

# Morphology evolution of yttria porous ceramics during replica processing

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## ABSTRACT

In the context of green economy supported and widespread by United Nations (UN), the renewable energy sources are the unique mechanism to universalize the access to energy. Face to this strategic direction of change is essential to develop efficient components for saving energy such as porous ceramics that associate light with mechanical strength. Thus, the objective of this work concerns to improve the homogeneity of porous size distribution of yttria porous ceramics by evaluating their morphology during replica processing. Samples were subject to immersion into 30vol% yttria aqueous suspensions during an interval from 1min to 120min and sintered through careful thermal conditions. Based on the results, the weight and morphology of porous structure samples were directly influenced by immersion time, whereby intervals from 30-120min showed the best final products.

*Keywords:* yttria, replica, ceramic processing, rheology, biomass.

## 1. Introduction

Today, garbage has become a difficult logistic, economic and environmental issue to governments due to an increase of economic power of population and saturation of landfills. A

suitable way that can solve this great problem consists in converting garbage to energy. Since biogas derived from anaerobic decomposition of organic wastes (urban, rural and sewage) has a considerable quantity of  $\text{CH}_4$  ( $\geq 50\%$ ), it may replace natural gas in engines and for lighting.

Lighting emission by gas combustion in porous components is more efficient rather than a free flame, seeing that porous structures work as a heat circulator improving flame speed and stability<sup>[1]</sup>.

As yttria ( $\text{Y}_2\text{O}_3$ ) has similar physico-chemical properties to rare earth elements, it is very used as matrix for lighting emission phosphors such as  $\text{Eu}^{3+}$ ,  $\text{Dy}^{3+}$ ,  $\text{Tb}^{3+}$ . In addition, properties as high melting point ( $2400^\circ\text{C}$ ) and high refraction index ( $\cong 1.9$ ) present  $\text{Y}_2\text{O}_3$  as a potential material to be used in porous burners for lighting<sup>[2]</sup>.

Replica<sup>[3]</sup> is considered the most used method to produce porous ceramics, where the control of the stability of suspension is important so that it can cover the organic template surface uniformly. Besides, the immersion time ( $I_T$ ) of the template may contribute to control the morphology of cells and thickness of struts. Thus, the following work extends evaluate the effect of  $I_T$  on replica method to produce yttria nettings ( $N_Y$ ) with potential to be used as biogas mantles for lighting.

## 2 . Experimental

### 2.1 *Starting material*

Yttria commercial powder ( $\text{Y}_2\text{O}_3$ , Aldrich) with mean particle size of 164nm, specific surface area of  $13.59\text{m}^2.\text{g}^{-1}$ , real density of  $5.83\text{g}.\text{cm}^{-3}$  and chemical purity higher ( $>90.0\text{wt}\%$ ). The details of the physico-chemical characterization of the powders were reported in the our previous works<sup>[4,5]</sup>.

### 2.2 *Rheological characterization of colloidal suspensions*

Based on our previous work<sup>[4]</sup>, yttria aqueous suspensions with 30vol% of solids ( $S_{Y30}$ ) were prepared, whereby tetra methyl-ammonium hydroxide (TMAH -Aldrich-Chime, Germany) was used as deflocculant and carboxymethylcellulose (CMC) as binder.

The weight percent (wt%) of CMC was based on the total weight of the ceramic suspension. The rheological behavior of suspensions was performed with a RS600 rheometer, (Thermo Scientific, Germany). The sensor system consisted on a double cone rotor and a stationary plate (DC60/1°). Characterizing the suspensions stability the flow curves were determined in control rate mode (CR). Measurements were performed by increasing the shear rate from 0 to 500 s<sup>-1</sup> in 5 min., maintaining at 500 s<sup>-1</sup> for 2 min and returning to 0 in 5 min, with constant temperature at 25°C during these experiments. For each CR mode 200 points were measured.

### ***2.3 Producing and evaluating N<sub>Y</sub>s by replica from different immersion times***

Nylon-Cotton templates (NC) were immersed into S<sub>Y30</sub>, which immersion time (I<sub>T</sub>) from 5min to 120min. Thereafter removing the excess of suspension, the samples were dried at room temperature for 24 h. The impregnated NCs were subjected to a thermal treatment in air atmosphere, at 1600°C/1h. The mass of impregnated templates (N<sub>Y</sub>) was measured in an analytical balance (AG 204, Mettler Toledo), taking NC mass as reference. The morphology evolution of N<sub>Y</sub> was observed with stereoscope (Jena GSZ, Carl Zeiss, Germany).

## **3. Results and discussion**

### ***3.1 Flow curves of S<sub>Y30</sub>***

The comparative rheological behavior of S<sub>Y30</sub> suspensions with 0 and 0.5wt% of CMC is shown in Fig. 1. With no CMC addition (0wt%), both up and down curves were quite similar and presented a linear flow like Newtonian model. This result indicates that the yttria particles were well dispersed due to effective electrostatic stabilization mechanism supplied by TMAH. In the other hand, adding 0.5wt% of CMC a difference in the flow behavior of S<sub>Y30</sub> was observed. While subject of a constant force at 500s<sup>-1</sup>, the viscosity of suspension decreased, due to break down of agglomerates formed by CMC, so that releasing liquid to the system and also the realignment of particles. As soon as this force started decreasing, a process of

recovering the structure of ceramic suspension forward to its initial state began. Finally, an area between up and down curves was formed. This behavior is known as thixotropy<sup>[6]</sup>. For replica method this behavior is suitable, seeing that as subject of an external force the suspension is fluid enough to enter, fill and coat uniformly the surface of template, whereas under static condition, its viscosity is high enough to remain on the surface of replica template.

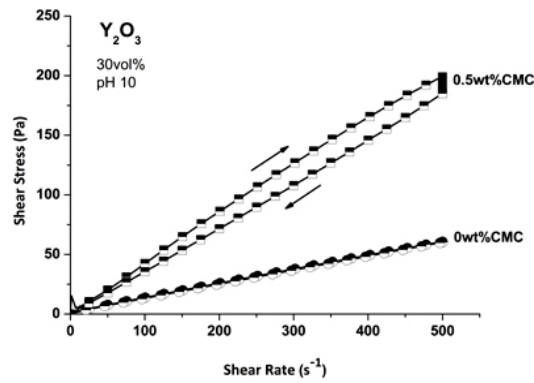


Fig. 1– Flow curves of  $S_{Y30}$  with 0-0.5 wt% of CMC in CR mode

According to previous rheological analyzes for replica method the following processing parameters are summarized in Table 1.

Table 1- Processing parameters for replica method

Solids load (vol%)	pH	Binder (wt%)	$\eta$ (mPa.s)	$\tau$ (Pa)	Thixotropy (Pa.s <sup>-1</sup> )
30.0	10	0.5	424.0	42.49	7.435

$\eta$ : apparent viscosity at 100s<sup>-1</sup>

### 3.2 Immersion time ( $I_T$ ) evaluation in replica method

Fig. 2 shows an increasing of the quantity (mass) of  $N_Y$  as a function of  $I_T$  from 5 to 120min. For times up to 15min the gain of mass was few (54.90%), meaning that the adhesion of the suspension on NC surface was not much efficient. Besides, from 15 to 30 min a significant shift in mass of 62.68% was observed. For further intervals this increasing still went on, although with less intensity.

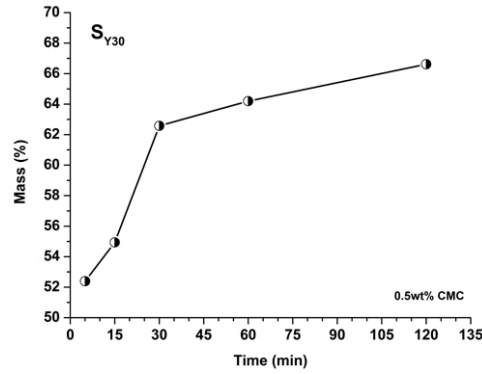


Fig. 2 - Increasing of  $N_{YS}$  mass as a function of  $I_T$ .

### 3.3 Morphology evolution of $N_Y$ as a function of $I_T$

The effect of  $I_T$  in morphology of sintered yttria nettings ( $N_Y$ ) is shown in Fig. 3. For  $I_T \leq 15$ min,  $N_{YS}$  presented a heterogeneous distribution of struts (thin/ thick), where it was more evident at  $I_T=5$ min. Even though no cells had been trapped, that confirms the rheological behavior of suspension was suitable, the following result shows that this interval did not supply favorable conditions for suspension cover the surface of NCs uniformly. However, the uniformity of struts was obtained apart from  $I_T=30$ min. Finally, the thickest strut was formed at  $I_T=120$ min. These results can be associated with those presented previously in Fig. 2, seeing that at  $I_T=30$ min a significant gain in mass of  $N_{YS}$  was observed.

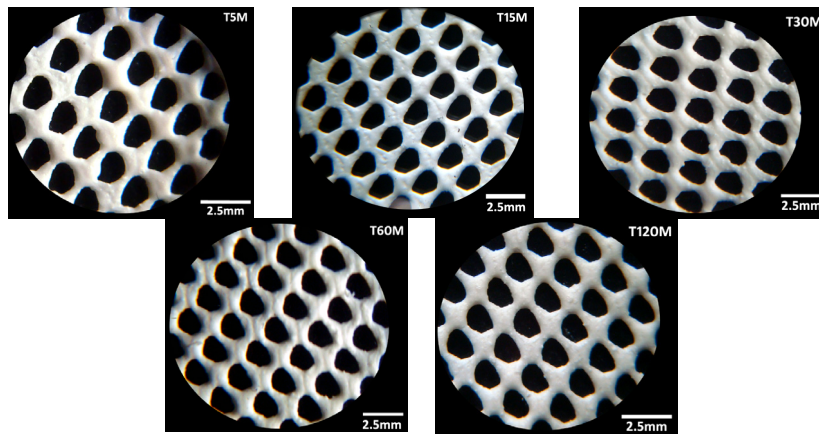


Fig. 3 –  $N_{YS}$  produced by replica, whereby  $I_T$  from 5-120min.

#### 4. Conclusions

Yttria nettings ( $N_Y$ ) were produced by replica, whereby immersion time ( $I_T$ ) of templates shifted from 5 to 120min was evaluated. A significant gain in mass of  $N_Y$ s (impregnated suspension), associated with homogeneous distribution of cells and thick struts could be gotten with  $I_T$  from 30min. Thus, even though the rheological behavior of a suspension is an important parameter for replica method, the role of immersion time ( $I_T$ ) is also very significant to control the morphology of cells, and also the thickness and homogeneity of struts.

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