

# **Advanced Ceramics Progress**

Research Article

Journal Homepage: www.acerp.ir

## Dispersion Properties of Nano YSZ Particles in Aqueous Suspensions

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PAPER INFO

ABSTRACT

Paper history:

Received 17 July 2019 Accepted in revised form 29 September 2019

Keywords:

YSZ Suspension Dolapix Viscosity Microstructure of Sedimentation Layer In the present research, the aqueous suspensions of nano-sized YSZ particles were prepared using a common and available dispersant (Dolapix CE64) at different pH values and their stability were evaluated through the sedimentation height, viscosity measurement, and microstructural observation. Different amounts of dispersant were tested and the optimum percentage was examined by measuring the viscosity of suspensions. It was found that the use of Dolapix CE64 resulted in a suspension with a low sedimentation height and viscosity. The suspension prepared at pH = 4 with the optimum amount of dispersant (Dolapix CE64 0.1wt%) indicated a reasonable value of zeta potential. Moreover, the packing of particles in sediment layers was evaluated by sedimentation parameters (sedimentation volume and packing density) and the sediment layers were characterized by the microstructural observation. The microstructural observation revealed homogeneous packing of particles in the sedimentation layer, demonstrating the suspension stability, and proper dispersion of particles.

#### 1. INTRODUCTION

The rheological properties of a suspension including the fluidity and stability determine the response of the material to shear stresses [1, 2]. Factors that affect the fluidity of a suspension are the size and shape of the dispersed particles, viscosity, pH, and solid loading percentage of the suspension. Suspension rheology is important in ceramics science especially in many shaping techniques, such as slip casting, electrophoretic deposition, extrusion, and spray drying and coating methods such as immersion and glaze applications [3-5].

Yttria stabilized zirconia (YSZ) suspension is used in many industries through the above-mentioned methods because of its excellent properties such as low thermal conductivity, thermal shock resistance, high ion conductivity, and high thermal expansion coefficient [6,7]. In fact, YSZ has thermal, mechanical, and electrical applications in fuel cells, thermal barrier other high-temperature coatings, sensors. and applications [8, 9]. Therefore, it is important to stabilize the YSZ suspension and study its rheological behavior to investigate the suspension repeatability properties. Many variables, such as pH, solid loading, dispersant, and dispersion medium, which affect the surface properties of YSZ particles have been examined. The absorption of dispersants and pH value can alter the surface properties and rheological behavior of particles and suspensions. In previous studies, various suspensions have been prepared by different dispersants such as Darven C (an Ammonium Polymethacrylate), Tiron (Sodium catechol sulfate), PAA (polyacrylic acid), iodine, ammonium polyacrylate, diammonium citrate, etc. in aqueous media [10-13].

The performance of dispersants is based on the three mechanisms of electrostatic, steric, and electrosteric stabilization. The electrostatic mechanism acts through the absorption of ions around the particle, which causes repulsion between them. Steric mechanism acts through attaching polymeric chains to particles and the electrosteric stabilization is their combination. repulsion develops between particles preventing particles from being flocculated while the strands cause particle separation alternatively by combining the two above-mentioned mechanisms [14, 15]. Furthermore, ions are absorbed onto the surface of particles to form a double layer. This double layer consists of a stern layer and a diffuse layer. In fact, the potential of the diffuse

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layer is considered as the zeta potential of the particle. The suspension stability diminishes when the potential and charge decrease on the surface of the particles in the suspension because the attraction between the particles grows and results in the rising of the viscosity and shear stress.

The limited number of effective parameters has been investigated for the preparation and processing of YSZ suspensions. Meanwhile, previous papers have rarely studied the differences between Dolapix dispersants on the stability of YSZ suspensions [16,17]. For example, it has been reported that 0.25wt% of PAA is required to prepare a stable YSZ suspension [10], or 200ppm Dolapix can yield the minimum viscosity in YSZ dispersion [18].

In this paper, it has been tried to prepare a stable suspension of YSZ using Dolapix, which is a common, available, inexpensive, and easy to use additive. The effective variables for suspension stability and pH value were examined. Attempts were made to present a comprehensive and systematic evaluation of the YSZ suspension stability through measuring the suspension viscosity, sedimentation parameters, zeta potential, and microstructural observation. Moreover, the effects of dispersant and pH were compared on the sedimentation height and viscosity of suspensions and the microstructures of sedimentation part of YSZ suspensions were characterized via SEM (Scanning electron microscopy). Finally, the stability of YSZ suspensions was documented under various conditions.

#### 2. EXPERIMENTAL PROCEDURES

#### 2.1. Materials and Methods

Yttria-stabilized zirconia (YSZ, 3mol%) was purchased from Tosoh Company (Japan, d<sub>50</sub>=80nm, purity = 99.6%, density = 6g.cm<sup>-3</sup>) used as the starting material. Dolapix CE64 from Zschimmer and Schwarz (Germany) was used as a dispersant for suspension preparation. XRD (X-ray Diffraction) pattern and SEM micrographs of YSZ powder are displayed in Figs. 1 and 2, respectively.

## 2.2 Suspension Preparation

The 40wt% solid loading suspensions were prepared through mixing the YSZ powder with the dissolved dispersant in water. Initially, the dispersant was added to water at different percentages and placed on a stirrer for 1h. YSZ powder was then added to the water and dispersant, further stirred for 20min by a planetary mill (200 rpm). The pH was adjusted by HCl and NaOH solutions after 24h. Four of the most important

suspensions have been denoted with N, D, P, and F in this manuscript whose characteristics are listed in Table 1.

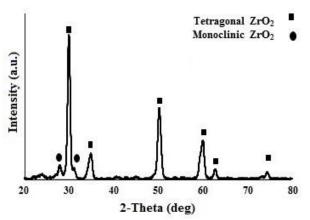


Figure 1. XRD pattern of YSZ powder

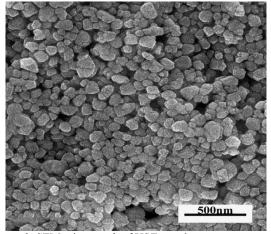


Figure 2. SEM micrograph of YSZ powder

#### 2.3 Characterization

40wt% suspensions were poured into a graduated cylinder after the mixing process, and sedimentation heights were measured within five days. The 40wt% solid loading was the highest value that could be handle in this project. The pH values were determined by a pH-meter (Titrino DMS 716, Metrohom). The rheological behavior of YSZ suspensions was examined at a temperature of 25+0.1 C using a controlled stress rheometer (MCR301, Anton Paar Physica). The zeta potential by measurement performed was zetasizer (Zetasizer3000 HAS, Malvern). Sedimentation volume (SV) and packing density (PD) of the suspensions were calculated according to Equations 1 and 2 [19, 20]:

SV (cm<sup>3</sup>.g<sup>-1</sup>) = Volume of the sedimentation layer / weight of the sedimentation layer

PD (
$$\%$$
) = 1/ (Volume of the sedimentation layer × powder density)

(Eq. 1)

Furthermore, the following procedure was applied for sample preparation to investigate the microstructural observation of sedimentation layers. Particles were starting to settle down and placing at the bottom of the container after preparing a suspension. Aggregations of these particles led to the formation of a layer of material at the bottom of suspension called the sedimentation layer. The challenging point in this research was the microstructural observation of these layers (there is a suggestive schematic view for the formation of the sedimentation layer by authors in fig. 3). To this end, the sedimentation layer of each suspension was separated by removing the stable suspension on top of each cylinder and the as-received sediment layer was placed in the furnace at 500°C to achieve a sufficient green density. The dried samples were put on and glued on a sample holder. The microstructural observation of the bottom and upper parts of the dried sedimentation layers of suspensions were characterized by SEM (Cambridge 360). The bottom layer housed the earlier settled particles while the upper layer encompassed the particles settled during the testing time.

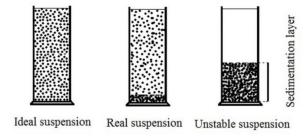


Figure 3. Schematic view of a suspension

**TABLE 1.** Codes of suspensions

Suspension code	Dispersant (wt.%)	рН
N D	- 0.1 wt% CE64	Not changed
P	-	4
F	0.1 wt% CE64	4

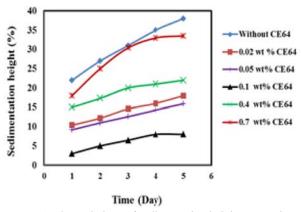
#### 3. RESULTS AND DISCUSSION

#### 3.1. Sedimentation Height

Fig. 4 demonstrates the sedimentation diagram of the YSZ suspensions prepared by Dolapix CE64. It can be observed that the sedimentation heights of suspensions increased during these five days since the sediment particles reveal the instability of suspension. If the YSZ particles are well dispersed in the media, they remain suspended and would not settles down.

Fig. 4 illustrates that the use of Dolapix CE64 as a dispersant decreases the sedimentation height of the N suspension (primary suspension). The lower slope of the sedimentation height during long storage of suspension suggests that the stability and durability of the suspensions have been improved over time. In other words, the descending slope of the graphs in the presence of the dispersant indicates their higher stable. The sedimentation height of the N suspension (without Dolapix CE64) was as much as 20% and reached 38% of the total volume of suspension within five days, which is the highest value. The polymeric chains of dispersant have been absorbed on the surfaces of YSZ particles in the presence of a small amount of Dolapix CE64 and resulted in electrostatic repulsion between them. This, in turn, increased the repulsive force between the particles and reduced the impact of the Van der Waals force culminating in the dispersion of particles. In addition, the polymer chains of polyelectrolyte create steric repulsion thereby preventing particles from being absorbed by the electrosteric mechanism [21,22].

According to fig. 4, small amounts of dispersant (0.02wt%) have had a minor effect on suspension stability.



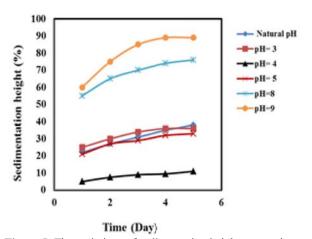
**Figure 4.** The variations of sedimentation height versus time for different amounts of Dolapix

Nevertheless, a greater amount of dispersant is absorbed on the surfaces of the particles with a further increase in the amount of dispersant from 0.02 to 0.05wt% whereby the suspension stability also increased. As long as the particle surfaces are not saturated, they tend to absorb more dispersants. Therefore, the stability of the system improves with the addition of the dispersant content up to 0.1wt%, since the minimum sedimentation height can be seen (3%) on the first day as well as after five days (8%). Higher values of Dolapix decreased the suspension stability while increased the sedimentation height. The reason is that the surfaces of the particles were saturated with the dispersant and the excess amounts

of dispersant were not absorbed on the particle surfaces and remained in the suspension [23,24]. This reason can be attributed to the fact that the ionic strength has been raised, the double layer has been compressed, the repulsive force between the particles has been decreased, and the probability of the flocculation of the particles has been increased [3,25-27]. Accordingly, the optimum percentage of Dolapix CE64 is as much as 0.1 wt%.

Fig. 5 indicates the sedimentation height of the suspension prepared at different pH values without dispersant.

It can be observed that the sedimentation of the suspensions is higher at basic pH rather than acidic ones. It suggests that YSZ suspensions are more stable at acidic pH values. Moreover, the slopes of the graphs are smaller at lower pH values, which indicates the greater durability of these suspensions. H<sup>+</sup> ions are absorbed on the particle surfaces and reinforce the double layer thereby enhancing the zeta potential of the particles by adding HCl into the suspension [27]. The electrostatic repulsion prevents particles from being absorbed together due to the formation of the mentioned layer. The surfaces of the particles have been saturated with H<sup>+</sup> ions and additional ions remained in the solution among the particles increasing the ion strength and hindering the movement of particles inside the suspension by increasing the amount of HCl from the optimum level (pH = 4).

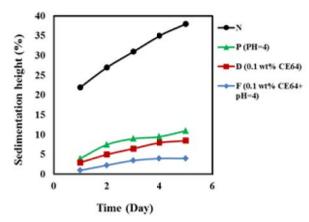


**Figure 5.** The variations of sedimentation height versus time for different pH values

As a result, the sedimentation height has been raised. Positive charges are neutralized by negative charges, reaching the pH at which the charge surface and zeta potential of particles became zero with the gradual increase of OH ions, which is called the isoelectric point. The negative hydroxyl group has been enhanced on the surfaces of YSZ particles with the further addition of OH. Also, a hydrogen bond is created

from the absorption between the O and H atoms of the hydroxyl group with the H and O atoms of the water molecule, which increased the suspension viscosity [9,28,29].

Finally, the F suspension was prepared by adding the optimum amount of Dolapix (0.1wt%) at pH = 4 and was examined over 5 days. The sediment height of N, D, P, and F suspensions has been compared in fig. 6.



**Figure 6.** The variations of sedimentation height versus time for different suspensions

According to fig. 6, the suspension sedimentation height decreases from 22 % to 1 % on the first day by applying optimal conditions. This suggests that the presence of both agents (dispersant and pH adjustment) in the suspension exhibits a more suitable effect and the electrosteric repulsion prevents flocculating and settling of particles thereby improving the suspension stability.

In addition, the suspension settling rate has become steady after 3 days and its sedimentation height (compared to the long time and suspension preparation time) remained constant. Fig. 6 also illustrates that the addition of 0.1wt% Dolapix CE64 is more effective than adjusting pH value at 4.

#### 3.2. Viscosity

Fig. 7 shows the viscosity of the prepared suspensions. N suspension had the maximum viscosity and pH variation of the suspension (P suspension) reduced its viscosity. The addition of Dolapix (D suspension) had also a remarkable effect on the viscosity. The addition of Dolapix CE64 and alteration of pH to 4 (F suspension) reduced the viscosity to 0.04Pa.s with a smoother slope. The viscosity is strongly reduced and the particles are separated from each other into the suspension prevented their flocculation at shear rates higher than 101.s<sup>-1</sup>. The prepared suspensions act as a Newtonian fluid.

The flow behavior of dispersions is controlled by the balance between hydrodynamic and thermodynamic interactions as well as Brownian particle motion. Thermodynamic interactions mainly include electrostatic and steric repulsion and Van der Waals and depletion attraction. The viscosity of a Newtonian fluid remains constant and has a linear relationship with shear stress [30]. The prepared N suspension departs from the Newtonian behavior and the viscosity is found to decrease with increasing shear rate, which refers the shear thinning behavior. The yield viscosity of N suspension shows that it behaves as a solid at rest and start to flow only when the applied external force overcomes the internal structural force. Yield viscosities of the other suspensions were low and their dependence on shear rate was decreased. It can be said that they tend to Newtonian behavior.

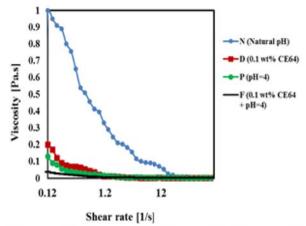


Figure 7. Viscosity of N, P, D, and F suspensions as a function of shear rate

### 3.3. Zeta Potential

Table 2 presents the zeta potential of N, D, P, and F suspensions. The zeta potential of YSZ suspension is low without adding Dolapix CE64, which suggest that these particles had a minor positive charge on their surfaces.

**TABLE 2**. Zeta potential and particle size of the prepared suspensions

Suspension code	Zeta potential (mV)	Particle size (nm)
N	+12	400
P	+35	200
D	-17	180
F	-25	100

Fig. 8(a) illustrates a suggested schematic representation of ion absorption on YSZ particles; the distances between the particles are supposed to be very low because of the insufficient repulsive force that result in instability and agglomeration of particles. The positive charge increased with the addition of HCl with the zeta potential of P suspension exceeding +35 mV. When HCl was added to the suspension to

adjust the pH value, H<sup>+</sup> ions were absorbed on YSZ, which provides relatively enough superficial charges and causes the particles to detach off each other and reach relative stability. Fig. 8(b) displays the suggested schematic of YSZ particles. It means that the stability of the YSZ suspension was improved with an alteration of pH to 4.

On the other hand, the positive charge was neutralized and reduced through adding 0.1wt% Dolapix CE64 where the zeta potential had a negative value of -17mV in the presence of dispersant (D suspension). The zeta potential approached -25mV when 0.1wt% Dolapix CE64 and HCl (pH = 4) were used (F suspension). The zeta potential of F and P suspensions implies that these suspensions are stable.

Dolapix CE64 was used in this research as an anionic polyelectrolyte. The dissociation reaction of Dolapix CE64 creates free carboxylic acid groups, which can be neutral COOH or dissociated to COO. When Dolapix is absorbed on the surfaces of particles, it creates electrostatic charges between particles to develop the spatial inhibition as illustrated in fig. 8(c), which kept the particles suspended inside the suspension and extended the distances between YSZ particles [2,10,31,32]. The negative charges on the surfaces were neutralized by adding HCl to the suspension where neutral polymer chains develop steric stability among particles. On the other hand, some of the charges on the polymer chains are not neutralized due to the lack of sufficient positive ions. As a result, the electrosteric charges cause repulsion and stability. The neutralization of several negative charges reduces the value of zeta potential in the F sample. Thereby, steric and electrosteric repulsion between particles (F suspension) plays a more important role.

# 3.4. Packing Density and Sedimentation Volumes of the Sedimentation Layer

Sedimentation volumes (SV) and packing densities (PD) of N, D, P, and F suspensions were measured during three days (Table 3). Low amounts of SV and high values of PD indicate a stable suspension. N suspension has the highest SV and the lowest PD. Particles were absorbed to each other without the presence of any dispersant as a result of creating a flocculated and heterogeneous state, which formed a deposit after 3 days. This sediment occupies a great space and therefore, shows a high PD value. In other words, when the superficial charges are non-uniform (low zeta potential), the particles attach each other and create agglomerate structures, in which many free spaces are created between them. Therefore, the structure of the sediment is not compactly compressed and there will be a great deal of free space [33,34]. Higher PD values can be seen for D and P suspensions where the PD value of F suspension is also approximately 3 times larger than that of the N suspension. In reality, it can be seen that deflocculated particles of F suspension occupies a little space of the

cylinder after 3 days suggesting higher packing density in the sediment.

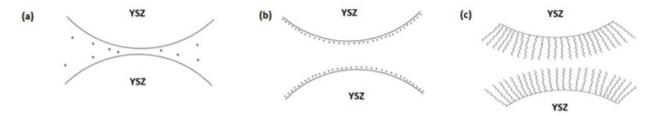


Figure 8. Suggested schematic representation of ion absorption on YSZ particles (a) N, (b) p and (c) F suspensions

**TABLE 3**. SV and PD values of YSZ suspensions

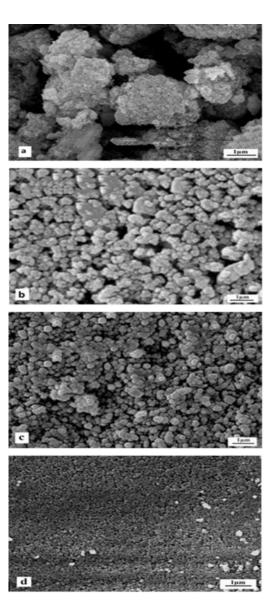
Suspension code	PD (%)	SV(cm <sup>3</sup> /gr)
N	6.2	0.67
P	10.6	0.53
D	12.5	0.47
F	18.0	0.38

#### 3.5. Microstructural Characterization

Fig. 9(a) shows the microstructure of the bottom layer of N suspension. N suspension was unstable and the repulsion forces between its particles were very week. Accordingly, the particles may approach each other and thus, agglomeration can occur. The large particles or agglomerates deposited very quickly at the bottom of the suspension vessel just after preparation of this suspension. Due to the instability of this suspension, the sedimentation height was high while its compaction was low. High SV and low PD value of N suspension (Table 3) are also confirming this result. According to Stoke's law, the sedimentation rates of larger particles are faster than smaller ones. A greater sedimentation height was seen within a short time in the case of N suspension with many agglomerated particles resulting in a fast formation of sedimentation including large pores (about 1-2µm). It may be concluded that particles had no opportunity to settle down in the desired site; they settled down rapidly and randomly on other particles/agglomerates.

The upper layer of N sedimentation after resting for three days is displayed in fig. 9b. These particles had remained suspending in the media for three days and settled down slowly. The upper layer has smaller particles (about  $0.4\mu m$ ) than the bottom layer (about  $3\mu m$ ). Higher compaction density can be seen in the sediment after layer in response to their lower sedimentation rate.

SEM photographs of settled agglomerates resulting from the F suspension are demonstrated in fig. 9c and d. It can be seen that the particles of this stable suspension have been separated from each other and completely dispersed.



**Figure 9.** SEM images of the (a) bottom layer of N suspension, (b) upper layer of N suspension, (c) bottom layer of F suspension and (d) upper layer of F suspension

In this suspension, agglomerates are small and include a few particles. It can also be observed that particles are very well dispersed and compacted, and indeed it is hard to distinguish the agglomerates. Large pores do not exist here; low sedimentation height of this suspension also shows the stability of particles, which did not settle immediately. Fig. 9d illustrates the upper layer of sedimentation including the completely dispersed particles with their original size. The estimated particle size has been about 200nm, which is settled after 3 days.

#### 4. CONCLUDING REMARKS

In this study, YSZ aqueous suspensions were prepared by a common dispersant at various pH values. Accordingly, the sedimentation height, rheological behavior, and zeta potential of suspensions were evaluated and compared. Sedimentation height tests revealed that the optimum percentage of dispersant was as much as 0.1wt% Dolapix CE64. The suspensions prepared by the optimum percentage of dispersant had small sediment particles and minimum viscosity. Moreover, the sedimentation tests indicated that the most stable suspension could be obtained at pH 4 while nano YSZ suspensions were instable at basic pH values. Viscosity, sedimentation parameters, and zeta potential of suspensions revealed that the suspension prepared by adding 0.1wt% Dolapix CE64 at pH=4 was the most stable one, which had a zeta potential value of -25mV and minimum viscosity. Investing the suspension sediment microstructure revealed that the particles of the stable suspension were separated from each other and completely dispersed. Although particles tend to attach each other and form agglomerations in YSZ suspensions, there was not any agglomeration in SEM photographs of the stable suspension.

#### 5. ACKNOWLEDGMENTS

The author would like to appreciate the Materials and Energy Research Centre (MERC) for providing the financial support throughout the project.

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