

# Biomorphic TiO<sub>2</sub> synthesized using paper as biotemplates

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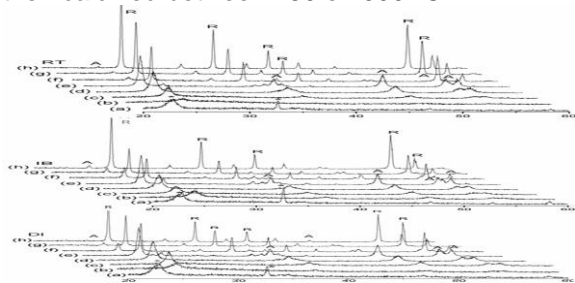
## Introduction

Biomorphic anatase-based plates were manufactured using paper as biotemplate. Ceramics with a microstructure similar to paper were produced by infiltration with TiCl<sub>4</sub> and hydrolysis in NH<sub>4</sub>OH at performed at temperatures between -25 and 25 °C. Then samples were dried (in air) and calcined at temperatures up to 1000 °C.

The anatase phase is formed at low temperatures (around 300 °C) but tends to be irreversibly converted to rutile at 600-700 °C [1-4]. The anatase to rutile transformation (ART) is deleterious for the photocatalytic activity of the material and then should be retarded/avoided. Crystallite growth upon firing seems to strongly affect the ART and then the remaining anatase content in the samples [5]. Anatase grains have an average diameter between 10 and 30 nm [2,3], and become unstable above that size. This occurs when the surface energy is no longer sufficient to compensate the lower energy of massive rutile grains; this process is obviously dependent of temperature and time of calcination.

## Results and Discussion

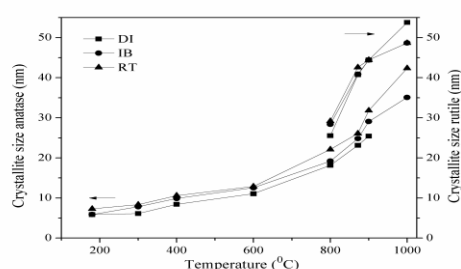
X-ray diffraction was used to quantify the anatase/rutile ratio in the calcined samples. **Figure 1** shows the diffraction patterns of X-ray infiltrates in templates: DI: ≅ -25 °C; IB: ≅ 0 °C e RT: ≅ 25 °C and then calcined between 180 e 1000 °C.



**Figure 1.** XRD samples infiltrated at -25 °C (DI), 0 °C (IB) and 25 °C (RT), and calcined at (a) 180, (b) 300, (c) 400, (d) 600, (e) 800, (f) 872, (g) 900, and (h) 1000 °C. A = anatase; R = rutile.

XRD results show that anatase-based biotemplates might be obtained at higher temperatures than reported previously.

The size of the crystallites of anatase and rutile were calculated from the Scherrer equation. As expected, anatase particles are smaller than rutile ones and tend to growth upon calcination. Rutile crystallites appear at around 800°C, when the crystallite size of anatase reached 18-22 nm, **Figure 2**.



**Figure 2.** Evolution of crystallite size of anatase and rutile phases upon calcination.

The use of templates while performing infiltration and hydrolysis at low temperature inhibited the network deformation, by delaying the nucleation and growth processes, then raising ART temperature.

## Conclusions

Biomorphic anatase plates were obtained from paper templates, by TiCl<sub>4</sub> infiltration and NH<sub>4</sub>OH hydrolysis. In the prepared samples the anatase content enhances with decreasing infiltration and hydrolysis temperature, being also reduced when the heat treatment temperature is higher. Samples produced at -25 °C and then calcined at 800 °C showed anatase as dominant phase. The preparation at lower temperatures seems to delay the crystallite growth, inhibiting the ART.

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