



**SYNTHESIS OF 3YSZ
NANOPOWDERS BY CO-
PRECIPITATION METHOD AND
INVESTIGATION OF INFLUENCE OF
PROPANOL ON AGGLOMERATION
STATE**

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Abstract

Partially stabilized zirconia is known for its superior mechanical properties such as high hardness and toughness. Partially stabilized zirconia are prepared by adding stabilizers such as CaO, MgO, Y₂O₃ and CeO₂ in monoclinic zirconia. However, due to aesthetic application for dental prosthesis, yttria partially stabilized zirconia

have been of great interest. The synthesis of 3YSZ by coprecipitation process that is washed with water resulted in agglomerated nanopowders that inhibit sinterability. To enhance sinterability the nanopowders need to be deagglomerated. Also it has been reported that washing of water washed coprecipitated mass of yttrium-zirconium hydroxide with solvents such as methanol, ethanol, propanol and butanol reduces the extent of agglomeration. In addition to the washing medium, drying temperature also significantly influences the agglomeration state of YSZ. Already it has been worked on the agglomeration state of 3YSZ nanopowders washed with ethanol. Hence an attempt has been made to wash with varying amount of propanol to reduce the agglomeration state of 3YSZ nanopowders.

Influence of propanol treatment of coprecipitated yttrium-zirconium hydroxide on agglomeration of calcined nano 3YSZ powder was studied. Nanocrystalline 3YSZ (3 mol% yttria stabilized zirconia) powders

were synthesized through co-precipitation followed by washing with propanol and calcination at different temperatures in the range of 100°–500 °C. Results indicated that the amount of propanol used in washing the zirconium hydroxide precipitate had a significant impact on the calcined powder characteristics. Different ratios of propanol to the water contained within the precipitate were used during washing and the calcined nano 3YSZ powders were characterized in terms of tap density, green density and FTIR(Fourier Transform Infrared Spectroscopy). The other characterization techniques such as X-ray diffraction(XRD), Thermo-gravimetric-Differential thermal analysis(TG-DTA), Transmission electron microscope(TEM) has been also carried. The results indicated that a minimum propanol amount of 2–3 times that of water in the precipitate was required to achieve superior agglomeration state of the calcined 3YSZ nanopowders. The nanopowders obtained by washing with optimum propanol amount resulted in high green density of compact and lower tap density with high volume occupancy of nanopowders when tapped. Agglomeration state of the calcined 3YSZ nanopowders attainment with varying volume% of powder characteristics was studied. The results revealed influence of varying amount of propanol on 3YSZ nanopowders reduced the agglomeration state of nanoparticles. The 1:4 ratio of 3YSZ precipitate washed with propanol amount

showed the good agglomeration state of nanoparticles.

Keywords: 3YSZ(3mol% yttria stabilized zirconia), Co-precipitation method, Agglomeration, Temperature, Powders

1. Introduction:

Zirconia ceramics have received a lot of attention from researchers due to their unique properties associated with different crystalline forms. Zirconia stabilized in tetragonal form is suited for structural applications owing to its high fracture toughness, strength whereas zirconia stabilized in cubic form has been used in fuel cells, sensors etc. because of its high oxygen ion conductivity and phase stability[1-38]. Besides the inherently high fracture toughness of the tetragonal phase, higher strengths in tetragonal zirconia have been achieved by producing submicrometer size grains in highly dense sintered ceramics. To achieve this, the starting powder particles have to be in the nanoscale (<100 nm) so that on consolidation and sintering they attain high densification with submicrometer grain size. Over the years many approaches have been developed for synthesis of partially or fully stabilized nanocrystalline zirconia ceramics but controlling the state agglomeration in nanocrystalline powders has remained a challenge. The extent of agglomeration in nanoceramic materials has been seen to be a function of the synthesis route including the temperature of calcinations of amorphous

intermediates which result in crystallization. Hydrothermal synthesis typically does not involve high temperature calcinations and thus results in lower extent of agglomeration of particles[1-11]. Zirconia is a white powdered material commonly used to produce dental frameworks for dental substructures such as crowns, bridges, etc. Unlike standard ceramics that tend to be brittle and hard, Zirconia has excellent wear resistance and strength, and comes with a flexibility which is far better than those of other technical ceramics. Pure zirconium dioxide undergoes a phase transformation from monoclinic (stable at room temperature) to tetragonal (at about 1173 °C) and then to cubic (at about 2370 °C), according to the scheme(Figure 1).

Monoclinic(1173 °C)→Tetragonal(2370 °C)
→ Cubic (2680 °C)→ Melt

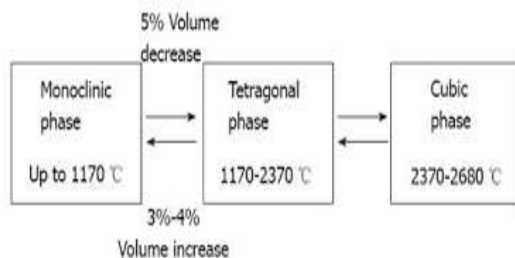


Figure 1:Phase transformation of Zirconia

Nanocrystalline zirconia has been synthesized by various chemical routes but among them coprecipitation method has been popular as it can be scaled up,using

low-cost accessories. In this method, the step of driving away water from coprecipitated mass, forces the particles to agglomerate. The follow-up step of high temperature treatment (crystallization) leads to further enhancement and strengthening of agglomerates.[12-35].Heuer et al.[36] reported that during drying of the coprecipitated mass, high surface tension of water (73 mN/m) is responsible for bringing the zirconium hydroxide nanoparticles closer resulting in hard agglomerates. If ethanol is used for surface treatment of zirconium hydroxide precipitate, the extent of agglomeration reduces considerably because of its low surface tension (24 mN/m). On drying, particles are not forced together and the hydroxyl bridging as encountered in case of water-washed powder is missing and hence result in lower degree of agglomeration.[39-53].Wang et al.[48] reported the synthesis of zirconia with 80% of ethanol added after dissolution of $ZrOCl_2 \cdot 8H_2O$ in minimum water to get rid of agglomeration problem to a considerable extent. In his work 12 10 mol% $MgO-ZrO_2$ was synthesized by coprecipitation by using mixed solvent (water and ethanol) and pure ethanol. In both cases the final precipitate was washed with absolute ethanol. Initial studies by the present authors while confirming the prior observations that ethanol washing contributes to deagglomeration suggested that amount of ethanol used in washing of coprecipitated mass may have a role in

determining calcined (crystalline) powder characteristics, however, the aspect of washing with Propanol as not been reported in the literature. H.G.Scott et.al[33] worked on Phase relationships in the zirconia—yttria system wherein the

Metastable and Equilibrium phase relationships in the system $ZrO_2:YO_{1.5}$ have been studied by X-ray diffraction. The conditions for the retention of a zirconia-rich tetragonal phase at ambient temperature are established. The existence of a miscibility gap, closed below the solidus temperature, in the yttria-rich solid solution region is proposed. Some evidence for partially ordered phases is presented. Mary Sue Kaliszewski & Arthur H. Heuer et.al[38] worked on the Alcohol interaction with zirconia powders wherein the mechanism by which alcohol washing of ceramic powders produces “soft” agglomerates has been investigated by studying the interaction of ethanol with hydrous ZrO_2 powders using Fourier transform infrared (FTIR) spectroscopy.

Unambiguous evidence of ethoxide formation has been found, which apparently prevents bond formation between adjacent particles and thus the formation of “hard” agglomerates

2.Experimental Method: Co-Precipitation Method is used for synthesis, it is a phenomenon where a solute that would normally remain dissolved in a solution precipitates out on a carrier that forces it to bind together, rather than remaining

dispersed. The simultaneous precipitation of more than one component (Figure 2).

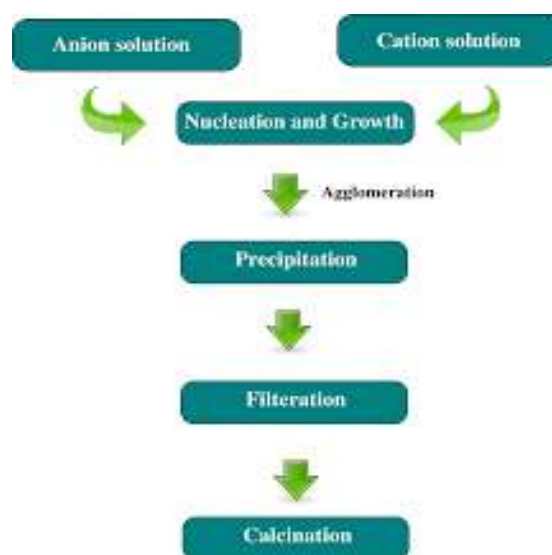


Figure 2: Flow chart of Co-precipitation method

Materials used:

- Zirconium Oxychloride (Mincometsal, Bangalore) (99.9% purity)
- Yttrium nitrate (99.9% purity)
- Ammonia (25% concentrated)
- Distilled water
- 2propanol (99.9% purity (GC), Water (≤ 0.5%))

Synthesis Process: Synthesis of 3 mol% yttria-stabilized zirconia was carried out by reverse-strike coprecipitation method followed by drying and calcination of the coprecipitated mass at temperatures that is, 100°C to 500°C by varying volume%.

Precursor solutions of 0.5 M zirconium oxychloride were prepared in double distilled water using magnetic stirrer. Yttrium nitrate solution was also prepared. Zirconium and yttrium-containing precursor solution was prepared by adding an appropriate amount of yttrium nitrate solution to zirconium oxychloride solution (1:1 ratio) to achieve 3 mol% yttria with zirconia in the calcined mass. The prepared precursor solution was added drop wise into a continuously stirred ammonia solution ensuring the pH remained above 10 till complete addition of precursor solution to ammonia. Stirring was continued for 2 h after complete addition of precursor solution into ammonia. The precipitate was left overnight to settle (~12 h) and then filtered on fabric cloth for the removal of excess ammonia. This precipitate was subjected to washing with distilled water by addition of measured quantity of water and stirring for 30 min. The precipitate was left to settle for 3 h and then filtered on fabric cloth.

The washing procedure was repeated seven times till Cl^- ion was completely removed from the precipitate. The absence of Cl^- ion was confirmed by absence of AgCl precipitate up on addition of 0.1 N AgNO_3 solution to the filtrate. Water-washed precipitate was divided into seven parts. One part was taken as it is and the crystalline powder obtained on its calcination is referred to as waterwashed powder. The other six parts of the precipitate were washed with different amounts of propanol. Quantity of propanol was chosen in

reference to the quantity of water present in the precipitate. Water associated with the washed and filtered coprecipitated mass was determined by drying known quantity of coprecipitated mass at 100°C . Following the last wash and after having been kept on the filter cloth for 12 h, the amount of water in the precipitate was reproducibly seen to be around 93% of the filtered coprecipitated mass. During washing and filtration of the precipitate the conditions in the entire study were maintained such that greater than 90% water was retained in the precipitate to facilitate its mixing in propanol and displacement of water with propanol. Propanol amount used for washing the precipitate was varied in the ratio of 1:1, 1:2, 1:3, 1:4, and 1:5 (water: propanol) with respect to the water associated with the precipitate and calcined at temperatures 100°C , 200°C , 300°C , 400°C and 500°C (Figure 3).

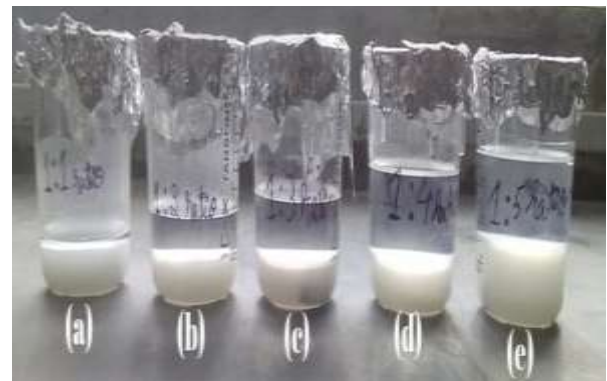


Figure 3 :3YSZ precipitate washed with propanol (a)1:1 ratio,(b)1:2 ratio,(c)1:3ratio,(d)1:4 ratio and (e)1:5 ratio

(the propanol amount used for washing the precipitate was varied with the varying volume% with respect to the water associated with the precipitate)

The precipitate was washed with each of the propanol amount twice followed by drying at 100°C. The dried precipitate was calcined at 200°C ,300°C ,400°C and 500°C separately for each of the powders washed with different propanol amounts. In the remaining text, zirconium hydroxide precipitate refers to the precipitate with composition expected to yield crystalline 3 mol% yttria stabilized zirconia (3YSZ) upon calcination. Also, water-washed precipitate refers to chloride-free precipitate. Water-washed and propanol-washed powders refer to crystalline 3YSZ powder obtained by calcination of water-washed and propanol-washed precipitate.

Results and discussion:

1.XRD(X-ray diffraction analysis):

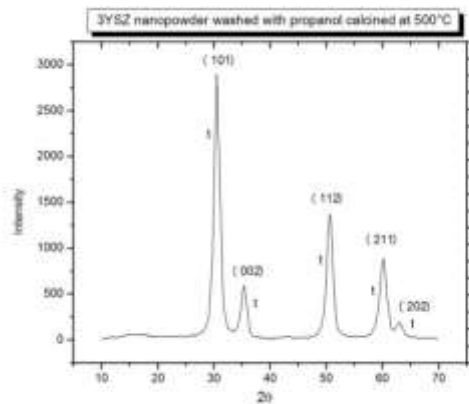


Figure 4 :XRD patterns for 3YSZ nanocrystalline powders wherein the precipitate was washed with propanol that was calcined at 500°C

All the 3mol% YSZ samples prepared in the study predominantly are of tetragonal as shown in figure above 4.The miller indices of 3YSZ nanocrystalline powders wherein the precipitate washed with propanol calcined at 500°C are (1 0 1),(0 0 2),(1 1 2), (211) and (2 0 2).This miller indices pertains to tetragonal 3YSZ structure wherein no monoclinic phase observed in the XRD.

2.TGA-DTA(Thermogravimetric Analysis-Differential thermal Analysis):

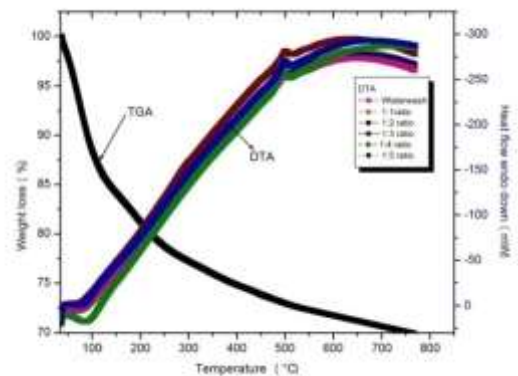


Figure 5.:TGA-DTA of precipitate that was washed with water followed by propanol washing (1:0,(Waterwash)1:1,1:2,1:3,1:4 and 1:5 water:propanol ratio) calcined at 100°C

The TGA-DTA carried for 3YSZ nanocrystalline powders produced by Propanol waterwash(1:0,1:1, 1:2,1:3,1:4 &

1:5 (water to propanol ratio)) that were calcined at 100°C and 500°C. The TGA-DTA graphs explain about exothermic peak in figure 5. TGA-DTA showed the presence of amorphous and attainment of crystalline peaks for 100°C wherein for 1:0 ratio (water wash) = 498.30°C, 1:1 ratio = 499.44°C, 1:2 ratio = 499.59°C, 1:3 ratio = 498.08°C, 1:4 ratio = 493.69°C and 1:5 ratio = 498.62°C with fast rate of heating from 40°C to 750°C at 20°C/min and TGA-DTA also carried for powders calcined at 500°C. The adsorbed propanol evaporates at 82.6°C with autoignition temperature of 399°C. Hence there are no exothermic peaks for the powders calcined at 500°C. The adsorbed propanol cannot be detected in 500°C calcined powders.

3. TEM (Transmission Electron Microscopy):

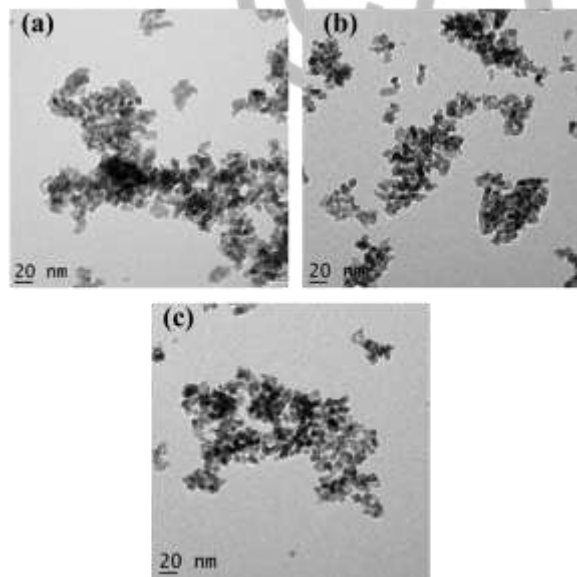


Figure 6: TEM image of 3YSZ nanocrystalline powders calcined at 500°C

powder produced by washing with propanol (a) 1:2 ratio, (b) 1:4 ratio and (c) 1:5 ratio

The TEM micrographs are for 3YSZ powders calcined at 500°C. These powders were washed with propanol. The propanol used for washing is varied with water:propanol ratio in 1:0 (water wash), 1:1, 1:2, 1:3, 1:4 and 1:5. The TEM micrographs showed for calcined powder (figure 6) for 1:2, 1:4 and 1:5 ratio. The TEM shown in figure 6 reveals that the size of primary particles are in the range of 5-20 nm.

4. CHN analysis (Carbon, Hydrogen and Nitrogen): The CHN analysis is carried for to detect the presence of %C and %H in the samples. As the work is done on 3YSZ nanopowder washed with varying volume% of propanol at different calcinations temperatures. The CHN analysis is done for 3YSZ nanopowder washed with varying volume% of propanol calcined at 100°C. The propanol structure is as shown below:

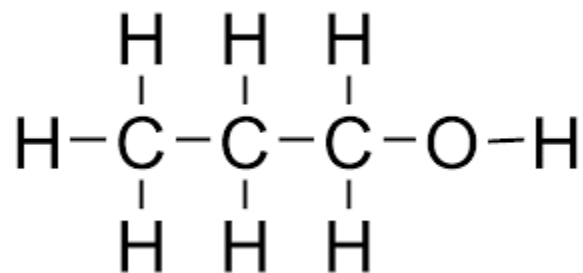


Figure 7: Structure of 2-propanol

The 2-propanol contains the Carbon and Hydrogen due to which the presence of adsorbed propanol on the 3YSZ nanopowders washed with varying volume%

amount of propanol calcined at 100°C is calculated with CHN analysis. The CHN analysis for agglomeration state of nanoparticles is discussed.

| Sample No | Sample Name | C% | H% | Sample Weight, mg |
|-----------|--------------------------|------|------|-------------------|
| 1 | 100 Degree Waterwash PPT | 1.24 | 0.89 | 6.06 |
| 2 | 100 Degree Propanol1-1 | 1.07 | 1.26 | 6.78 |
| 3 | 100 Degree Propanol1-2 | 1.52 | 1.52 | 7.10 |
| 4 | 100 Degree Propanol1-3 | 1.34 | 1.53 | 6.73 |
| 5 | 100 Degree Propanol1-4 | 1.02 | 1.86 | 7.16 |
| 6 | 100 Degree Propanol1-5 | 1.18 | 1.67 | 6.71 |

Table 1:CHN Analysis results for samples

The Table 1 listed above explains about CHN analysis. The adsorption of propanol on 3YSZ precipitate calcined at 100°C samples carried out. Due to varying weight of samples taken by STICcochin wherein the presence of C% and H% cannot be attained for agglomeration state of nanoparticles. The %C and %H shows adsorbed propanol on 3YSZ precipitate washed with it. The CHN analysis results varied with 3YSZ precipitate washed with varying volume% of propanol was not appreciable. This proves the fact that tap density and green density is to be carried out for achieving best possible

degree of agglomeration state of nanoparticles. The samples are of calcined at 100°C.

5. Tap Density: Tap density is obtained by mechanically tapping a graduated cylinder containing the sample until little further volume change is observed. Tap density for 3YSZ nanopowders calcined at 100°C is discussed.



Figure 8: Volume occupied upon tapping by 3YSZ powders calcined at 100°C wherein the precipitate were washed with water and different amounts of propanol; Water-washed powder (a) and propanol-washed powders (b) 1:1 (c) 1:2 (d) 1:3 (e) 1:4 & (f) 1:5 water to propanol amount ratio.

Table 2: Tap density of 3YSZ nanopowders by the coprecipitated mass washed with varying volume% propanol calcined at 100°C.

| Sl.no | Water:Propanol Ratio | Tap Density(g/cc) |
|-------|----------------------|-------------------|
| 1. | Waterwash(1:0) | 1.041 |
| 2. | 1:1 | 0.86 |
| 3. | 1:2 | 0.83 |
| 4. | 1:3 | 0.71 |
| 5. | 1:4 | 0.55 |
| 6. | 1:5 | 0.55 |

It is known that agglomerates of different sizes lead to different extents of packing. The differences in packing behavior of water washed powder and powders washed with different propanol amounts were characterized by measuring tap density. The tap density measurements revealed that the powder obtained by calcination of water-washed precipitate occupied the least volume (shown in Figure 8) and hence highest apparent density of 1.041g/cc due to presence of large dense aggregates. For 3YSZ nanopowders washed with varying volume% propanol at 1:1ratio,1:2ratio,1:3ratio,1:4ratio and 1:5 ratio (water of precipitate to propanol) ratio the tap density decreased to 0.86g/cc,0.83g/cc,0.71g/cc,0.55g/cc and 0.55 g/cc respectively. This decrease in tap density indicated that increase in propanol amount in washing of the precipitate results in a fluffy mass and hence increased volume occupancy (refer Figure 8) and lower tap density. On increasing the propanol amount for washing of the precipitate to 1:5 water to propanol ratio the calcined powder occupied larger volume and thus had the least tap

density of 0.55g/cc suggesting presence of voids greater than 80%. For further increase in propanol amount while washing the precipitate the calcined powder volume (tapped) remained same. These observations indicated presence of larger, denser agglomerates in water washed powders and smaller agglomerates in powders obtained by washing with 2.5 times or greater propanol amount (Table 2).

6.Green density: The green density is of before sintering stage. Green density values for 3YSZ nanopowders by the coprecipitated mass washed with varying volume% propanol calcined at 100°C is discussed.

Table 3: Green density of 3YSZ nanopowders by the coprecipitated mass washed with varying volume% propanol calcined at 100°C

| Sl.no | Water:Propanol Ratio | Green density(% TD) | Green density(g/cc) |
|-------|----------------------|---------------------|---------------------|
| 1. | Waterwash(1:0) | 36 | 2.18 |
| 2. | 1:1 | 36 | 2.19 |
| 3. | 1:2 | 39 | 2.36 |
| 4. | 1:3 | 40 | 2.42 |
| 5. | 1:4 | 41 | 2.44 |
| 6. | 1:5 | 41 | 2.44 |

The powders that were calcined at different temperatures were compacted uniaxially at 200MPa. Pellets are produced by without any binders with the identical weight of 1gm(Figure 9).

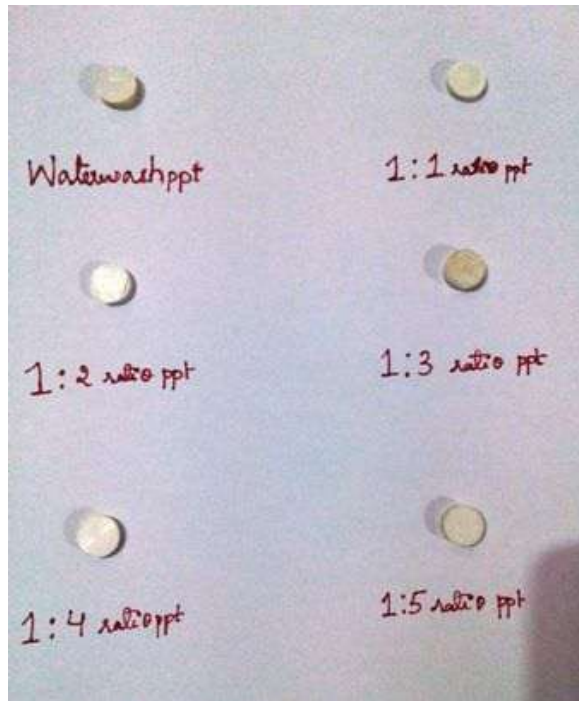


Figure 9: Pellets produced from 3YSZ nanopowders by the coprecipitated mass washed with varying volume% propanol calcined at 100°C on hydraulic press machine

The pellets are of 3YSZ nanopowders produced by the coprecipitated mass 1:0 (Waterwash), 1:1, 1:2, 1:3, 1:4 and 1:5 calcined at 100°C. Hence the green density is listed in above table 3. The green density goes on increasing and gets stable after 1:4 as with the volume variation the identical weight of 1gm taken. From table it shows that the green density is increasing and getting stable after 1:4 precipitate washed

with propanol. It is worth to note that the propanol washed powders which had the least tap density showed the highest green density (41% of Theoretical density) when powders compacted uniaxially at 200MPa. The waterwashed powder with highest tap density showed the lowest green density in the compacted state (36% Theoretical density). From green density values the 1:4 is the best for agglomeration state of nanoparticles.

7. FTIR (Fourier Transform Infrared Spectroscopy) Analysis:

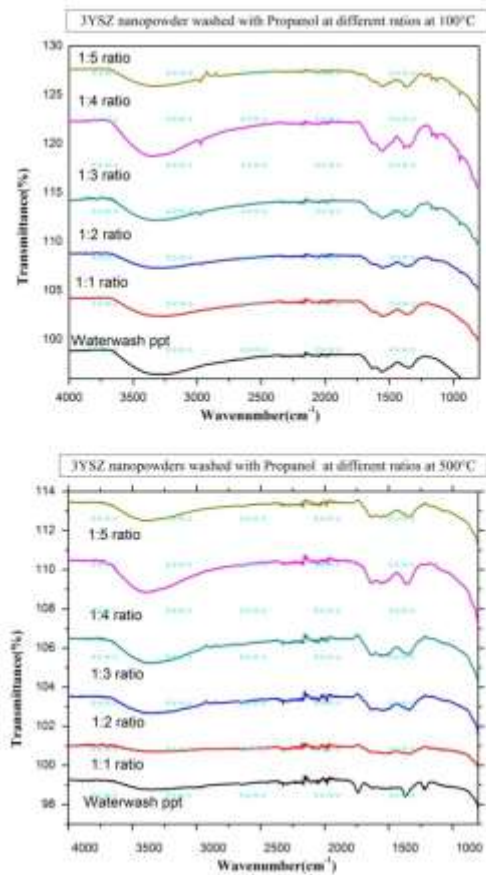


Figure 10: FTIR graph of 3YSZ nanopowder washed with propanol calcined at 100°C and 500°C (a) 1:0 ratio (Water Wash), (b) 1:1

ratio,(c)1:2 ratio,(d)1:3 ratio,(e)1:4 ratio and (f)1:5 ratio

The FTIR of 3YSZ nanopowders washed with propanol calcined at 100°C and 500°C is studied. The some amount of chemisorbed propanol on the 3YSZ precipitate washed with varying volume% of propanol is studied. The Table listed below 7.6 explains about functional group with stretch.

Table 4: The transmittance with functional group occurred in 3YSZ nanopowder washed with varying volume% of propanol calcined at 100°C and 500°C.

| Sl.no | Transmittion(cm^{-1}) | Functional group |
|-------|----------------------------------|------------------|
| 1. | 3000-3800 | O-H |
| 2. | 3000-2900 | C-H |
| 3. | 1580-1080 | C-O |
| 4. | 1160-1070 | C-O |
| 5. | 1250-900 | C-H |

FTIR spectra of 3YSZ nanopowders by the coprecipitated mass washed with varying volume% propanol calcined at 100°C and 500°C obtained as shown in figure 7.6. The table 7.6 explains about functional group stretch and transmittance(%) at (3800cm^{-1} - 3000cm^{-1}) are due to O-H stretch, the two peaks at (3000cm^{-1} - 2900cm^{-1}) and (1250cm^{-1} - 900cm^{-1}) are due to C-H stretch and the two peaks at (1160cm^{-1} - 1070cm^{-1}) and (1580cm^{-1} - 1080cm^{-1}) are due to C-O stretch due to some amount of chemisorbed propanol. The figure shows the bonds of the 3YSZ precipitate washed with varying volume% of propanol [35 and 43]. From figure 10 the clear indication of O-H stretch, C-H stretch and C-O stretch can be

notified as the nanopowders are calcined at 100°C (other propanol and propoxide peaks cannot be resolved because of overlap with water and carbon dioxide bands). There is only presence of O-H stretch and C-O stretch in 3YSZ nanopowders washed with varying volume % propanol calcined at 500°C. Due to evaporation of propanol at 82.6°C the chemisorbed propanol on 3YSZ nanopowders calcined at 500°C, the C-H stretch cannot be detected in 3YSZ nanopowders washed with varying volume % of propanol calcined at 500°C as shown in figure 10. So, from graphs it clearly indicates that there is presence of organic compounds, C-H stretch present in propanol adsorbed on Zr-OH (Zirconium hydroxide precipitate), this shows the agglomeration state of nanoparticles.

CONCLUSION & FUTURE SCOPE

Agglomeration state of nanoparticles attained with steady volume in calcined nano YSZ powders improved with increase in amount of propanol used for washing of the yttrium-zirconium hydroxide precipitate. Presence of adsorbed propanol on the yttrium-zirconium hydroxide was confirmed through Tap density, Green density and FTIR (Fourier Transform infrared spectroscopy) has been evaluated. The water present in the precipitate was required to reduce extent of agglomeration considerably in the calcined powders at 100°C and 500°C are studied.

XRD study showed that the samples are of tetragonal phase as with of JCPDS (Joint Committee of Powder Diffraction Society)

in with respective Miller indices(101),(202),(112),(211) and (202) which shows the tetragonal phase of 3YSZ, there is no presence of monoclinic phase. TGA-DTA showed the presence of amorphous and attainment of crystalline at peaks for 100°C wherein for 1:0(waterwash)=498.30°C, 1:1=499.44°C, 1:2=499.59°C, 1:3=498.08°C, 1:4=493.69°C and 1:5=498.62°C with fast rate of heating from 40°C to 750°C at 20°C/min and TGA-DTA also carried for powders calcined at 500°C there are no exothermic peaks observed due to evaporation of propanol as it evaporates at 82.6°C with autoignition temperature of 399°C. The adsorbed propanol didn't detected in 500°C calcined powders. TEM results showed the morphology and particle size of Zirconia nanoparticles with respect to agglomeration state of nanoparticles. It can be seen from TEM images that particle size increased with increase in varying volume % of propanol when added. CHN analysis showed the C%, H% & N% in the nanopowders, wherein varying sample weights of powders of 100°C didn't attained the deagglomeration of nanoparticles with varying volume% of propanol influence. The Tap Density showed the best when there is increase in flow character for nanopowders washed with propanol at 1:0 ratio(waterwash), 1:1 ratio, 1:2 ratio, 1:3 ratio, 1:4 ratio and 1:5 ratio(water of precipitate to propanol ratio), the tap density decreased and increase in volume occupancy observed as

1.041g/cc, 0.86g/cc, 0.83g/cc, 0.71g/cc, 0.55g/cc and 0.55 g/cc respectively. This decrease in tap density indicated that increase in varying volume % of propanol amount in washing of the precipitate results in a fluffy mass and hence increased volume occupancy and lower tap density. On increasing the propanol amount for washing of the precipitate to 1:4 water to propanol ratio the calcined powder occupied larger volume and thus had the accurate tap density of 0.5568 g/cc suggesting presence of voids greater than 80%. For further increase in propanol amount while washing the precipitate the calcined powder volume (tapped) remained same. These observations indicated presence of larger, denser agglomerates in water washed powders and smaller agglomerates in powders obtained by washing with 2.5 times or greater propanol amount. The Green density shows the superior particle packing as there is increase in the green density and getting stabled from the results. The green density(% Theoretical density by taking 6.06g/cc) and green density(g/cc) for 1:0(Water wash) is 36(%TD) and 2.18g/cc, 1:1 washed with propanol ppt is 36(%TD) and 2.19g/cc, 1:2 washed with propanol ppt is 39(%TD) and 2.36g/cc, 1:3 washed with propanol ppt is 40(%TD) and 2.42g/cc, 1:4 washed with propanol ppt is 41(%TD) and 2.44(g/cc) and for 1:5 washed with propanol ppt is 31(%TD) and 2.44g/cc carried for 100°C calcined powders uniaxially compacted at 200MPa. Hence nanopowder washed with varying volume%

of propanol shows the agglomeration state of nanoparticles in green compacts. The green density goes on increasing and gets stable after 1:4 as with the volume variation the identical weight of 1gm taken. Hence from this 1:4 washed ppt with propanol is the best for agglomeration state of nanoparticles. These observations of tap density and green density pointed towards the role of propanol amount used in washing the precipitate on influencing calcined powder characteristics.

FTIR spectra of 3YSZ nanopowders by the coprecipitated mass washed with varying volume% propanol calcined at 100°C and 500°C. The functional group stretch and transmittance(%) at (3800 cm^{-1} -3000 cm^{-1}) are due to O-H stretch, the two peaks at (3000 cm^{-1} -2900 cm^{-1}) and (1250 cm^{-1} -900 cm^{-1}) are due to C-H stretch and the two peaks at (1160 cm^{-1} -1070 cm^{-1}) and (1580 cm^{-1} -1080 cm^{-1}) are due to C-O stretch due to some amount of chemisorbed propanol. The figure shows the bonds of the 3YSZ precipitate washed with varying volume% of propanol. The clear indication of O-H stretch, C-H stretch and C-O stretch can be notified as the nanopowders are calcined at 100°C (other propanol and propoxide peaks cannot be resolved because of overlap with water and carbondioxide bands). There is only presence of O-H stretch and C-O stretch in 3YSZ nanopowders washed with varying volume % propanol calcined at 500°C. Due to evaporation of propanol at 82.6°C the chemisorbed propanol on 3YSZ

nanopowders calcined at 500°C, the C-H stretch cannot be detected in 3YSZ nanopowders washed with varying volume % of propanol calcined at 500°C. So, from graphs it clearly indicates that there is presence of organic compounds, C-H stretch present in propanol adsorbed on Zr-OH (Zirconium hydroxide precipitate), this shows the agglomeration state of nanoparticles.

Hence 3mol% partially YSZ agglomerated state of nanoparticles synthesized by coprecipitation method with influence of propanol volume variation of powder characteristics.

Further the composites of Zirconia as a matrix can also be prepared & the results such as Tensile, Impact, Hardness, Wear Studies can be carried out.

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