

# Colloidal Stability of Aqueous Suspensions of Nano-Yttria Powders

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**Abstract**—Colloid processing of transparent yttria ceramics has been reported in this work. Aqueous suspensions of nano yttria powder were prepared by dispersing commercial powder into deionized water with triammonium citrate (TAC) as the dispersant. Effects of adsorbed dispersant, pH of suspending medium and solid loadings on the stability of the suspensions were characterized by means of sedimentation test, Zeta potential and viscosity measurements. Addition of 1.0 wt% TAC was enough to stabilize Y<sub>2</sub>O<sub>3</sub> suspensions. The prepared colloidal suspensions with 15vol% solid loadings were subsequently consolidated into green compacts via centrifugal casting and the transmittance of the sample sintered in vacuum was measured. Ceramics prepared has an highest in line transmittance of ~55% at 800 nm .

**Index Terms**—yttria, transparent ceramics, colloidal processing

## I. INTRODUCTION

Recently, yttria (Y<sub>2</sub>O<sub>3</sub>) ceramics have received considerable attention due to their attractive properties, including high corrosion resistivity, wide optical transmission range [1], good thermal stability and high melting point of around 2410°C [2]. Yttria ceramics are usually consolidated by conventional dry pressing method. This method has strong limitations particularly when components with a large size and complex shapes are to be produced [3]. Colloidal processing has attracted the attention of ceramists in the last decades because it is compatible with many consolidation techniques such as slip casting, gel casting, tape casting [4], centrifugal processing, etc. The colloidal technique enables to minimize strength-degrading defects, form complex shapes and high-volume production [5]. To prepare green sheets with a high packing density and uniform microstructure, a well-dispersed suspension is essential. Aqueous suspensions can be dispersed by electrostatic, steric, or electrostatic mechanisms [6]-[8]. Electrostatic stabilization is accomplished by generating like-charges of suspended ceramic particles. For steric stabilization, polymeric adsorption of a reasonable coverage and adsorption layer thickness is necessary to promote stability. Electrostatic stabilization is achieved by the

presence of adsorbed polymer or polyelectrolyte and significant electrical double-layer repulsion.

Triammonium citrate (TAC) is widely used as a highly effective dispersant to enhance slurry stability and to modify the rheology in the colloidal processing [9]. In this work, TAC was used as the dispersant to obtain stable Y<sub>2</sub>O<sub>3</sub> suspensions; the prepared colloidal suspensions were subsequently consolidated into green compacts via centrifugation.

## II. EXPERIMENT PROCEDURE

**Starting Materials:** Commercial Y<sub>2</sub>O<sub>3</sub> powder was used as the starting material. Triammonium citrate (TAC) with an average molecular weight of 243g/mol was used as the dispersant. Deionized water was used as the dispersing medium and when needed the pH was adjusted via the addition of HCl or NaOH aqueous solution.

**Suspension Preparation:** The suspensions used for all measurements were prepared by adding different Y<sub>2</sub>O<sub>3</sub> powder volume percentages to deionized water with the desired TAC content. The suspension was then dispersed via ball-milling for 8 h. subsequently; the suspensions were further dispersed by stirring magnetically for 15 min before each analysis.

**Characterization Techniques:** The adsorbed amount of TAC was analyzed by measuring the concentration of TAC in the clear supernatant using UV/VIS spectrometer (Lambda 750S, Perkin Elmer, CT, USA). The sedimentation test was performed using suspensions of 2.5vol% solid content. The prepared suspensions were dropped into 15mL centrifuge tubes, with the tubes sealed to avoid the evaporation of water. The height of the interface between the clear supernatant and the sedimented cake was recorded as a function of time. For the measurement of  $\xi$ -potential, suspensions with 1wt% powder content were prepared. Viscosity of the suspensions was conducted using a cone-plate viscometer (Brookfield DV-II+Pro, Brookfield Engineering Laboratories, USA) under different shear rate.

**Consolidation and Sintering:** Suspensions for consolidation were prepared by ball-milling the suspended powders in a plastic bottle for 2 h using ZrO<sub>2</sub> balls. Consolidation was performed by centrifugation at 3000 rpm for 40 min using a laboratory centrifuge. The consolidated cakes were subsequently dried, demoulded

and then sintered at 1700 °C for 5 h in vacuum of  $1.0 \times 10^{-3}$  Pa in a furnace with molybdenum heating element (VSF-7, Shenyang, China).

### III. RESULTS AND DISCUSSION

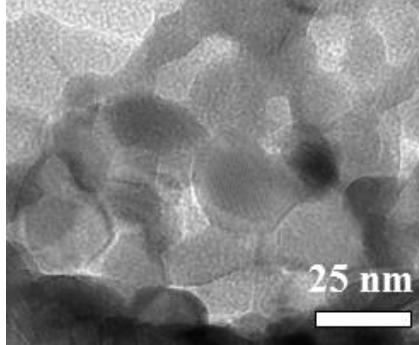


Figure 1. TEM micrograph showing particle morphologies of the starting  $Y_2O_3$  powder

Fig. 1 shows the TEM micrographs of starting  $Y_2O_3$  powder. The particles have an average size of 33 nm and show morphologies of round edges. The specific surface area was  $33.89\text{m}^2/\text{g}$  as determined by BET method. The average particle size of  $Y_2O_3$  calculated by BET method (35 nm) was coincident with that determined by TEM, which indicates the particles were well-dispersed and homogeneous.

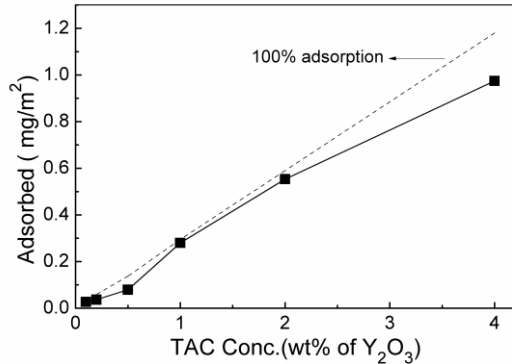


Figure 2. Amount of TAC adsorbed on  $Y_2O_3$  as a function of the initial amount of TAC added

Fig. 2 shows the adsorbed amount of TAC versus the initial amount of TAC added. It is clearly shown that almost all the TAC was adsorbed on the particle surface. This phenomena is similar to aqueous alumina suspensions using di-ammonium hydrogen citrate (DHC) as dispersant and MgO as coagulating agent [10].  $Y^{3+}$  ions produced from  $Y_2O_3$  suspension react with the unadsorbed TAC and form precipitate. This shifts the dispersant adsorption equilibrium towards the unsaturation side. This also explains that the optimal dosage of dispersant is not determined by the research of adsorption amount.

To investigate the effect of adsorption on the surface  $Y_2O_3$  powder, the zeta potentials are measured. Fig. 3 shows that the addition of 1.0wt% TAC shifted the IEP from 6.4 to a more acidic point. When the pH is higher

than 7.5, addition of 1.0wt% TAC increased the absolute  $\xi$ -potential of the suspension. The absolute  $\xi$ -potential of the suspension reaches a maximum of  $\sim 50$  mV at pH 11. Since the citrate anion has three carboxyl groups and is trivalent when fully dissociated, it can introduce more negative charges to the surface relative to the number of positive surface sites it neutralizes. For a given concentration, the citrate ions neutralize the existing positive surface sites and decrease the pH value of the isoelectric point. Some studies on the role of TAC in  $ZrO_2$  suspension have reported that the citrate ion can produce a short-range steric potential between  $ZrO_2$  particles in water [11].

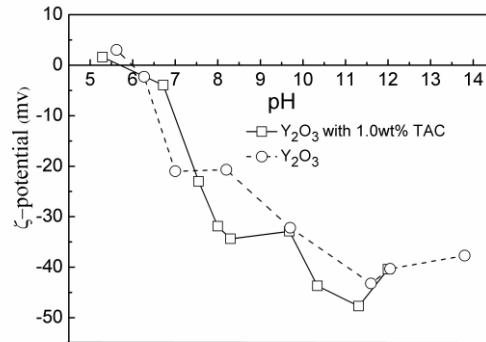


Figure 3. Zeta potential of 1wt%  $Y_2O_3$  suspension in different pH values

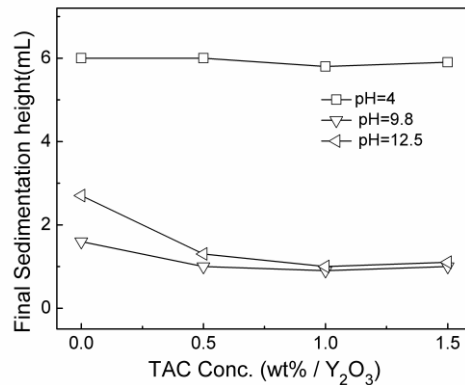


Figure 4. Final sedimentation height of 2.5 vol%  $Y_2O_3$  suspensions as a function of pH and DAC amount added after 3days

Fig. 4 displays the final sedimentation height of 2.5vol%  $Y_2O_3$  suspensions with different TAC concentrations recorded after 3 days. A critical concentration of 1.0wt%, which separates two different stages of sedimentation, is noticed. The final sedimentation height initially decreases as the TAC concentration increases and attains a smallest value when the concentration of TAC is 1.0wt%, and then the final sedimentation height increased because of the bridging effect between unabsorbed dispersant. Similar results are observed at other investigated pH values and also are included in Fig. 4. When the initial pH of suspension is 4, a mass of  $Y^{3+}$  dissolve into the solvent inducing the decrease in the electric double layer thickness and thereby cause unstable suspension with sedimentation

height of 6 MI [11]. At pH of 12.5, the final sedimentation heights of suspension were higher than those of pH at 9.8 owing to the increase of ionic strength.

The colloidal stability of  $Y_2O_3$  suspensions added with 1wt% of TAC is analyzed by measuring rheological behavior. Fig. 5 shows the variation of viscosity with different TAC concentrations for 2.5 vol%  $Y_2O_3$  suspensions. For the suspensions without and with a TAC concentration of 1.0wt%, the viscosity decreased as the shear rate increases, which is indicative of a shear-thinning behavior. For the suspension with 1.0wt% TAC, the viscosity results show a significant decrease initially from 237 to 125 MPa s at shear rate of  $1.32s^{-1}$ , compared with the suspension without TAC. This suggests that TAC enhance the stability of  $Y_2O_3$  suspension, which is accordance with the final sedimentation heights observed in Fig. 4.

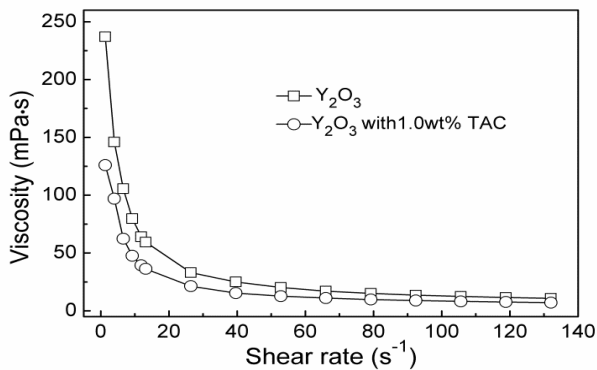


Figure 5. Viscosity for 2.5 vol%  $Y_2O_3$  suspensions without and with 1.0wt% TAC

Based on the data from adsorption, pH sedimentation test and  $\zeta$ -potential, it can be concluded that addition of 1.0 wt% TAC should be enough to stabilize  $Y_2O_3$  suspensions.

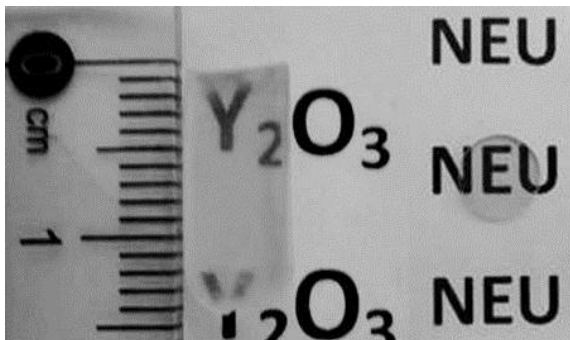


Figure 6. Image of vacuum-sintered and polished  $Y_2O_3$  transparent ceramics

Fig. 6 shows transparent  $Y_2O_3$  ceramics vacuum-sintered using centrifugally consolidated green compacts with suspensions of 15vol%  $Y_2O_3$  at solid loadings at pH of 9.8. The sample was double-polished and was of 1.5 mm thickness. The letters underneath the sample were clearly observed. Ceramics prepared has a highest in line transmittance of ~55% at 800 nm in Fig. 7.

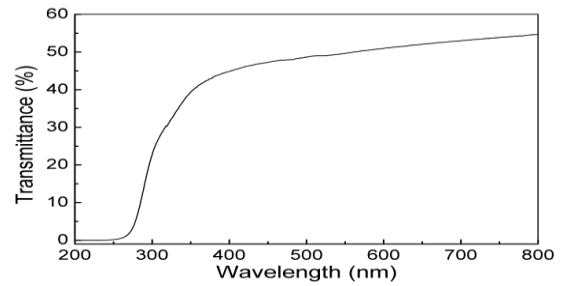


Figure 7. The inline transmittance of 1.5-mm thick yttria ceramics

#### IV. CONCLUSIONS

In this work, TAC was used as the dispersant to prepare stable  $Y_2O_3$  suspension. The characteristics of  $Y_2O_3$  suspensions were evaluated by the adsorption isothermal, sedimentation, and rheological measurements. It is shown that a more stable suspension was obtained at TAC concentrations of 1wt% at pH 9.8. When the solid loading of  $Y_2O_3$  suspensions was 15vol%, ceramics prepared was transparent and has a transmittance of ~55% at 800 nm.

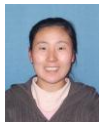
#### ACKNOWLEDGMENT

This work was supported in part by the Program for New Century Excellent Talents in University (Grant No. NCET-11-0076), National Natural Science Foundation of China (Grants No. 51172037 and 50990303), and the Fundamental Research Funds for the Central Universities (Grants No. N110410001, N110802001, and N100702001).

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