



UNIVERSITY OF ROME "LA SAPIENZA"
NANOTECHNOLOGIES ENGINEERING

**PRODUCTION AND
CHARACTERIZATION OF
NANOPARTICLES**

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GENERAL PRINCIPLES

Synthesis strategies

Can be divided by different approaches:

- ❑ **Method:** Top-Down or Bottom-Up
- ❑ **Nature of the process:** physical, chemical, biochemical or some combination.
- ❑ **Nature of the energy source**
- ❑ **Phase:** gas, liquid or solid

TOP-DOWN

- ❑ A coarse material piece is modified such to reach the target product requirements. This is performed mainly by discarding part of the material.
- ❑ This is a traditional process for nanoparticles production, based on milling of coarse material down to nanopowder.
- ❑ γ -Al₂O₃ powder may be treated by this method giving rise to a finer powder, of high specific surface up to 100 m²/g, depending on the process atmosphere in the milling device (inert gas or air).
- ❑ (-) Difficulty to reach nanosize, impurities in the coarse material will heterogeneously distribute in the final product
- ❑ (+) Easy and low cost: 20-30 €/kg
(TiO₂ 60%wt, 40nm +/- 20%, Degussa P25)

BOTTOM-UP

- This strategy aims to produce the product starting from single components. Generally, the strategy do not give rise to wasted material, although secondary reaction and/or post treatment processes may lead to discard part of the production.
- This strategy is the most used one, since it permits to target high quality and high purity nanoproducts by “construction” adding atoms together.
- For ease of an example, the production of TiO₂ is performed in two steps:
 - Reaction between TiCl₄ (gas) and oxygen to produce TiO₂ :
$$\text{TiCl}_4 + \text{O}_2 \rightarrow \text{TiO}_2 + 2 \text{Cl}_2$$
 - Nucleation and growth phenomena which determines the final habit and size of the nanoproduct.
- (+) Purity depends only on purity of reactants, even in cases of impurities they distribute homogeneously in the product
- (-) Costly: from 200 up to 2000 €/kg (99% TiO₂, 40 nm +/- 2%)

PHYSICAL METHODS

- Physical methods imply to produce nanomaterials by state modification, such as dimension, shape, phase.
- In this group following processes should be included:
 - Milling of material leading to a lower dimension;
 - Production of nanoparticles from solid through vapor condensation on quenching reactors or inert gas.

CHEMICAL METHODS

- In this group all processes involving a chemical reaction leading to the nanoparticles production are included.
- Some examples are (liquid):
 - Chemical precipitation
 - sol-gel processes
 - Hydrothermal processes
- Moreover (gas):
 - Flame processes
 - Laser assisted processes
 - Aerosol synthesis

BIOTECH METHODS

- Some nanostructures may be grown by processes involving bacteria, and are based on the same principles of the “membrane crystallization” (which will be discussed in detail later on).
- The processes includes biomineralization, that is the formation of crystals by biological membranes.
- Biomimetic methods includes the use of micelles or polymeric pockets that may produce nanoparticles of Ag_2O less than 10 nm; production of nano Pt, Pd, Rh and Ir is possible.

ENERGY SOURCES

The production of nanoparticles requires ALWAYS energy consumption.

- Possible sources includes:
 - laser
 - Plasma
 - Joule heating devices
 - Electron beams
 - microwaves
 - Hydrothermal
 - freeze drying
 - Ball milling
 - combustion
 - flame
 - Supercritical fluids

PHASE

- Nanoparticles may be produced in gas, liquid or solid phase.
- This is generally the first classification adopted to define a process.

	ADVANTAGES	DISADVANTAGES
GAS	-Dry products - Possibility to produce a board range of materials	-High energy costs - Undesired sintering
LIQUID	-Ease of control (size, purity) - Bottom-up approach	-Product is in suspension, powder requires post treatment - Requires identification of proper chemical reactions to obtain specific products
SOLID	-Low costs	- Top-down approach