

## **Investigation of processing time and molecule size nano molecule from $\alpha$ -Al<sub>2</sub>O<sub>3</sub>**

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### **Abstract**

*In this paper, ball milling of Industrial Alumina ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) powder was studied with varied milling time. For reducing the size of the particle Ball milling top down approach is adopted. The average size of micro level alumina was 70  $\mu$ m. The conversion was carried out by grinding process at two different stations for different time periods. Rotational speed, balls to powder ratio, water to powder ratio and milling time are the parameters included in this study. The scanning electron microscopy (SEM) result of the sample which was carried out by Insmart System for 120 hours ground materials indicates non contaminated particle but, did not give any clear picture of size. The grinding work by ball milling was carried out for 10 hours after rinsing of the jar with alumina. The result indicates that the size of Alumina is reduced from 70  $\mu$ m to 1.4  $\mu$ m with grey color agglomeration particles.*

**Keywords:** Alumina powder, ball milling, particle size reduction, scanning electron microscopy, transmission electron microscopy

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### **I. INTRODUCTION**

Nanotechnology is considered to be a multidisciplinary and an interdisciplinary area of Research and development. The wide-ranging of applications that nanotechnology is and will be catering to speaks of its omnipresence. Nanotechnology finds a defining role to play in the field of agriculture, energy, electronics, medicine, healthcare, textiles, transport, construction, cosmetics, water treatment etc., as suggested by many researchers worldwide [1]. In this context, a particle is defined as a small object that behaves as a complete unit with respect to its transport and properties. Nanoparticles in nanofluids are made of metals, oxides, carbides or carbon nano tubes and the size ranging from 1 to 100 nanometer (10<sup>-9</sup> mm). The materials include in nanoparticles are namely, oxide ceramics (Al<sub>2</sub>O<sub>3</sub>, CuO), Metal carbides (SiC), Nitrides (AlN, SiN), Metals (Al, Cu), Non-metals (Graphite, Carbon nanotubes), Layered (Al + Al<sub>2</sub>O<sub>3</sub>, Cu + C). A nanofluid are the new class engineered fluid with high thermal conductivity obtained by suspending nanometer size (1-100nm) particles in a base fluid like water, Ethylene- or tri- ethylene-glycols and other coolants, Oil and other lubricants, Bio-fluids and Polymer solutions. In other words, nanofluids are nanoscale colloidal suspensions containing condensed nanomaterials and it is agglomerate-free stable suspension for long durations without causing any chemical changes in the base fluid [2]. Solid and liquid phase are the two phases in the system. Nanofluid have been found to possess boosted thermo physical properties for instance thermal conductivity, thermal diffusivity, viscosity, and convective heat transfer coefficients compared to those of base fluids like water or oil.[3]. Nanoparticles suspended in different base fluids can alter the momentum and heat transfer features of the velocity and thermal boundary layers by significantly increasing the liquid viscosity and thermal conductivity[4]. Owing to the outstanding features of nanofluids, it finds wide applications in enhancing heat transfer. Most engineering products brought about from nanotechnology will be imperilled to heat transfer analysis, as thermal considerations have always been an essential part of any new design. Researchers realized that the obtainable macroscale data were inadequate to forecast fluid flow and heat transfer characteristics in heat exchangers and many engineering devices. Cooling plays a significant role in most of the engineering devices. Conventional methods adopted were providing fins to increase the surface area, increase pump power for increasing the flow rates. Also conventional heat transfer fluid like engine oil, ethylene glycol and water have naturally poor thermal conductivity compared to solids and also conventional fluid do not work with the emerging "miniaturized" knowledge due to chance of clogging the tiny channels of these devices. However, novel and superior cooling technology is the want of the hour.

Alumina, silicon, aluminum, copper, and silver shows higher magnitude heat transfer rate in increasing order. The thermal conductivity of silver, alumina and engine oil is 429 W/m-K, 40 W/m-K and 0.145 W/m-K correspondingly. Dispersing solid particles in fluids to enhance thermal conductivity give the basic concept of nanofluids. The foremost challenge is the quick settling of these solid particles in fluids. Nanoparticles differs from microparticles by better dispersion behavior, less clogging and abrasion and much larger surface area to

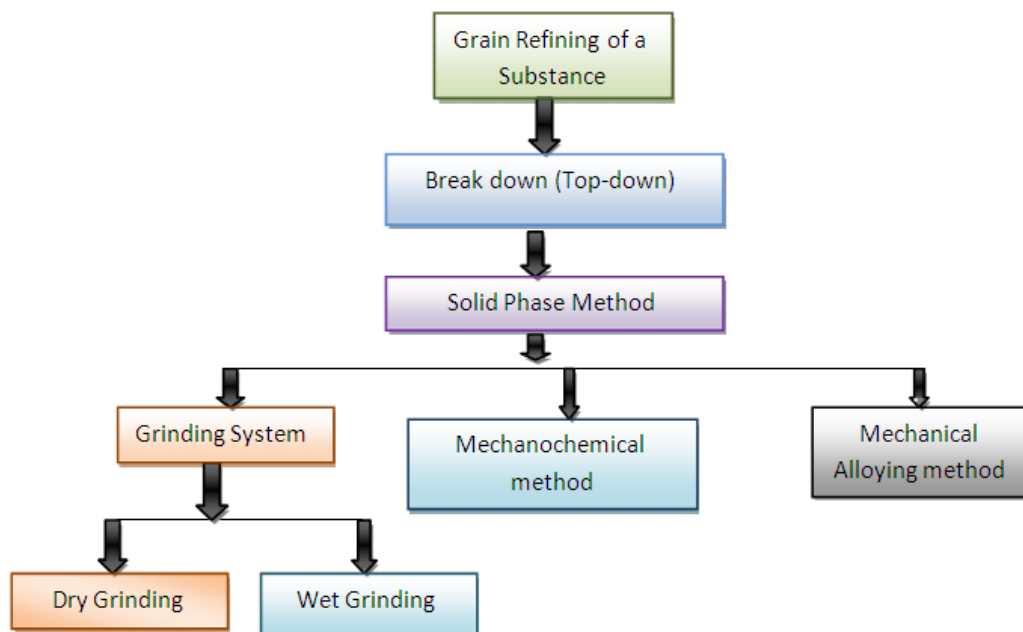
volume ratio.

Theoretical and experimental investigations have been conducted by many researchers for production, preparation and its steadiness. Nanofluids were first originated by Choi and Eastman in 1995 at the Argonne National Laboratory, USA. Since then, there has been rapid development in the synthesis techniques for nanofluids [5]. Sridhara and Sathapathy have abridged the fundamentals of nano fluid, its preparation methods and the factors effecting the thermal conductivity boost in the  $\text{Al}_2\text{O}_3$  base nano fluid [2]. PramodWarrier and ArynTeja concluded that the decrease in the thermal conductivity of the solid with particle size must be considered when developing models for the thermal conductivity of nanofluids.[6] A number of investigations revealed that the nanofluid heat transfer coefficient could also be increased by more than 20% in case of very low nano particles concentrations[7-8]. Das et al. concluded that nanofluids show great potential for use in cooling and the connected technologies. Oxide nanoparticles-based nanofluids are relatively less promising in the enhancement of thermal conductivity of fluids. Also the enhancement diminishes rapidly with the increase in particle size [9]. Researchers discovered that nanofluids has shown numerous distinct properties with large enhancements in thermal conductivity as compared to the base liquid, temperature and particle size dependence reduced friction coefficient , and significant increase in critical heat flux [10-14]. Mishra et al. studied that the thickness of nano fluid depends on many parameters such as base fluids, particle volume fraction, particle size, particle shape, temperature, shear rate, pH value, surfactants, dispersion techniques, particle size distribution and particle accumulation [15].

The current study focused on the conversion of micro alumina to nano alumina by top down approach and wet grinding process, characterization of ground particles and investigation on the effect of grinding time on the characteristics of the particles.

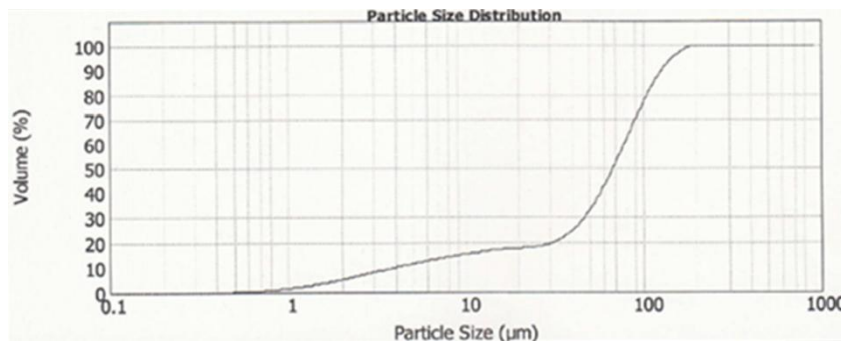
## II. MATERIALS AND METHODS

Top down process is a traditional route scaling down processes acquainted in macro and micro engineering. In top down approaches an outside force is applied to a solid particles which is then break-up into smaller particles. Concerted incremental improvement in the entire manufacturing process transforms precision engineering into ultra precision engineering. The stiffness of the parts of mechanical devices used to form objects is particularly significant. Material is removed by grinding. A characteristic of particles in grain refining process is that their surface energy upturns, which causes the combination of particles to increase. In dry grinding method the solid substance is ground as a result of shock, a compression, or by friction, using such popular methods as a jet mill, hammer mill, a shearing mill, a roller mill, shock shearing mill a ball mill and a tumbling mill. It is tough to obtain particle less than  $3\ \mu\text{m}$  size by means of dry grinding since condensation of small particles takes place simultaneously with pulverization. But, in wet grinding the solid substrate is carried out using a tumbling ball mill, a planetary mill or a vibratory ball mill, a centrifugal fluid mill, an agitating beads mill, a flow conduit beads mills an annular gap bead mill or a wet jet mill. The wet process is appropriate for averting the condensation of the nanoparticles so formed, and thus it is possible to obtain highly dispersed nano particles. A distinctive synthetic method for nanoparticles for the top-down methods is given in Fig.1.



**Fig.1.** A distinctive synthetic method for nanoparticles for the top-down approaches

The average particle size of the raw material is found to be 68.308  $\mu\text{m}$  which has been tested in PSA (Particle Size Analyzer). The tested report is shown in Fig. 2.



**Fig.2.** The average particle size of alpha alumina sample

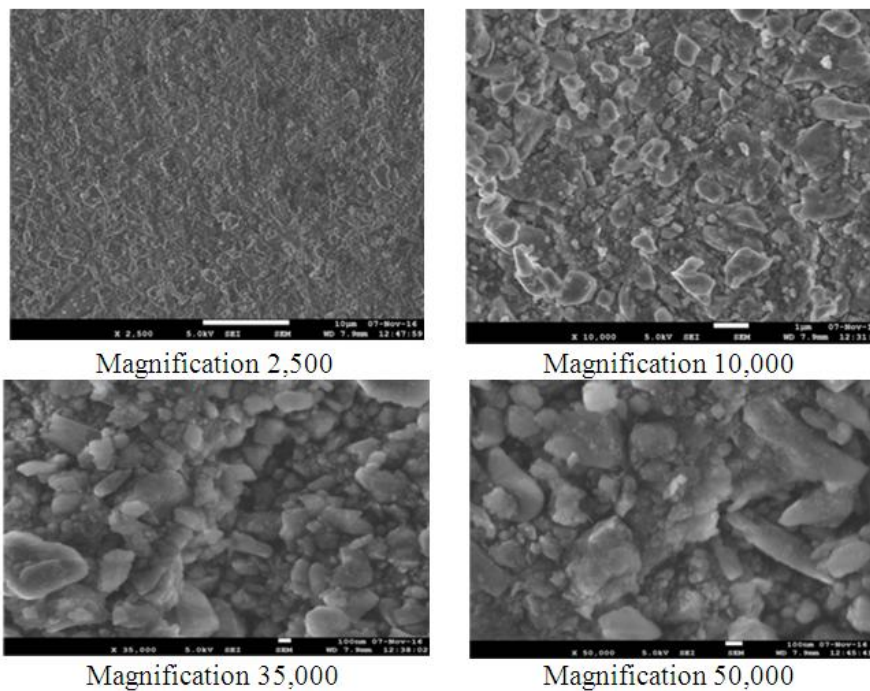
The conversions from micro to nanoparticles were carried out by wet grinding process at two different stations for different time periods.

- Station I
- Station II

### III. RESULTS AND DISCUSSION

#### Station I results

The 1<sup>st</sup> phase of grinding work was carried out for 80 hrs, 120 hrs and 140 hrs. The Field emission - Scanning electron microscope (FE-SEM) and Energy dispersive X-Ray spectroscopy (EDS) tests were carried out in Indian institute of chemical technology (IICT) Hyderabad. FE-SEM data for 120 hours ground sample at different magnification is shown in Fig. 3. EDS result of 120 hours sample is shown in Fig. 4. The FE-SEM data for 140 hours ground sample at different magnification is shown in Fig. 5. EDS result of 140 hours sample is shown in Fig. 6.



**Fig. 3.** FE-SEM data for 120 hours ground sample at different magnification.

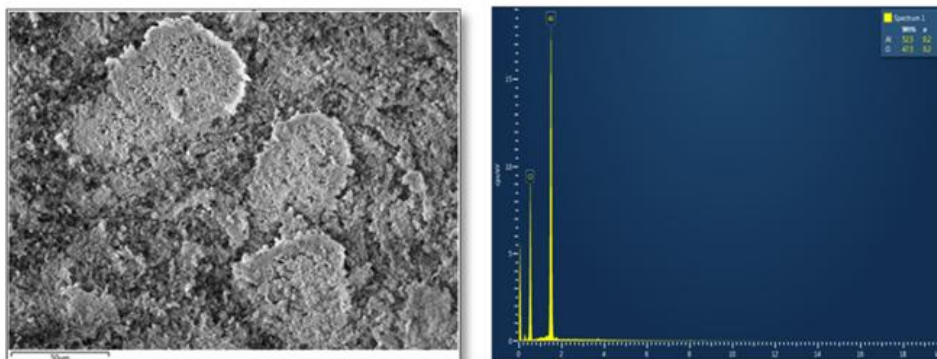


Fig.4. EDS result of 120 hours sample

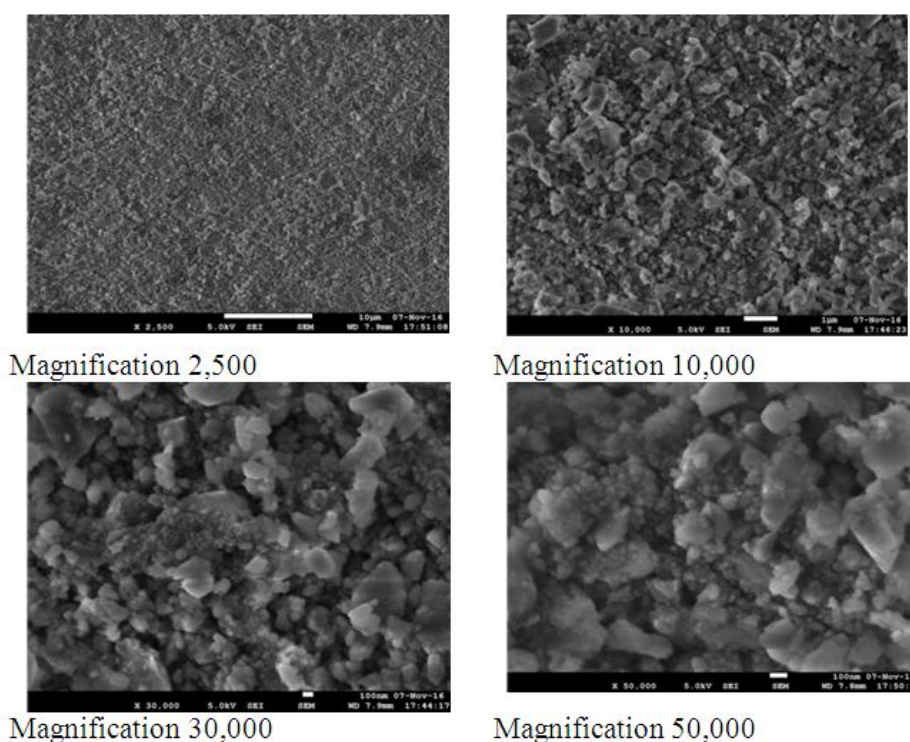


Fig. 5. FE-SEM data for 140 hours ground sample at different magnification.

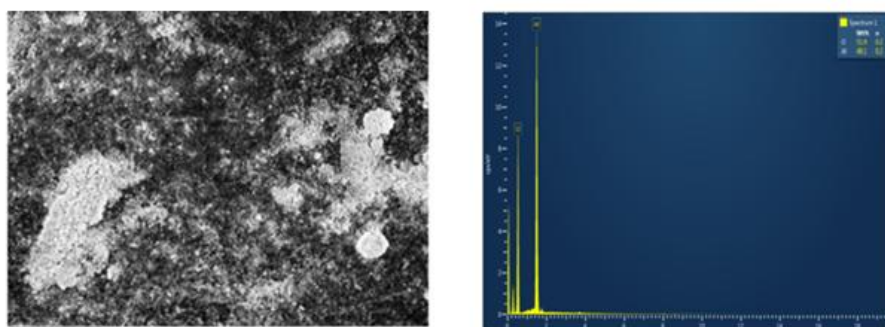


Fig.6. The EDS result of 140 hours sample

Fig. 5 and Fig. 6 indicated that the size of the Alumina is not as per our requirement and is above 100 nm. The ground samples indicates that there was no contamination but does not give any clear picture of size. Thus, the above said sample has been tested for TEM and PSA to get a clear picture of size shown in Fig. 7 and

Fig. 8.

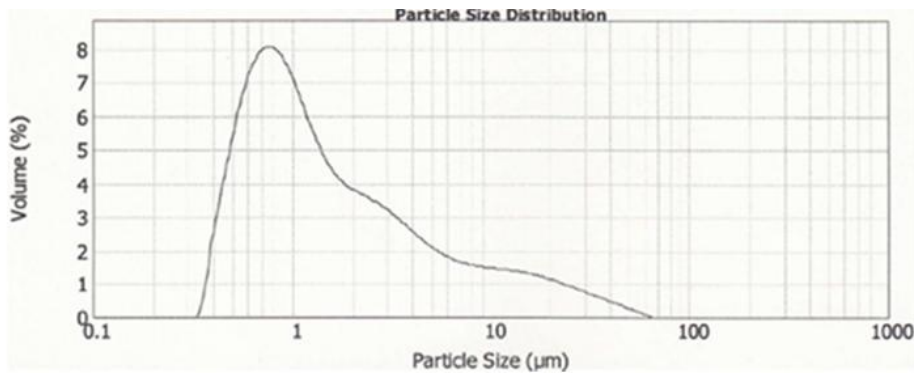


Fig. 7. Particle size analysis of 120 hours ground alumina

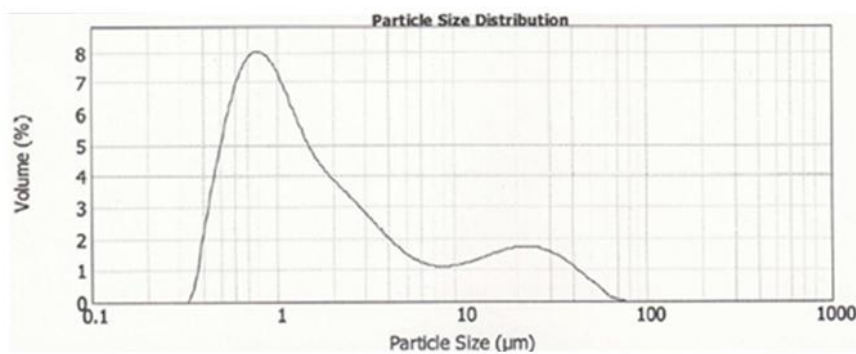


Fig. 8. Particle size analysis of 140 hours ground alumina

The above result showed that the average particle size of 120 hours ground sample was 1.268  $\mu\text{m}$  and for 140 hours ground sample was 1.301  $\mu\text{m}$ . Thus further grinding is required for the above ground sample to achieve nano.

#### Station II results

The grinding process was carried out in a dual drive mill which is a planetary ball mill shown in Fig. 9. The material of jar and balls are made up of stainless steel and the size of ball is of 10 mm diameter. The principle behind this is that during the mechanical alloying the powders are cold welded and fractured. The mill comprises of a single turn disc and double bowls. The turn disc rotates in one direction while the bowls rotates in the reverse direction. The centrifugal forces, created by the rotation of the bowl around its own axis composed with the rotation of the turn disc, are applied to the powder mixture and balls in the bowl are interchangeably synchronized. Thus friction caused from the hardened milling balls ground the powder mixture. Thus the powder mixture is fractured and cold weld under high energy impact.

Initially the jars were rinsed with sample alumina for 10 hours as previously the jars were exposed to iron oxide material shown in Fig. 10. 220 gm of alumina per jar (2 jars) was placed in the ball mill and was ground for 5 hours and 10 hrs in their dual drive mill laid open to high-energy collision from the balls.



Fig. 9 Planetary ball mill and jar



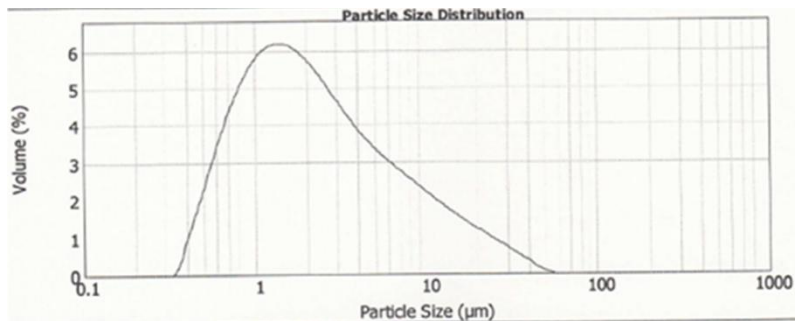
**Fig.10.** 10 mm stainless steel grinding balls before rinsing and after rinsing with  $\text{Al}_2\text{O}_3$ .

The ground sample was collected by rinsing with toluene acid shown in Fig. 11.

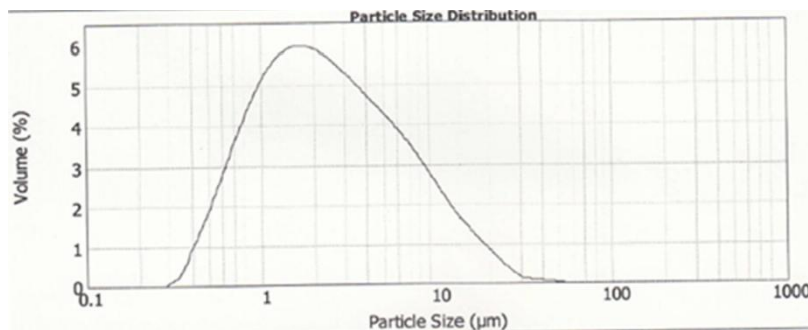


**Fig. 11.** The ground sample collection after rinsing with toluene.

The above ground sample was tested for particle size analysis. The report is shown in Fig. 12 and Fig. 13. The average size of particles milled at different times and at different stations are shown in Table 1.



**Fig.12.** Particle size analysis of 5 hours ground alumina



**Fig .13.** Particle size analysis of 10 hours ground alumina.

**Table 1** The average size analysis result of different machines ground for different hours

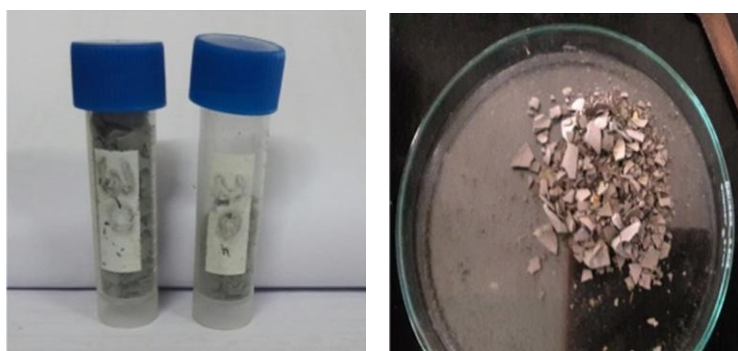
Sl. No.	Work Station	No of hours ground	Average size ( $\mu\text{m}$ )
1	Raw Material ( $\alpha\text{-Al}_2\text{O}_3$ )	0	68.308
2	Station-I	120	1.268
3	Station-I	140	1.301
4	Station-II	5	2.052
5	Station-II	10	2.306

After grinding in different ball mills and looking to their average sizes, it has been observed that the average size of every sample is about 1 micron. This might be because of the particle agglomeration or coating of some other material. Leaching process was carried out for removing the coating, hopeful that the size will reduce to nano size. Result of the leaching with 10 % HCL shown in Fig.14, is not satisfactory, followed by higher percentage of HCL leaching with 30%.



**Fig. 14.** Sample with 10% leaching.

Leaching of ground Alumina powder with higher concentration (20% and 30%) of HCL has been carried out shown in Fig. 15 and the samples are sent for characterization.



**Fig. 15.** Sample with 30% leaching.

#### IV. CONCLUSIONS

- Micro sized  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>as received from National Aluminum Company Limited (NALCO) was successfully converted to nano alumina.
- The alumina powders were converted to nano size through wet grinding process at different times to investigate the effect of grinding time on characteristics of the particles.
- It was observed that the more was the grinding time the lesser was the particle size.

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