



**UNIVERSITY OF ROME “LA SAPIENZA”
NANOTECHNOLOGIES ENGINEERING**

DISPERSION OF NANOPARTICLE SUSPENSIONS

OBJECTIVE OF THE RESEARCH WORK



The main objective was the development of an overall process, based on chemical precipitation, for the production of nanoparticles of Titanium Dioxide (TiO_2) of controlled size.

A new apparatus was adopted to perform the precipitation process: a rotating Spinning Disc Reactor (SDR), 30 cm in diameter.

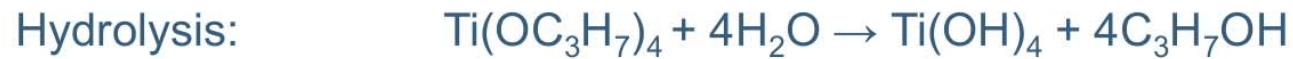
The performances given by the SDR were compared with those of a STR.

THE EXAMINED SYSTEM



The reaction was carried out by separately feeding titanium tetraisopropoxide (TTIP) and aqueous nitric acid solution (NAS).

REACTION SYSTEM



TiO₂ NANOPARTICLES PRODUCTION



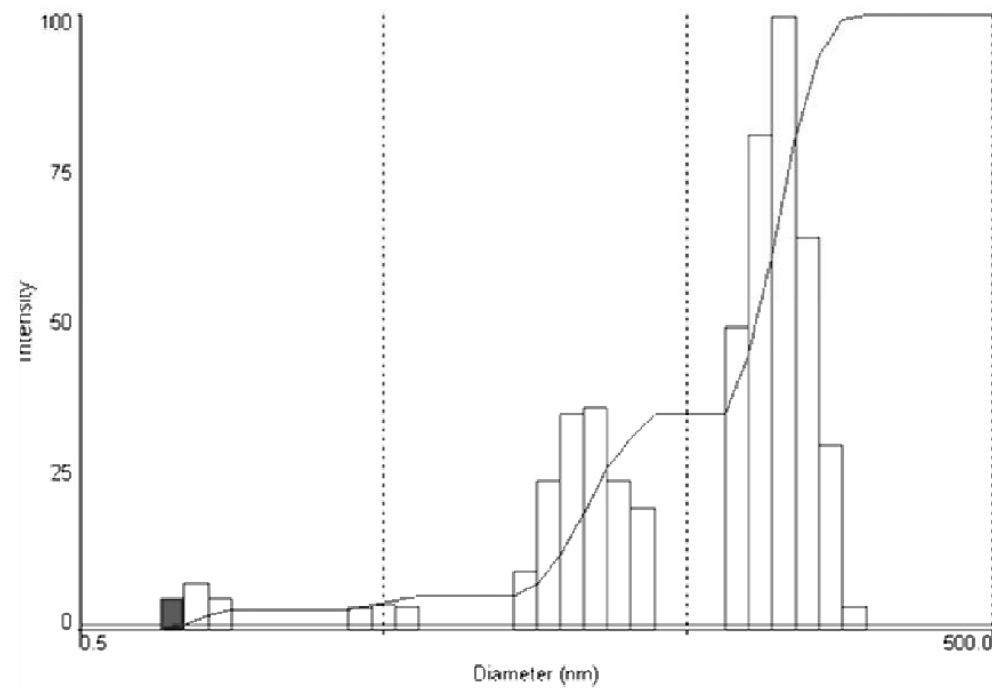
- The reaction-precipitation apparatus was a rotating **Spinning Disc Reactor - SDR** with a disc, made by brass 300 mm in diameter, rotating up to 1500 rpm.
- Both the apparatuses were operated at a temperature of 25°C, at a volumetric ratio distilled water-TTIP equal to 2/15 and in presence of a small amount of nitric acid.

PRODUCTION OF NANOPARTICLES



- The precipitated nanoparticles were highly agglomerated and were up to a few microns in size.
- After the precipitation step, the particles suspension was maintained in a stirred vessel at 1000 rpm for 24 hours in order to promote the redispersion of agglomerates due to the action of nitric acid.
- At fixed interval of time the polydisperse particle size distribution of titania was measured by means of a dynamic light scattering instrument (Plus 90 supplied by Brookhaven).

EVIDENCE OF AGGLOMERATION



TiO₂ particles are generated at a size around 1 nm, then they undertake a dramatic agglomeration.

Agglomerates Redispersio



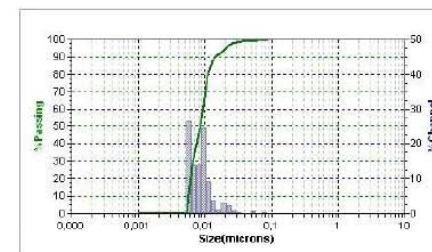
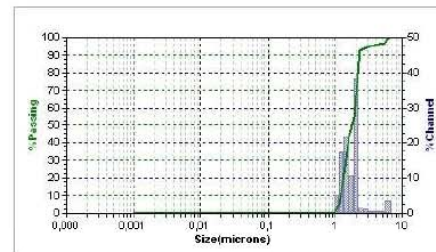
Under gentle agitation and at 25 °C

Nitric Acid

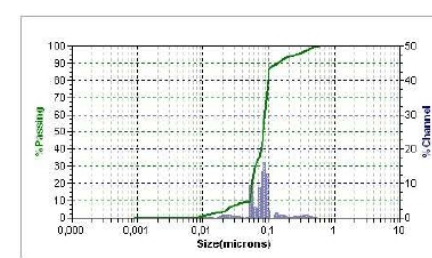
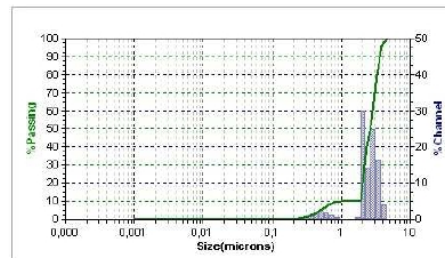
At the starting

After 24 h

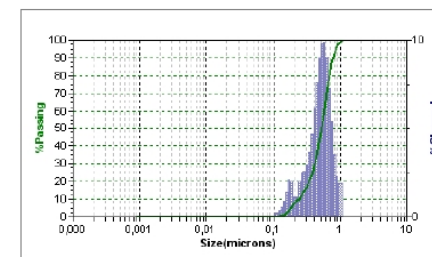
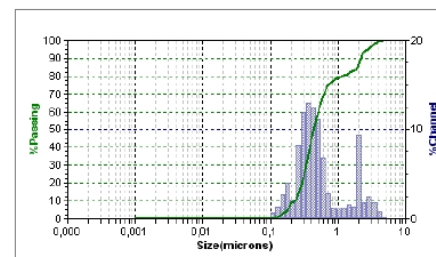
0,62 M



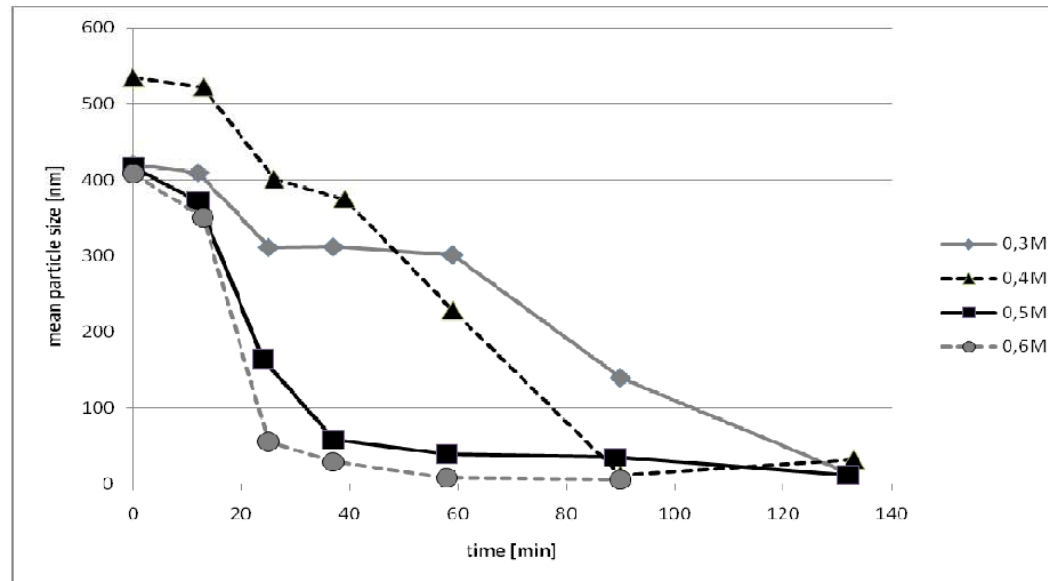
0,40 M



0,10 M



Agglomerates Redisperion



DISPERSION MODEL



An agglomerate particle, called i-mer, is formed by a number of identical elementary particles. In general, the aggregates are prone to a reversible process of agglomeration and redispersion:



The dispersion of an agglomerate composed by (j+i) elementary particles gives rise to two smaller aggregates, i-mer and j-mer ones. The corresponding dispersion rate can be assumed proportional to the number per unit volume of the original particles, N_{i+j} , as follows:

$$v_{\text{red},j,i} = b_{i,j} \times N_{i+j}$$

DISPERSION MODEL



The population balance of the agglomerated particles may be, then, written as:

$$\frac{dN_i}{dt} = -\frac{1}{2} N_i \cdot \sum_{j=1}^{i-1} (1 + \delta_{j,i-j}) \cdot b_{j,i-j} + \sum_{j=1}^{i \max - i} (1 + \delta_{i,j}) \cdot b_{i,j} \cdot N_{i+j}$$

where $\delta_{i,j}$ is Kronecher symbol:

$$\delta_{i,j} = 0 \text{ for } i \neq j;$$

$$\delta_{i,j} = 1 \text{ for } i = j$$

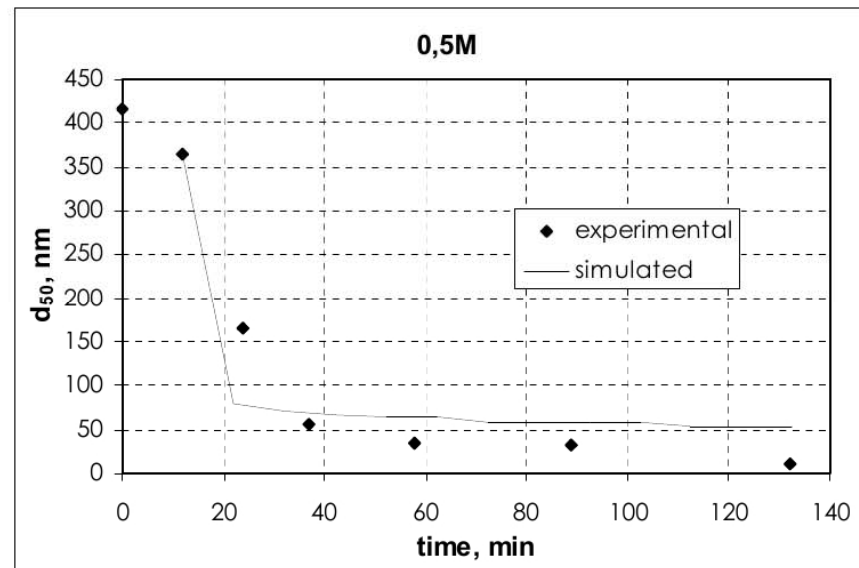
The coefficient $b_{i,j}$ is proportional to the volume of the (i+j)-mer V_{i+j} . By assuming a constant value for the volume shape factor of the particles, k_v :

$$V_{i+j} = k_v \cdot d_{i+j}^3$$

The kinetic coefficient of the redispersion process can be written as:

$$b_{i,j} = b_D \cdot d_{i+j}^3$$

RESULTS OF DISPERSION MODEL



The trend of the experimental data is adequately predicted, but the values of the final crystal size are not well estimated. More work is required.