Design and Characterization of an Experimental Apparatus for Nanoparticles Synthesis

G. Zizak°, F. Cignoli°, R. Dondè°, S. Maffi°, G. Solero*

° CNR-IENI, Istituto per l'Energetica e le Interfasi - Milano

*Dipartimento di Energia – Politecnico di Milano

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Introduction

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.

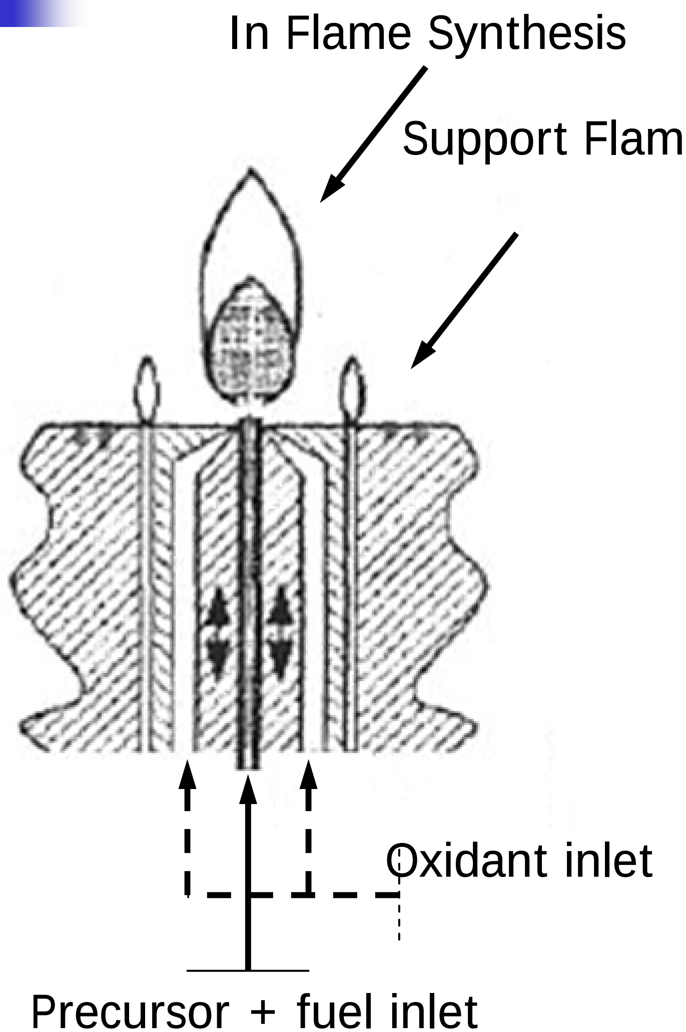
Introduction

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 TiO_2, Al_2O_3), 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.

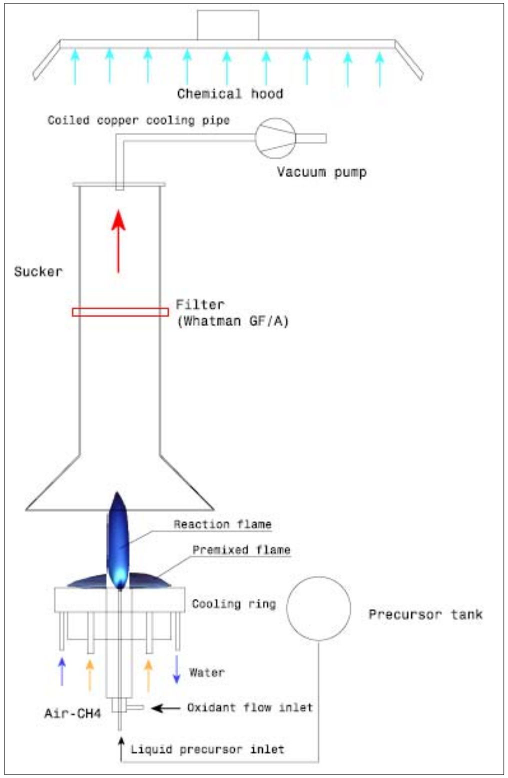
Due to the complexity of the physical and chemical phenomena involved in the controlled synthesis by FSP, investigation has to be performed about the influence of the operating conditions of the spray (flow field, dimensional distribution, precursor typology and physical properties, gas to liquid mass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

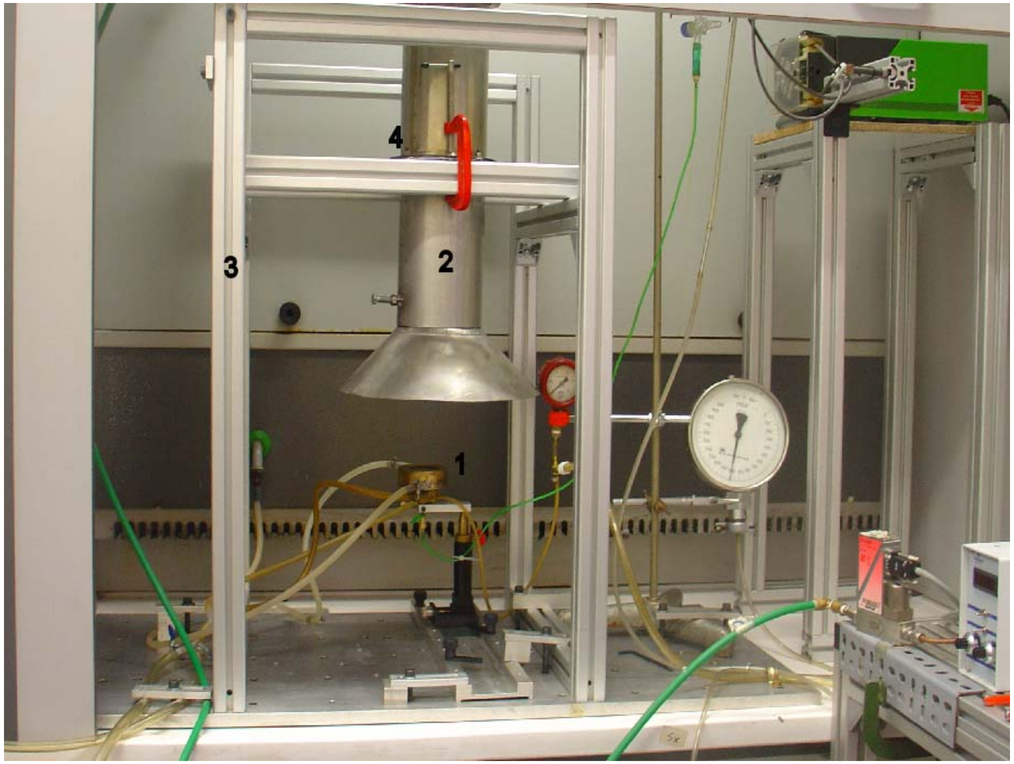
Introduction: the FSP



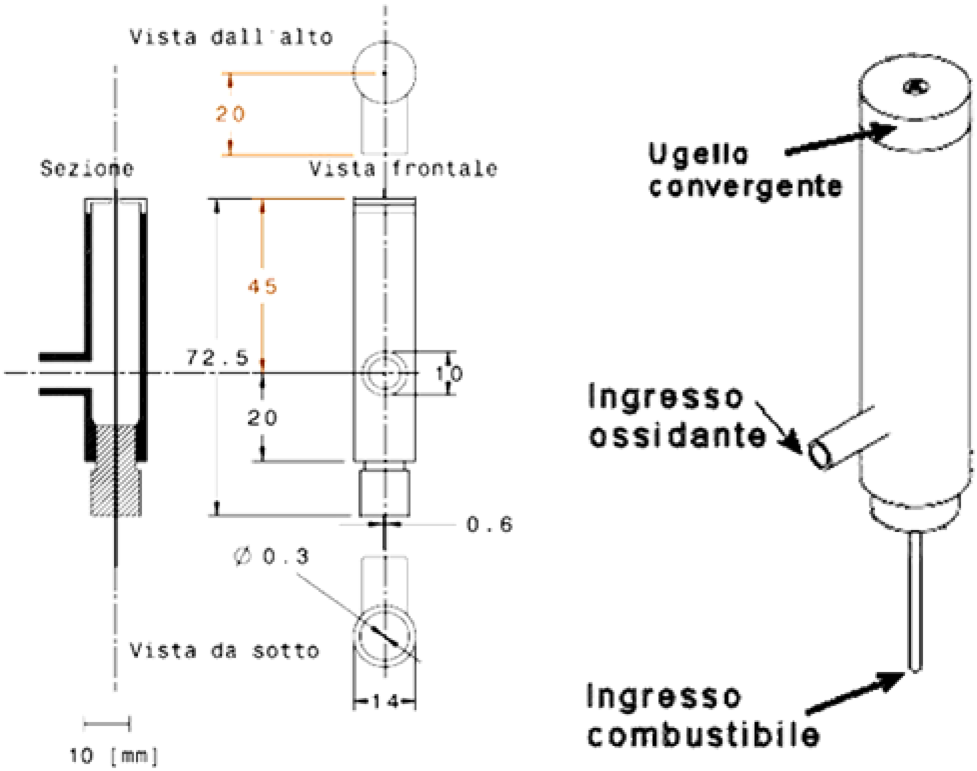
Standard configuration for nanoparticles synthesis through FSP

Experimental setup



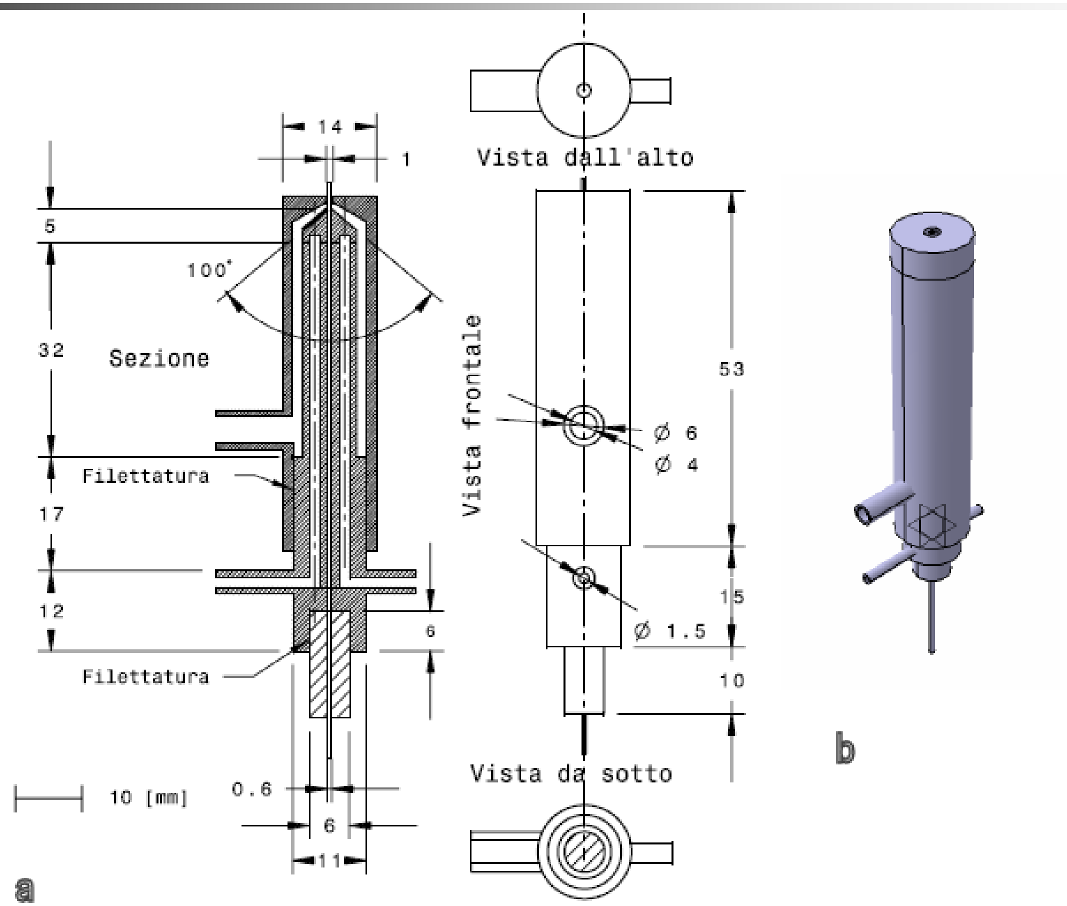


Experimental setup



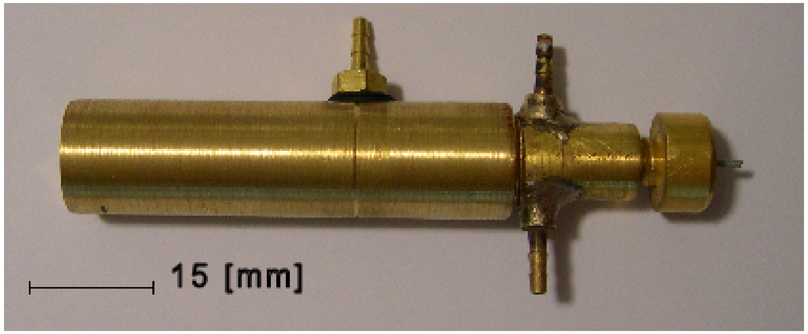
Conventional atomizer

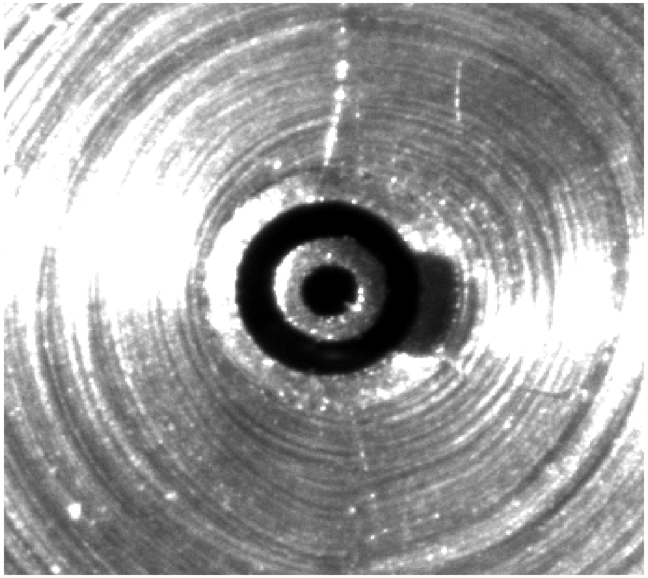
Experimental setup



The “new” designed atomizer

Experimental setup





The “new” atomizer

Experimental techniques

Ante Synthesis analysis:

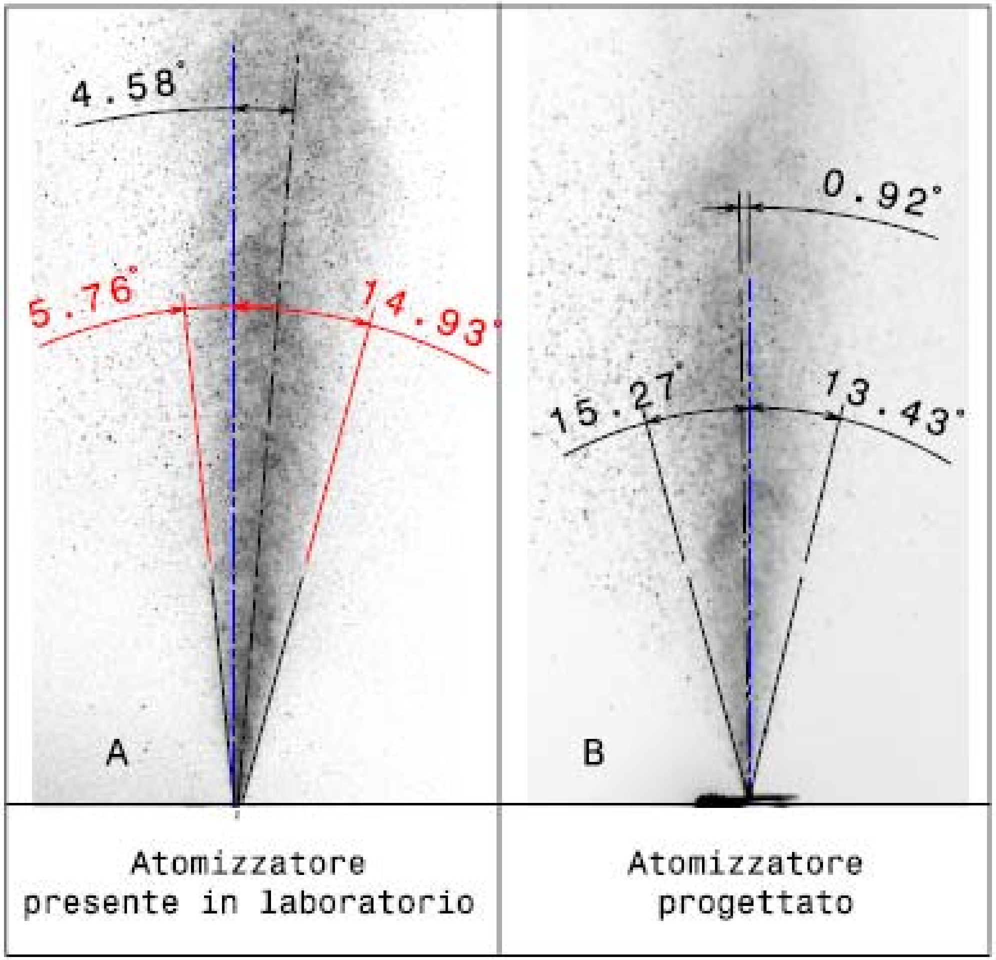
• PDA (Phase Doppler Anemometry) and visualizations to investigate the flow field and the dimensional distribution of the droplets generated by the atomizer. In fact, the fluid dynamic features of the spray in “cold” conditions strictly affect the quality of the final product (mainly dimensional homogeneity). Determination of the optimal operating field of the atomizer for the nanoparticles synthesis;

Post Synthesis analysis:

• TEM (Transmission Electron Microscopy) for dimensional analysis of nanoparticles;

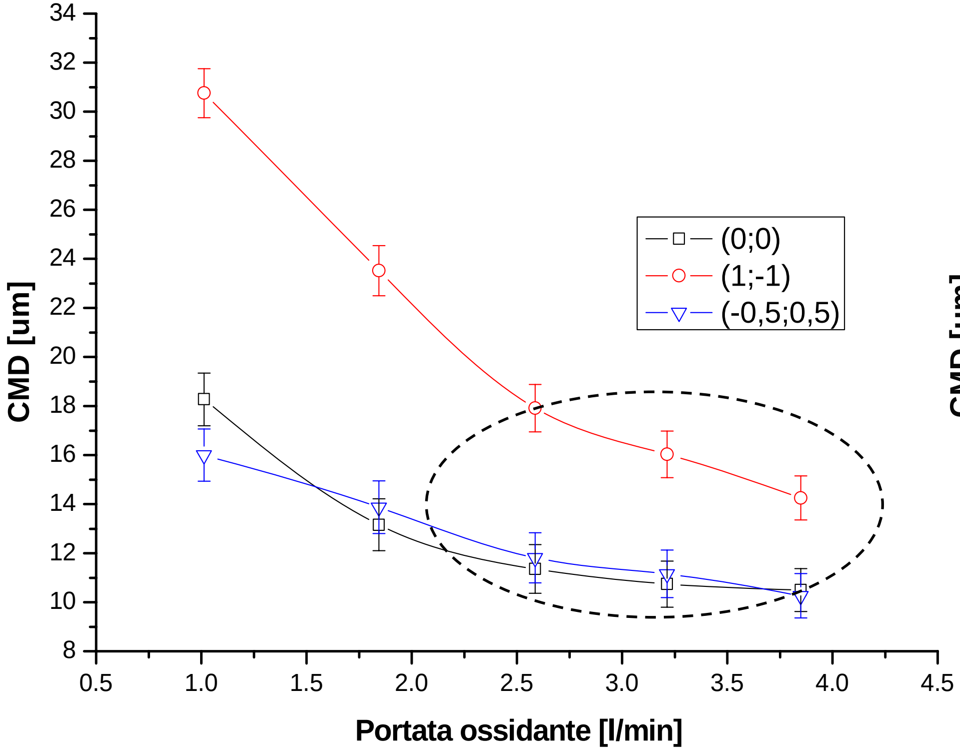
• XRD (X Ray Diffraction) for investigation of cristalline structure of nanoparticles (important mainly for TiO_2).

Experimental Results

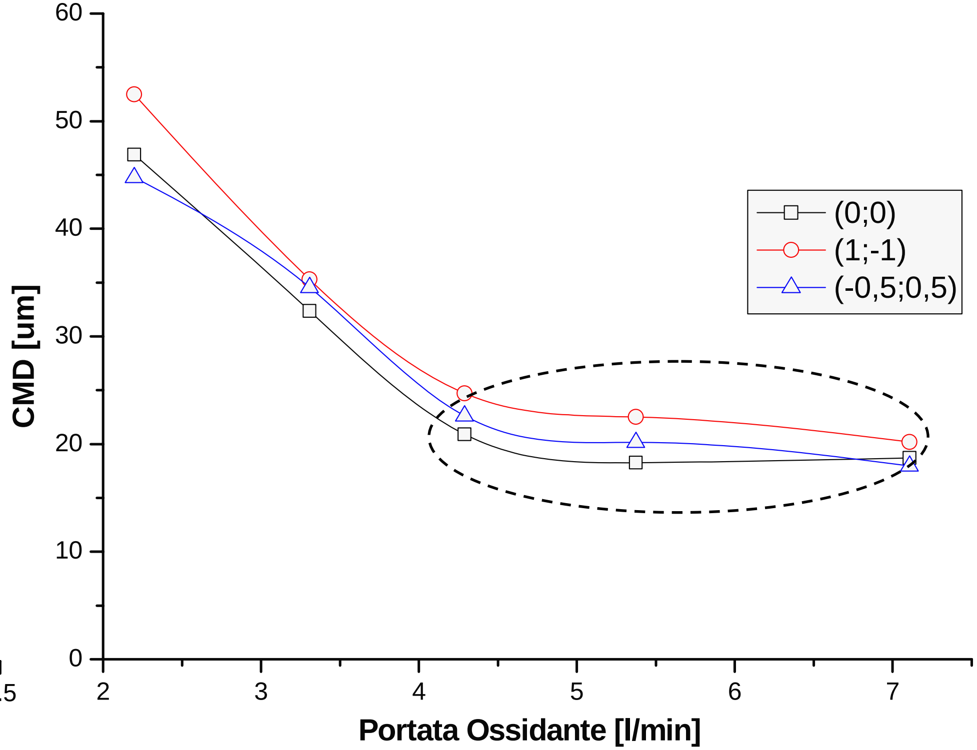


Experimental results: PDA

Conventional atomizer

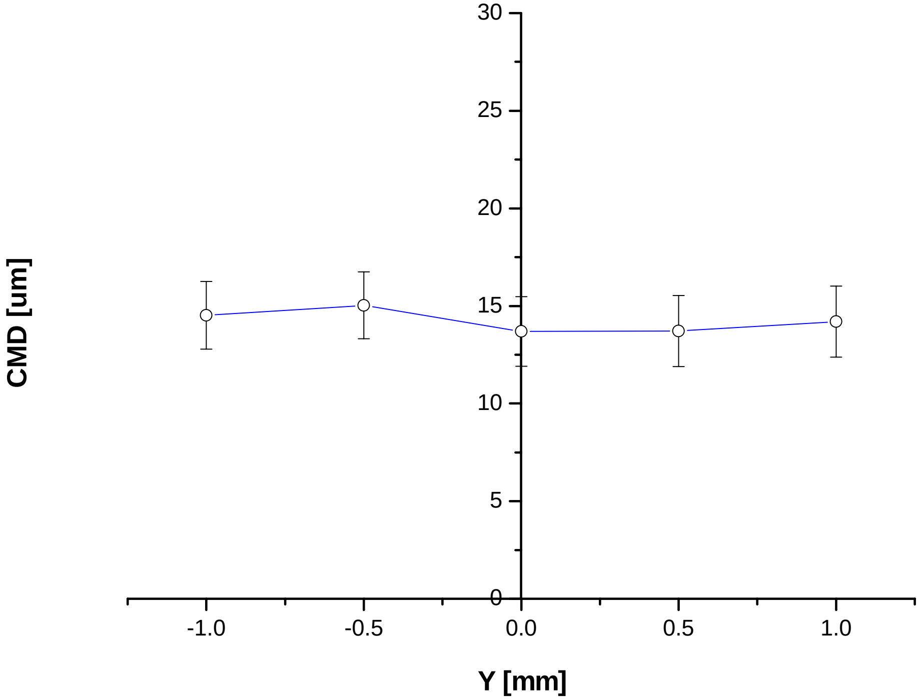


The “new” atomizer



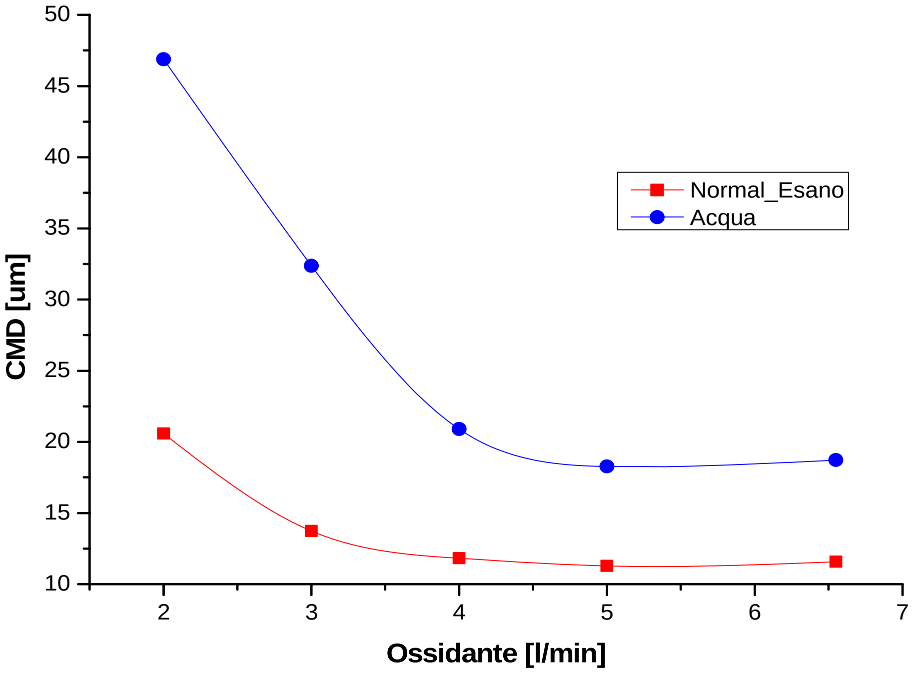
Counter medium diameter (CMD) Vs oxidant flow rate in different measurement positions

Experimental results: PDA



CMD radial profile

(50 mm downstream the efflux)

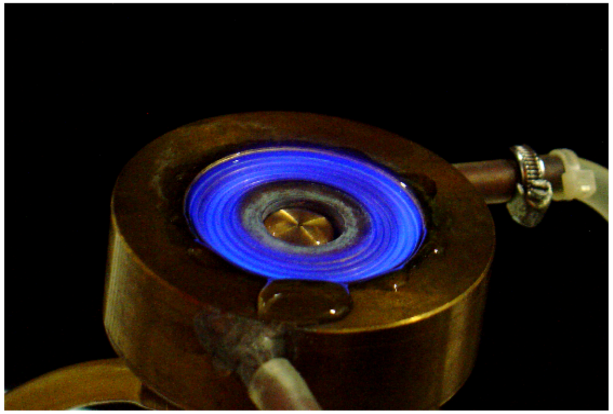


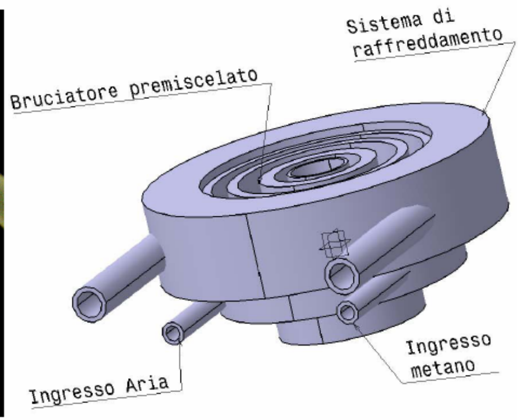
CMD Vs oxidant flow rate

Blue line: water

Red line: n-hexane

Experimental setup





Collecting system

Test-tube for powder analysis

Experimental Results

Selection of precursor and dispersion fuel for nanoparticles synthesis through FSP:

- Tetraethoxysilane (TEOS) in n-hexane for SiO_2 synthesis;
- Titanium tetraisopropoxide (TTIP) in ethanol for TiO_2 synthesis;
- In both cases: oxygen as dispersion-oxydation gas.

Experimental Results

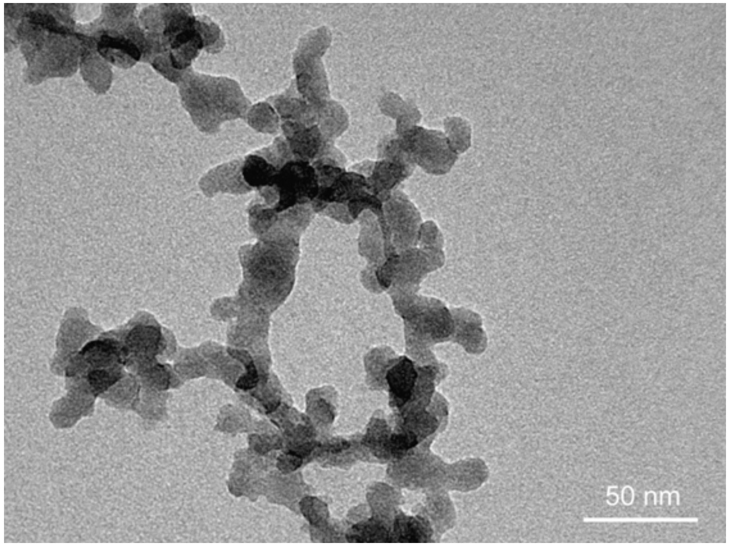
Visualization of the flame synthesis

Ethanol with TTIP spray flame

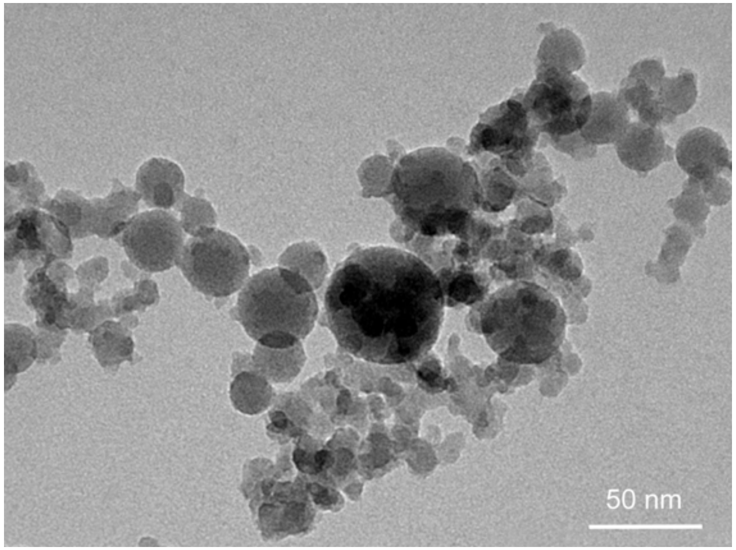
n-hexane with TEOS spray flame

Experimental Results

TEM analysis of SiO_2 nanoparticles



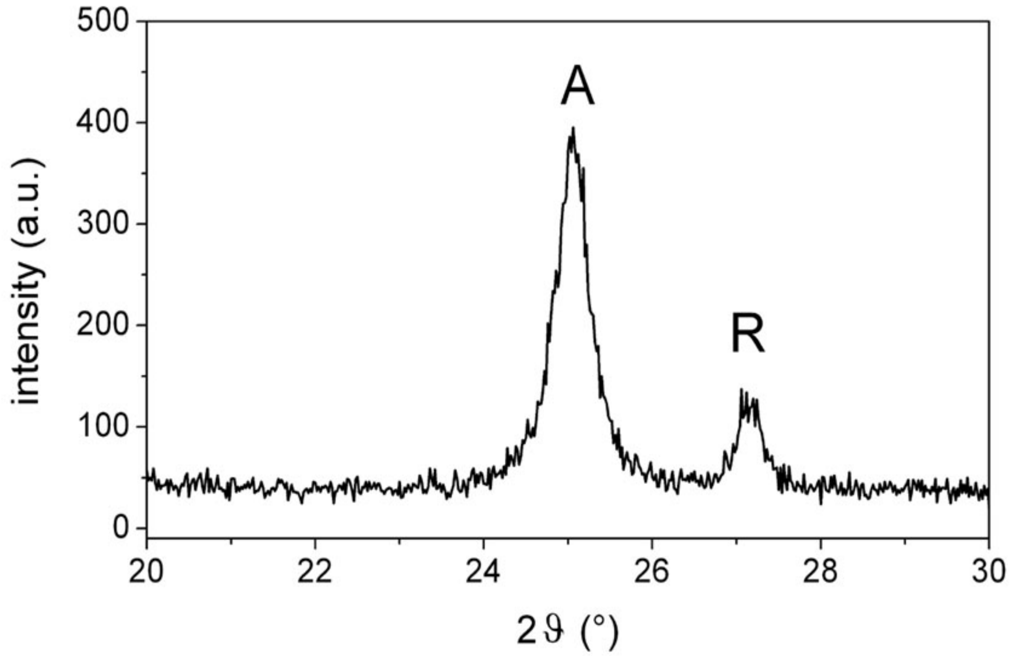
0.5 molar solution of precursor;



1 molar solution of precursor;

Experimental Results

XRD analysis of TiO_2 nanoparticles



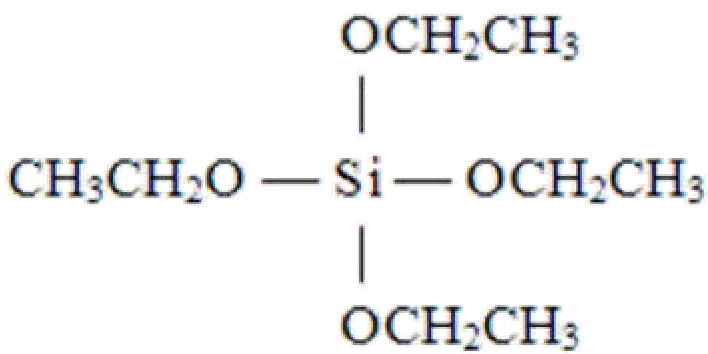
	%
Anatase	86.3
Rutile	13.7

A = Anatase; R = Rutile

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. PDA technique has been a very useful tool for system design, development and identification of optimal operating field for subsequent nanoparticles synthesis;
- 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.

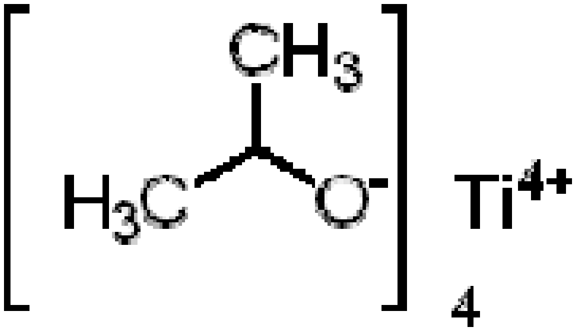
TEOS



Struttura della molecola di TEOS

	n-esano	TEOS
Formula molecolare	$CH_3(CH_2)_4CH_3$	$(C_2H_5O)_4Si$
Massa molare [g/mol]	86.18	208.33
Densità [g/cm^3]	0.66 (293 K)	0.934
Temperatura di fusione [K]	179	196
Temperatura di ebollizione [K]	342	442
Tensione superficiale [din/cm^2]	18.43	-

TTIP



Struttura della molecola di TTIP

	Etanolo	TTIP
Formula molecolare	C_2H_6O	$Ti(OC_3H_7)_4$
Massa molare [g/mol]	46.06	284.22
Densità [g/cm^3]	0.79	0.96
Temperatura di fusione [K]	158.8	289
Temperatura di ebollizione [K]	351.5	505

Numero di Weber

$$We = \rho_a U_r^2 \left(\frac{d_0}{\sigma} \right)$$

Liquido di lavoro	*We atomizzatore progettato*	*We atomizzatore presente in lab.*
Acqua	0.8	2.4
n-esano	3.3	9.5
etanolo	2.6	7.6