nanoparticles, using an external mixing gas assisted nozzle. Particularly, an experimental apparatus has been designed, realized and characterized for the synthesis of nanoparticles by the flame spray pyrolysis method.

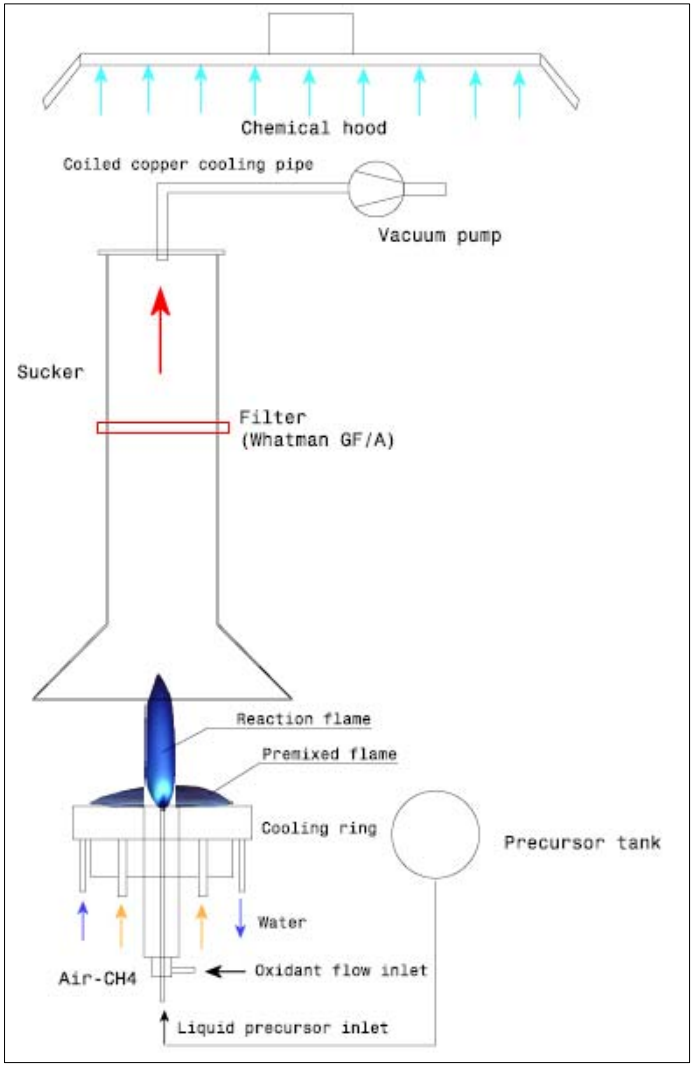
Introduction (2)

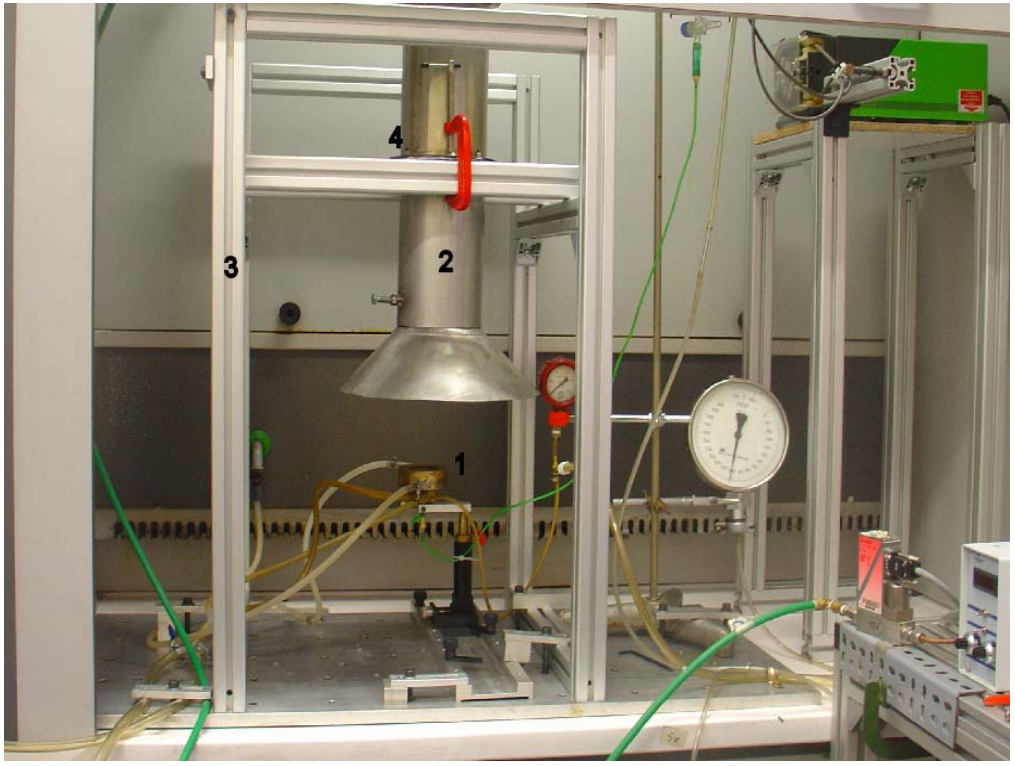
The advantage of the Flame Spray Pyrolysis technique (FSP) is the use of a wide variety of possible low cost precursors (mainly in the field of metal oxides such as TiO2, Al2O3), obtaining a final product with high purity and relatively narrow size distribution.

A typical experimental set-up is constituted by a unit for droplets generation and dispersion (usually a gas-assisted spray), a heat source for droplets evaporation and ignition and an oxidant for combustion.

Due to the complexity of the physical and chemical phenomena involved in the controlled synthesis by FSP, investigation has to be performed about the influence of the operating conditions of the spray (flow field, dimensional distribution, precursor typology and physical properties, gas to liquid mass ratio, oxidant typology) on the final product, that is on the morphological and structural properties of the derived nanoparticles.

Experimental set-up (1)





Experimental set-up (2)

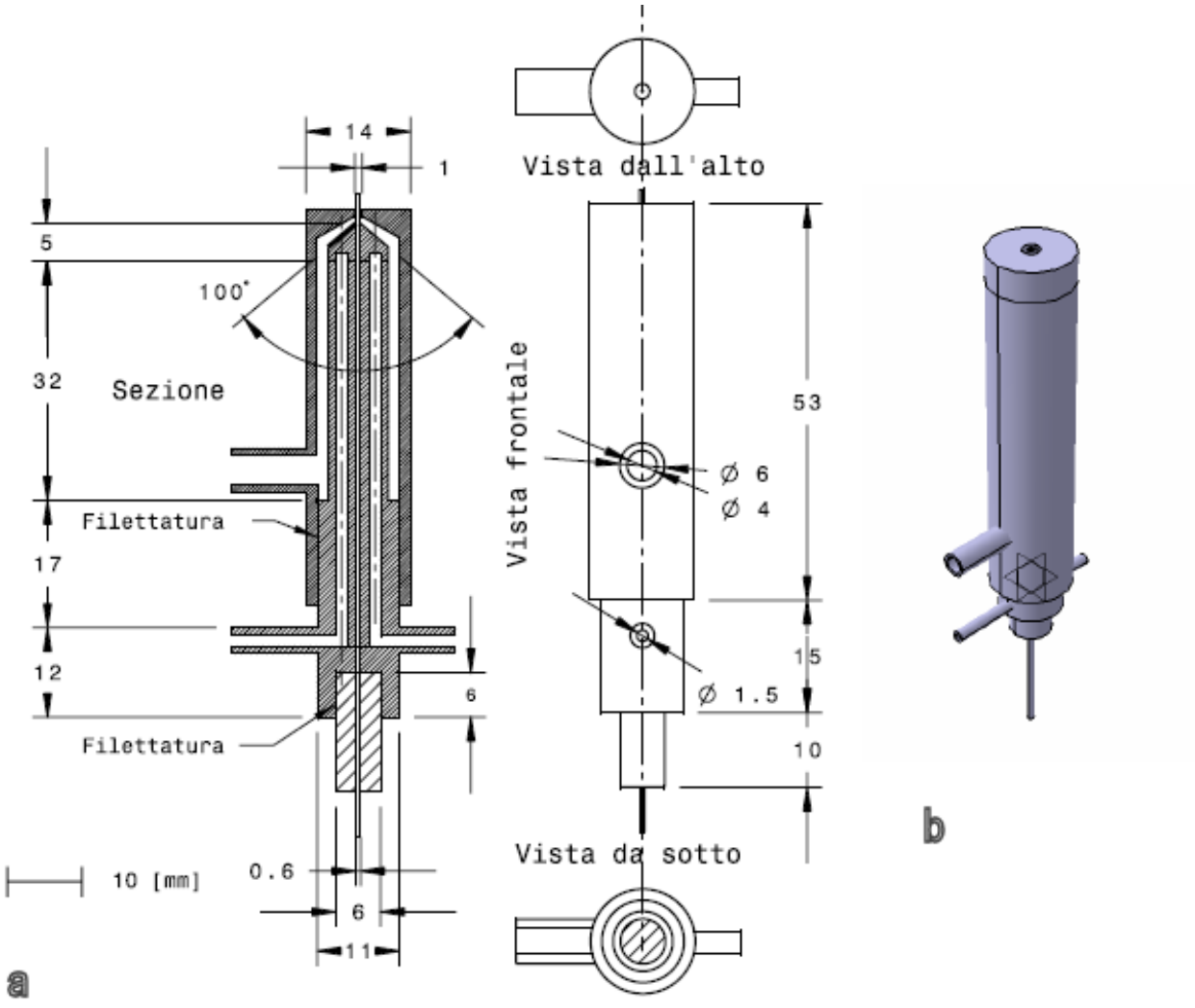
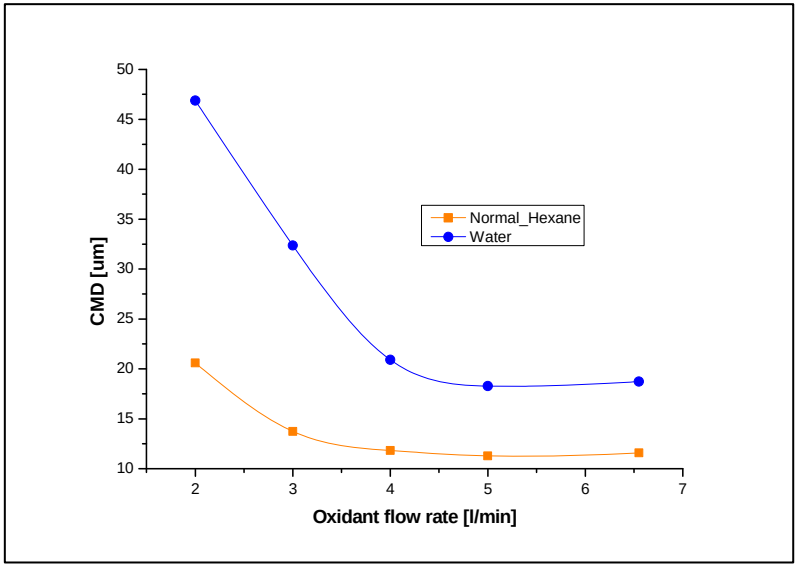
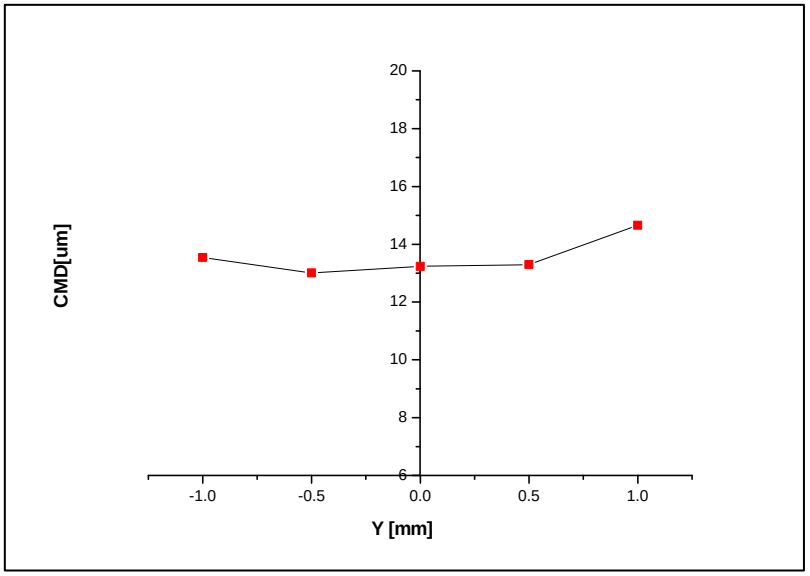


Image of the water spray

•PDA (Phase Doppler Anemometry) and visualizations to investigate the flow field and the dimensional distribution of the droplets generated by the atomizer;



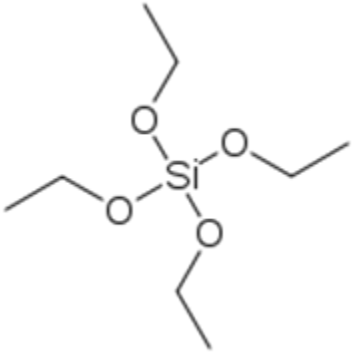
Mean droplets diameter for water and n-hexane spray at 5 mm from the exit nozzle



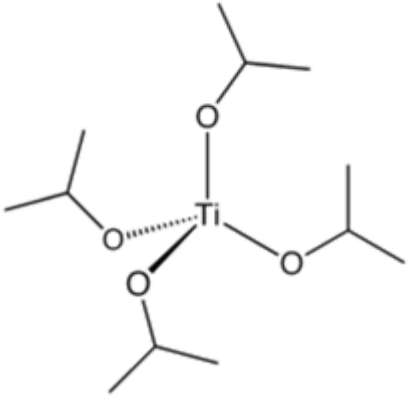
Radial profile of mean droplets diameter

Selection of precursor and dispersion fuel for nanoparticles synthesis through FSP

- Tetraethoxysilane (TEOS), $Si(OC_2H_5)_4$, in n-hexane for SiO_2 synthesis;



- Titanium tetraisopropoxide (TTIP), $Ti\{OCH(CH_3)_2\}_4$), in ethanol for TiO_2 synthesis;



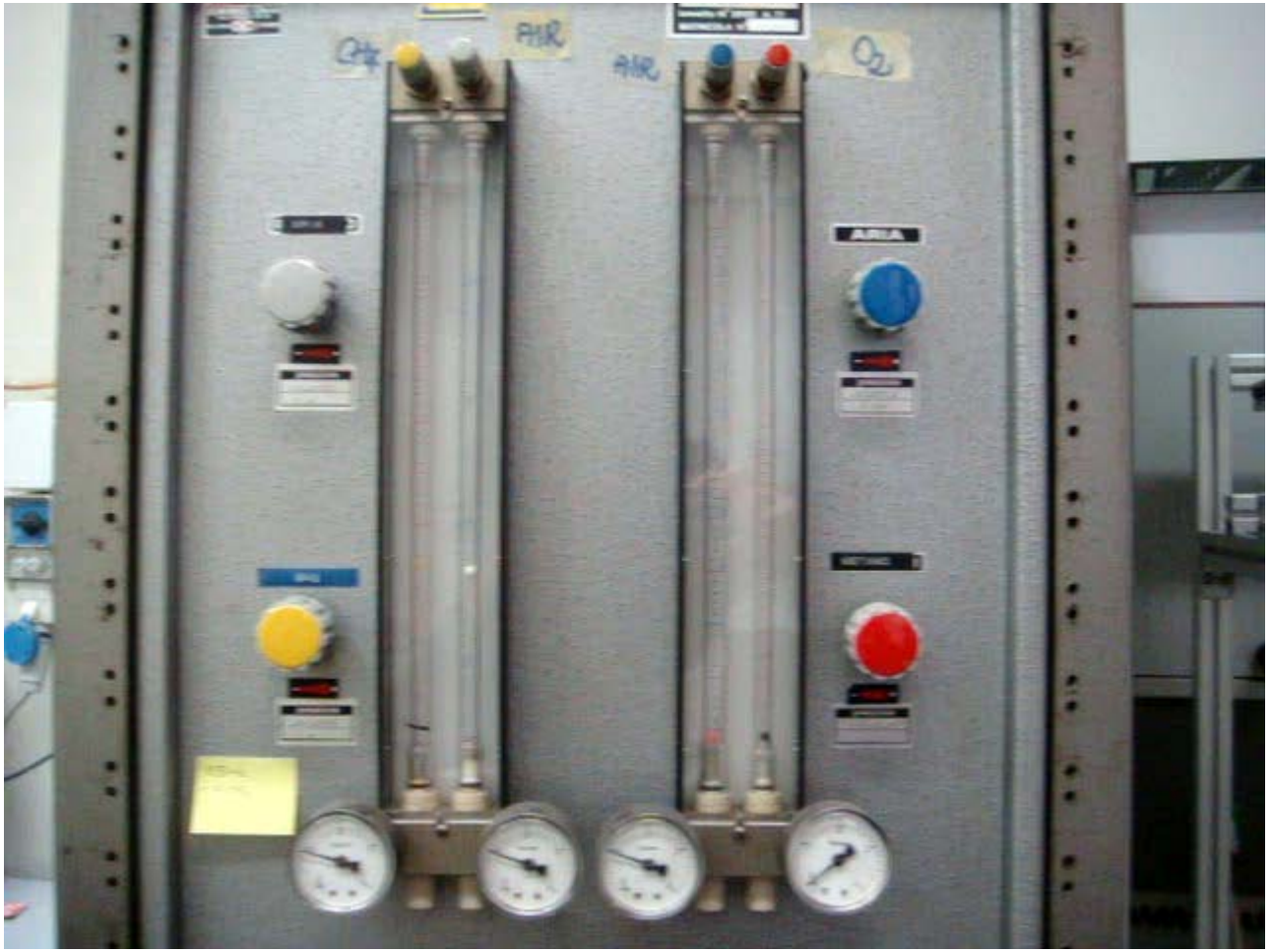
In both cases: oxygen as dispersion-oxydation gas.

Flame visualization

n-hexane spray flame

n-hexane with
TEOS spray flame

ethanol with TTIP
precursor spray flame

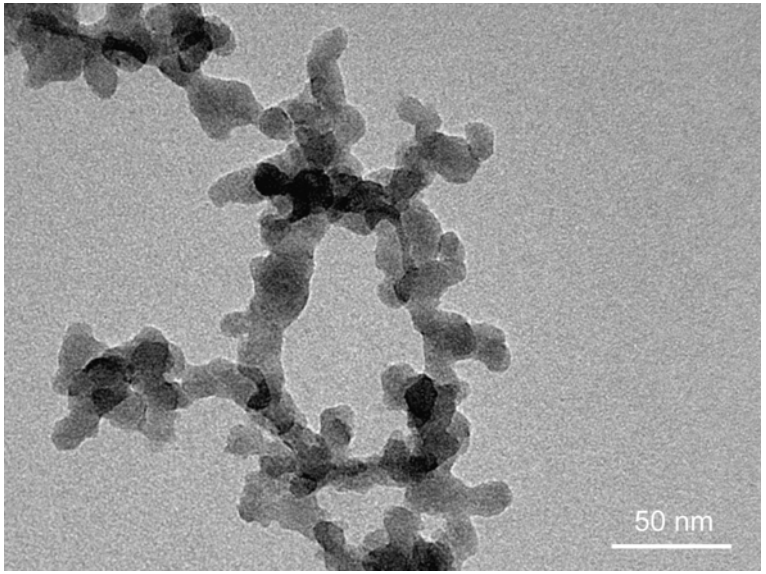

AIR
AIR
O2
ARIA

Collecting system

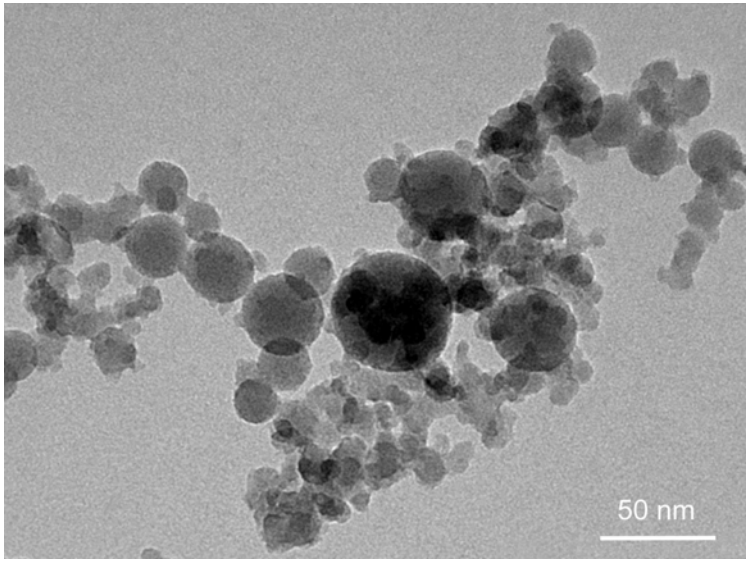
Test-tube for powder analysis

TEM (Transmission Electron Microscopy) for dimensional analysis of nanoparticles

SiO_2 nanoparticles synthesized in the spray flame



0.5 mol., 1 ml/min

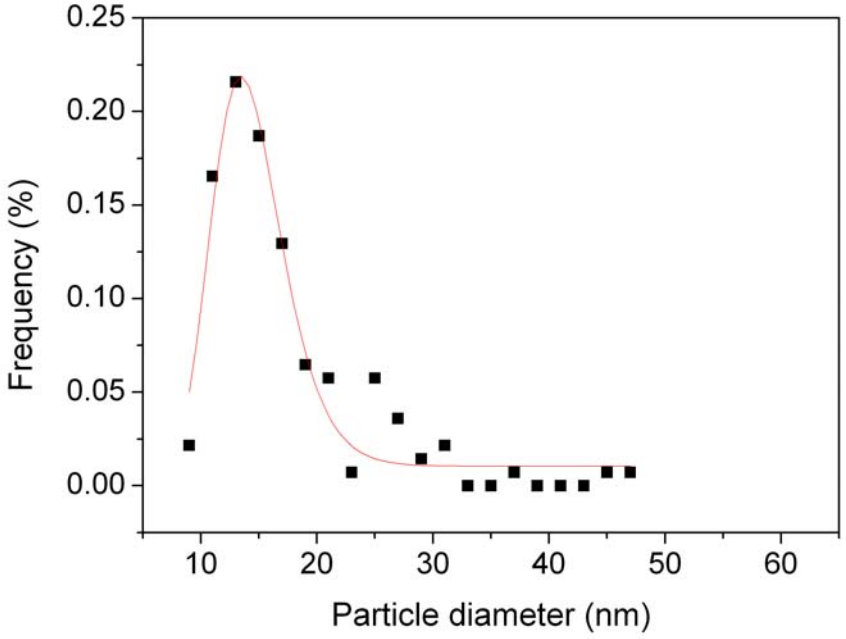


1 mol.,3 ml/min

Lognormal distributions

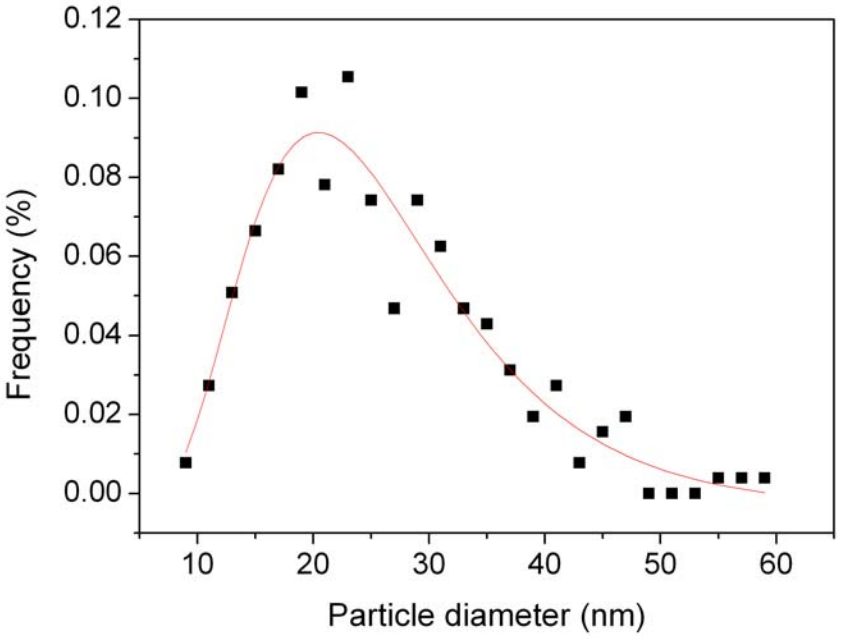
SiO_2

0.5 mol., 1 ml/min



d_0 = 14.11 nm - σ = 1.25

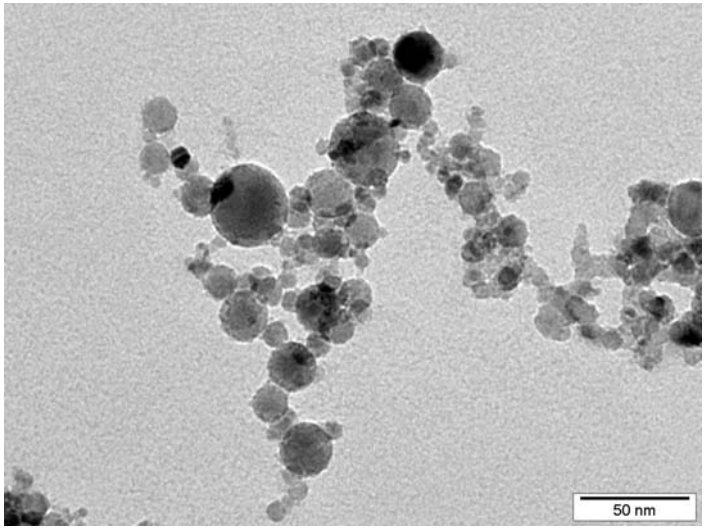
1 mol., 3 ml/min

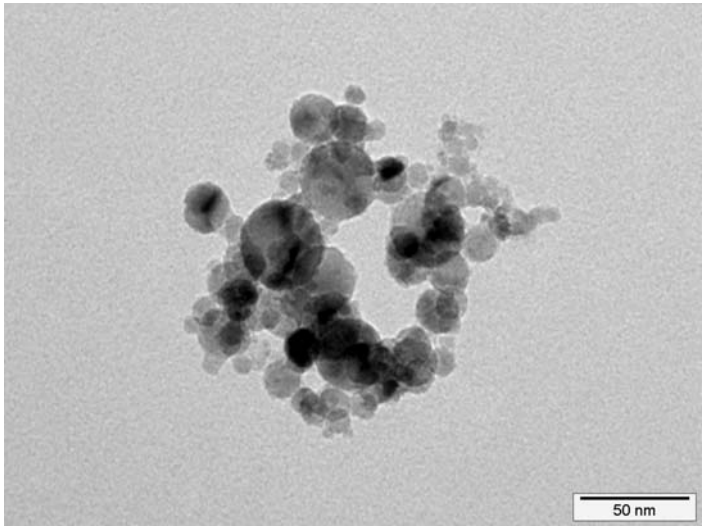


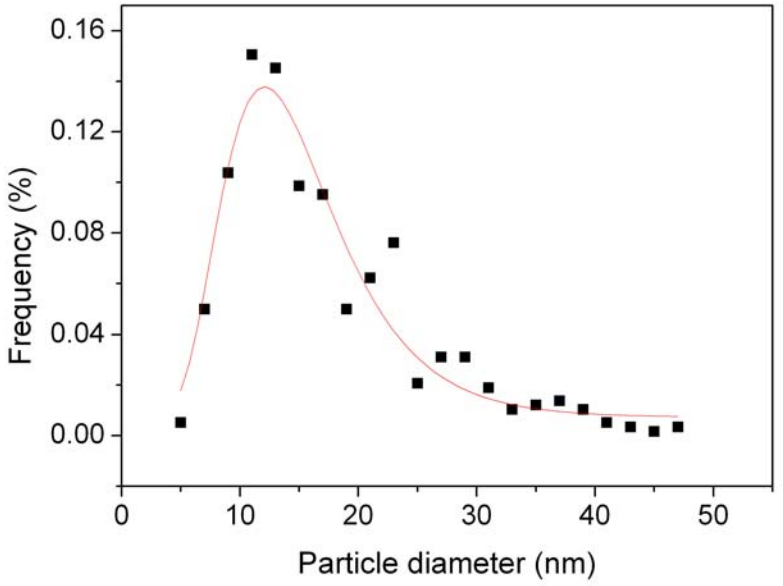
d_0 = 24.39 nm - σ = 1.52

TiO_2 nanoparticles synthesized in the spray flame

(TTIP, 1 mol.)







d_o = 14.09 nm – σ = 1.48

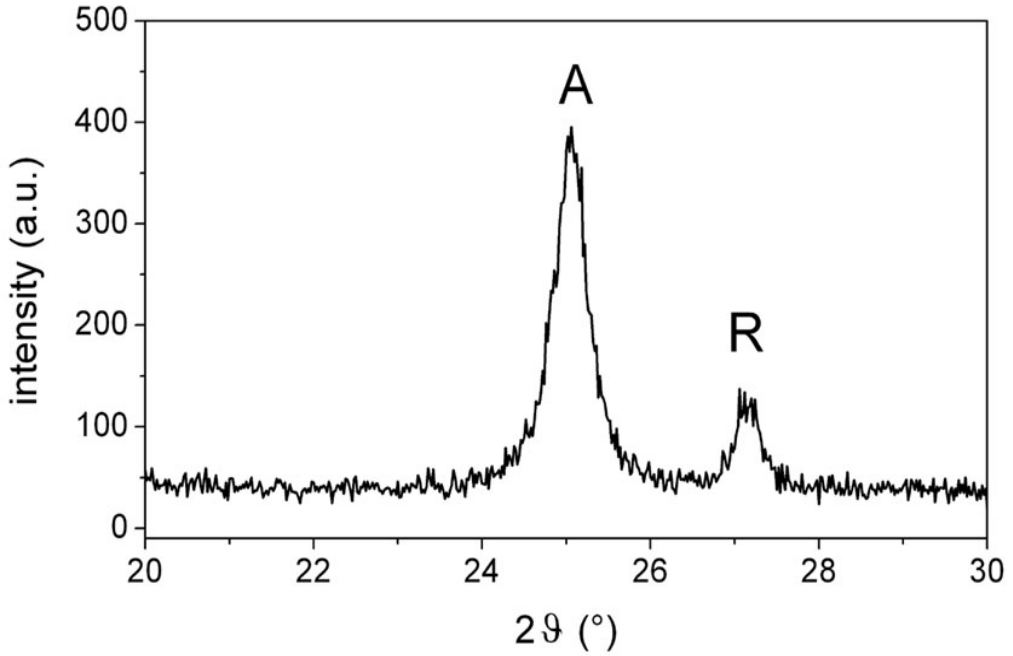
<d> = 17.12 nm

XRD (X Ray Diffraction) for investigation of cristalline structure of nanoparticles

TiO_2 analysis

$$\%\ R = \frac{1}{\left(\frac{A}{R}\right) \times 0.884 + 1} \times 100$$

A = Anatase; R = Rutile



The anatase phase is largely prevailing in the collected powder (86,3% anatase and 13.7% rutile).
From the structure of the peaks it is also possible to derive an estimate of the average crystallite size, that in this case results to be between 15 and 20 nm.

Conclusions

An experimental apparatus has been designed, realized and characterized for the synthesis of nanoparticles by the flame spray pyrolysis method. The apparatus consists of a gas-assisted spray for droplet generation and dispersion in a secondary pilot flame. By dissolving suitable precursors in a liquid fuel, different types of nanoparticles have been produced.

In the preliminary tests SiO_2 and TiO_2 have been synthesized and characterized by TEM analysis and XRD, respectively. The apparatus shows good stability and reproducibility of the reaction flame and, therefore, of the material produced.

Further tests will be devoted to the improvement of the collection system and to the synthesis of other types of nanoparticles.

Thanks for your attention!