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Sonochemical Synthesis of Zinc Oxide Nanoparticles and its Applications

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Abstract-This work is to synthesize Zinc Oxide nanoparticles using ultrasound using Sonication probe with Zinc Acetate as the reagent. These sonochemically synthesized Zinc Oxide nanoparticles are crystalline structure and the morphology of synthesized Zinc Oxide nanoparticles were observed using powder X-ray Diffraction, Fourier Transform Infra-Red analysis, Scanning Electron Microscopy. The optical properties of Zinc Oxide nanoparticles are characterized using UV-Visible spectroscopy. X Ray Diffraction results shows the sonochemically prepared Zinc Oxide sample is highly crystalline in nature and having wurtzite crystal structure. Fourier Transform Infra-Red spectra peak is 417.52 cm^{-1} which is the characteristic absorption bands of Zinc Oxide nanoparticles. UV-Visible absorption spectrum shows that a typical spectrum for Zinc Oxide nanoparticles. The Scanning Electron Microscope image shows that Zinc Oxide nanoparticles prepared in this study are spherical in shape with smooth surface. And the prepared Zinc Oxide nanoparticles are used for the application of textile industry. Here, Zinc Oxide nanowires are grown on cotton fabric to impart self-cleaning, super hydrophobicity and ultraviolet light blocking properties.

Index words- Zinc Oxide nanoparticles, Sonication, Scanning Electron Microscope, X-Ray Diffraction, Fourier Transform Infra-Red Spectroscopy, Ultrasound, applicatrions of Zinc Oxide

1. INTRODUCTION

There are many process happening in the industry to produce Zinc Oxide nanoparticles for several applications. But, use of ultrasound using sonication bath and sonication probe is the new type of technology used to synthesize nanoparticles in an effective manner. The aim is to synthesis nanoparticles of Zinc Oxide, and using it for some applications. This work of sonication process to produce Zinc Oxide nano particles is taken. Zinc Oxide is an inorganic compound and it is named by the formula ZnO. It is insoluble in water and it is widely used as an additive in numerous materials and products including rubbers, plastics, ceramics, glass, cement, lubricants, paints, ointments, adhesives, sealants, pigments, foods, batteries, ferrites, fire retardants, and first-aid tapes. Although Zinc Oxide occurs naturally as the mineral zincate, most Zinc Oxide is produced synthetically. Pure ZnO is a white powder, but in nature it occurs as the rare mineral zincite, which usually contains Manganese and other impurities that confer a yellow to red color. Crystalline Zinc Oxide is thermochromic, changing from white to yellow when heated in air and reverting to white on cooling. This color change is caused by a small loss of oxygen to the environment at high temperatures. Zinc Oxide is an amphoteric oxide. It is nearly insoluble in water, but it will dissolve in most acids, such as Hydrochloric acid. Zinc Oxide also forms cement-like material when treated with Phosphoric acid. A major component of zinc Phosphate cement produced by this reaction is Hopeite.

2. LITERATURE SURVEY

S. Jurablu, M. Farahmandjou, and T. P. Firoozabadi', *etal,* investigated on the Sol-Gel Synthesis of Zinc Oxide Nanoparticles. Study of Structural and Optical Properties and concluded that Zinc Oxide nano powders were synthesized by the Sol–Gel method from an Ethanol solution of Zinc Sulfate heptahydrate in the presence of Diethylene Glycol surfactant. Detailed structural and microstructural investigations were carried out using X-Ray Diffraction (XRD), High-Resolution Transmission Electron Microscopy (HRTEM), Field Emission Scanning Electron Microscopy (FE-SEM), Fourier Transform Infrared Spectroscopy (FTIR) and UV-Vis Spectrophotometer. X Ray Diffraction (XRD) pattern showed that the Zinc Oxide nanoparticles exhibited hexagonal wurtzite structure. The average particle size of Zinc Oxide was achieved around 28 nm as estimated by X Ray Diffraction (XRD) technique and direct High Resolution Transmission Electron Microscopy observation. The surface morphological studies from Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) depicted spherical particles with formation of clusters. The sharp peaks in Fourier Transform Infra-Red (FTIR) spectrum determined the purity of Zinc Oxide nanoparticles and absorbance peak of UV-Vis spectrum showed the wide bandgap energy of 3.49 ev.

Byeong Woo Lee, Jin Heui Koo, Tae Suk Lee, Yun Hae Kim, and Jae Suk Hwang’,*etal*, investigated on the Synthesis of Zinc Oxide Nanoparticles via Simple Wet-Chemical Route and concluded that Zinc Oxide powders were synthesized by a simple Precipitation and a Hydrothermal process at the temperature range of 100ºC. In precipitation process, the powders were formed by mixing aqueous solutions of Zinc Nitrate with aqueous Sodium Hydroxide solution under controlled process conditions such as precursor concentration, reaction pH and temperature. Single phase Zinc Oxide particles can be easily synthesized in lower precursor concentration, higher reaction pH and temperature. The powders synthesized at room temperature exhibited plates, rods or pointed multiped morphologies depending on the concentration and pH. Zinc Oxide crystallites synthesized by hydrothermal process consisted mostly of well-developed large or elongated crystallites of plates or rods in shape. The results reveal that the Zinc Oxide crystallite sizes and shapes would be efficiently controllable by changing the processing parameters of the preparation processes.

Tuğba Isık, Mohamed Elhousseini Hilal and Nesrin Horzum’*etal*, investigated on the Green Synthesis of Zinc Oxide Nanostructures and concluded that Zinc Oxide based nanomaterials have been proven to be of great use for several leading applications since the beginning of nanoscience due to the abundance of Zinc element and the relatively easy conversion of its oxide to nanostructures. Nowadays, Zinc Oxide as nanoparticles, nanowires, nanofibers as well as plenty of other sophisticated nanostructures takes place among the pioneer nanomaterials employed in the photovoltaic systems, fuel cells, and biomedical fields. Nevertheless, optimizing energy consumption and being eco-friendly are the challenging requirements that are still to be overcome for their synthesis. Green chemistry has been strongly presented recently in the scientific arena as an adequate potential alternative; worldwide investigations have been held on subjects involving bacteria, fungus, or algae-based synthesis as efficient options, and some of the intriguing scientific findings on this subject are reported hereafter.

Dattatraya B. Bharthi, A.V Bharthi’,*etal*, investigated on the Synthesis of Zinc Oxide nanoparticles by Hydrothermal process and concluded that Zinc Oxide nanoparticles with a granular morphology were synthesized using a hydrothermal method. Structural analysis revealed that Zinc Oxide Nano particles had a single crystal wurtzite hexagonal structure. Solvent polarity was responsible for varying and controlling their size and morphology. The process was very trouble free and scalable. In addition, it could be used for fundamental studies on tunable morphology formation. This hydrothermal method showed different morphology with different co-surfactants such as a floral-like or wire-like belt sheet structures etc. Based on their surface morphology, the same material had different applications as a catalyst in various organic reactions and also could be used as a photocatalyst and fuel cell, solar cell or in semiconductors etc. X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Ultra Violet–visible Spectroscopy and Photoluminescence of the resulting product was performed to study its purity, morphology and size, plus its optical properties via measurement of band gap energy and light absorbance.

Niranjan Bala, S. Saha, R. Basu and N.Nandy’, *etal***,** Recent progress in the Green synthesis of Zinc Oxide nanoparticles using Hibiscus Sudorific and concluded that Zinc Oxide Nanoparticles have been synthesized using *Hibiscus subdariffa* leaf extract. Temperature dependent synthesis and particle growth have been studied. Formation of Nanoparticles was confirmed by UV-visible (UV-Vis) Spectroscopy, Fourier Transform Infra-Red (FTIR) Spectroscopy and X-Ray Diffraction (XRD). Electron Microscopy has been used to study the morphology and size distribution of the synthesized nanoparticles. The synthesized Zinc Oxide nanoparticles as potential anti-bacterial agents have been studied on *Escherichia coli* and *Staphylococcus aureus*. Another study has indicated that small sized Zinc Oxide nanoparticles, stabilized by plant metabolites had better anti-diabetic effect on Streptozotocin induced diabetic mice than that of large sized Zinc Oxide nanoparticles. It has also been observed by Enzyme Linked Immuno Sorbent Assay (ELISA) and Real Time Polymerase Chain Reaction (RT-PCR) that Zinc Oxide can induce the function of Th1, Th2 cells and expressions of insulin receptors and other genes of the pancreas associated with diabetes.

Kenneth Maduabuchi Ezealisiji,Xavier Siwe‑Noun dou, Blessing Maduelosi, Nkemakolam Nwachukwu, Rui Werner Macedo Krause’*etal*, investigated on the Green synthesis of Zinc Oxide nanoparticles using Solanum Torvum (L) leaf extract and concluded that current study reports a simple and one-pot synthesis of Zinc Oxide Nanoparticles using an aqueous extract of Solanum Torvum and evaluation of its toxicological profile (0.5% w/w and 1.0% w/w) in Wistar albino rats with respect to the biochemical index. The Zinc Oxide nanoparticles were characterized using Ultra Violet–Visible (UV-Vis) Spectroscopy, Transmission Electron Microscopy (TEM), Fourier Transform Infrared (FTIR) Spectroscopy and X-Ray Diffraction(XRD) technique. Dynamic Light Scattering (DLS) and zeta potential of synthesized nanoparticles were analyzed to know the average size and stability of particles. Biochemical markers of hepatic and renal functions were measured. Zinc Oxide nanoparticles significantly decreased serum uric acid level ($p < 0.001$) in a dose-dependent manner, while the serum alkaline phosphatase level was increased at the two test doses. This study concludes that biogenic Zinc Oxide nanoparticles-infused hydrogel applied dermatologically could affect hepatic and renal performance in rats, and there was an observed cumulative toxicological effect with time of exposure.

Anjali Valsa Rajan , Aditya Singh’,*etal*, investigated on the Synthesis of Zinc Oxide Nanoparticles by Wet Chemical Method and concluded that the study was conducted with the aim of preparing Zinc Oxide nanoparticles within a short timeline by using standard laboratory technique. Zinc Oxide Nanoparticles were prepared by Wet Chemical method. Antibiotic Susceptibility and enhancement in the activity of those antibiotics when applied in combination with Zinc Oxide Nanoparticles for Escherichia coli and Staphylococcus aureus were performed. For E. coli, the enhancement was seen for the following Antibiotics –Ampicillin, Erythromycin and Tetracycline. For S. Aureus, enhancement was seen for the following antibiotics- Ampicillin, Cloxacillin, Cefotaxime, Cotrimoxazole, Chloramphenicol, Tetracycline, Erythromycin, Penicillin, Amoxicillin, Ciprofloxacin.

3. EXPERIMENTAL SECTION

The use of ultrasound prepared using sonication bath and it is the act of applying sound energy to agitate particles in a sample, for various purposes such as the extraction of multiple compounds from plants, microalgae and seaweeds. Ultrasonic frequencies (>20 kHz) are usually used, leading to the process also being known as ultrasonication or ultra-sonication. In the laboratory, it is usually applied using an ultrasonic bath or an ultrasonic probe, colloquially known as a sonicator. In a paper machine, an ultrasonic foil can distribute cellulose fibred more uniformly and strengthen the paper.

The procedure for preparing Zinc Oxide nanoparticles are as follows. 6g of Zinc Acetate Dihydrate was added to 200 ml of double distilled water with continuous stirring to dissolved Zinc Acetate completely. Then the solution was heated to 50℃ and 300 ml of absolute alcohol was added slowly with stirring. After this, 6ml of Hydrogen Peroxide was added drop wise to the vessel and mixed it using a magnetic stirrer to get an almost clear solution. Then the beaker containing the solution was placed in a sonication probe and the process was initiated at the power of 500 watts. Here the tip of the sonicator was inserted directly into the solution and a frequency of 20 kHz was maintained having a overall voltage of 220.. The operating pulse ranges between 30 second pulse ON to 30 second pulse OFF. And the sonication process was carried out for 90 minutes for the prepared mixture of Zinc Oxide solution. After the process gets over this solution was incubated for 24 hours and the solution was dried at 80°C for several hours to obtain white nano Zinc Oxide. Nano Zinc Oxide was washed several times with double distilled water to remove the byproducts. After washing, the Zinc Oxide nanoparticles were dried at 80℃ in hot air oven. Complete conversion of Zinc Oxide will occur during the drying process. And the obtained Zinc Oxide was subjected to characterized and used in some of the applications.

5. RESULTS AND DISCUSSION

X-Ray Diffraction Analysis

The phase purity and composition of the particles obtained by a sonication process examined by X Ray Diffraction. Figure (5(a)) shows a typical X Ray Diffraction pattern of Zinc Oxide nanoparticles, prepared in this work. A number of Bragg reflections with 2θ values of 31.74°, 36.83°and 47.62° are observed corresponding to (100), (101) and (102) planes, shows a typical X Ray Diffraction pattern of Zinc Oxide nanoparticles in the range of 5°-50° at a scanning step of 0.01. Average size of the Zinc Oxide nanoparticles was determined as 58.3 nm from the width of dominate peaks (100) and (101) reflections according to the Debye - Scherrer equation. All diffraction peaks are indexed according to the hexagonal phase of Zinc Oxide. No characteristic peaks of impurity phases except Zinc Oxide are found which revealed that good crystalline in nature of the samples. The broadening of the peaks in the above X Ray Diffraction pattern can be attributed to the small particle size of the synthesized Zinc Oxide

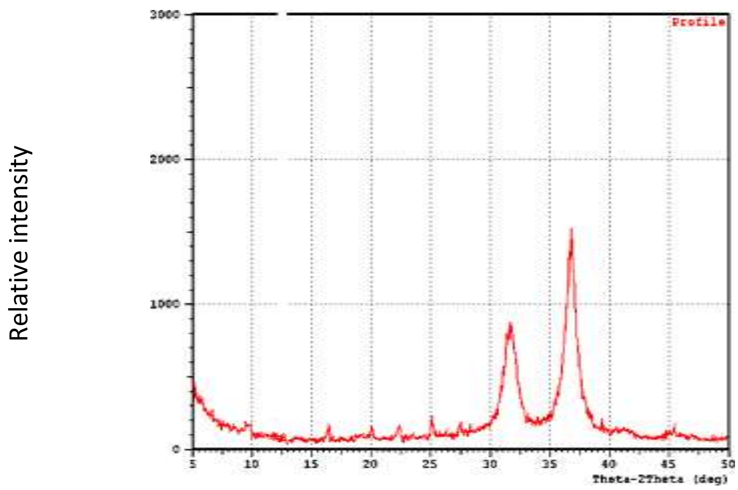

Figure 5(a). Shows Typical XRD pattern of ZnO nanoparticles

Fourier Transform-Infra Red Analysis

Fourier Transform-Infra Red is an effective method to reveal the composition of products. Figure (5(b)) is a typical Fourier Transform-Infra Red spectrum of pure Zinc Oxide nanoparticles, the peak at 417.52 cm^{-1} is the characteristic absorption of Zinc-Oxide bond and the broad absorption peak at 3438 cm^{-1} can be attributed to the characteristic absorption of hydroxyl. Anyhow, the Fourier Transform-Infra Red and X Ray Diffraction results show high purity of the obtained Zinc Oxide nanoparticles.

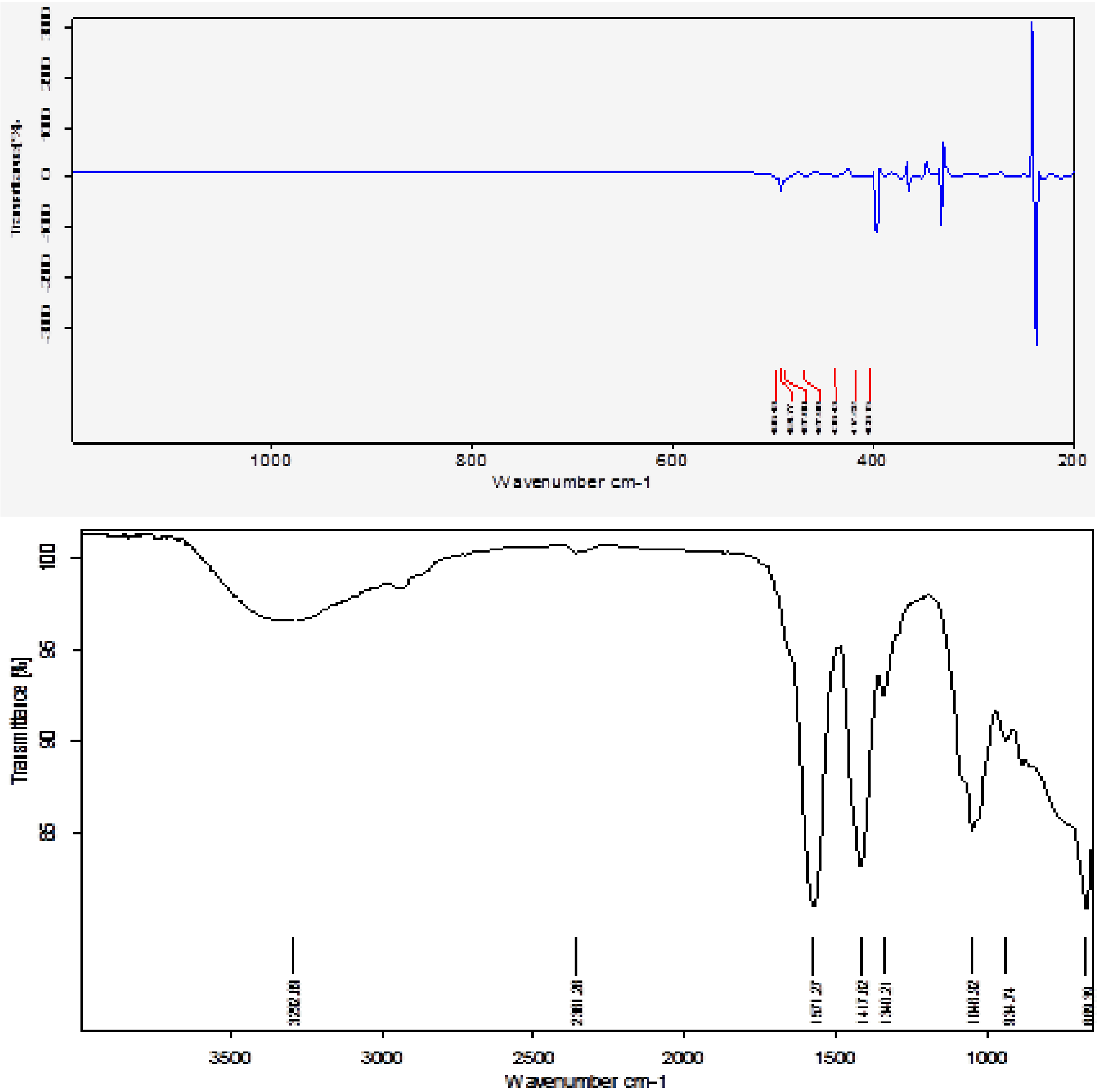

Figure 5(b). Fourier Transform Infra Red Transmition spectra of pure Zinc Oxide nanoparticles

Scanning Electron Microscope (SEM)

Figure 5(c) shows the Scanning Electron Microscopy image of Zinc Oxide nanoparticles. The Scanning Electron Microscopy image was taken at 25,000 times magnification. The image shows Zinc Oxide particles are spherical in shape with smooth surface and the size of the particles around 100-200 nm. In another experiment, freshly prepared Zinc Oxide Sol-gel was coated onto polyethylene thin film and dried at 80°C. The particle size of Zinc Oxide nanoparticles prepared via this method was about 50-60 nm (image not shown here). We can clearly conclude that Zinc Oxide nanoparticles continue to grow after synthesis, even when stored at room temperature.

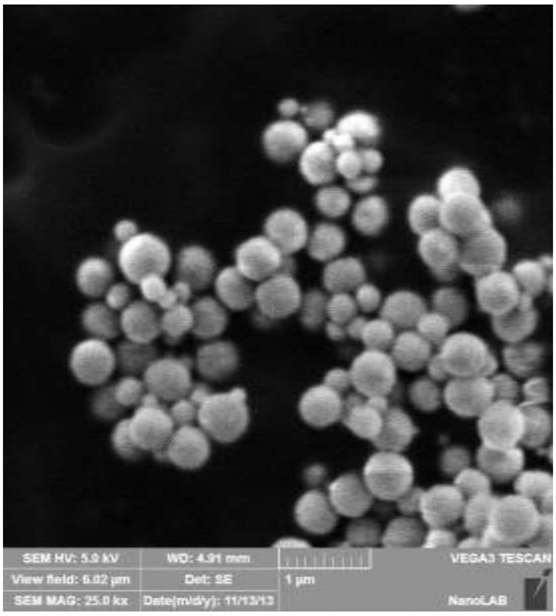

Figure 5(c). Shows the Scanning Electron Microscope image of Zinc Oxide nanoparticles

Ultra Violet Visible Spectrophotometer Analysis

Figure 5(d) shows the Ultra Violet-Visible oOptical absorption spectrum of Zinc Oxide Nano particles dried in air at 80°C. The absorption spectrum shows a sharp absorbance onset at 345 nm, which indicates an almost uniform size of the nanoparticles. However, upon change in particle size or particle shape, a slight shift in the absorption was observed.

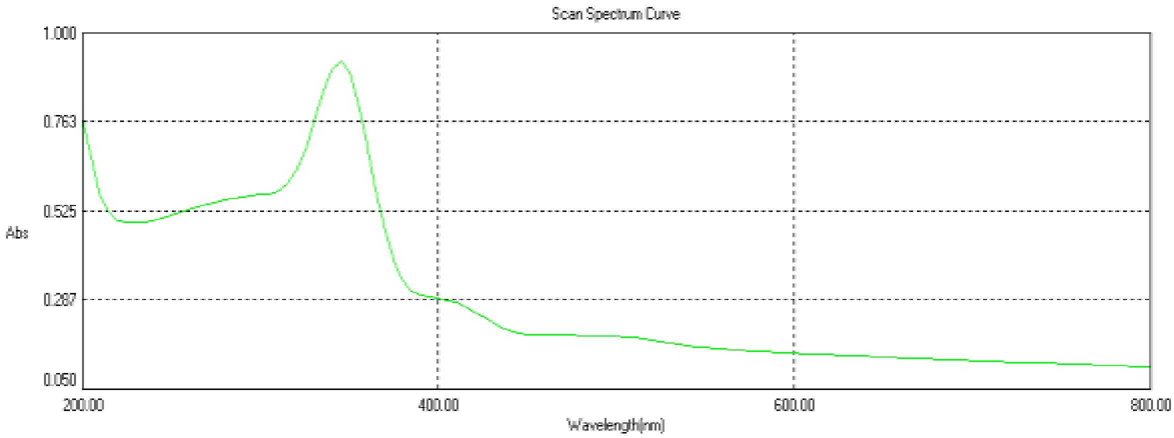

Figure 5(d). Ultra Violet-Visible Optical absorption spectrum of Zinc Oxide nano particles

6. APPLICATON OF ZINC OXIDE NANOPARTILES

The Textile Industry

In the textile industry Zinc Oxide a vast potential for the commercialization of nanotechnological products. In particular, water repellent and self-cleaning textiles are very promising for military applications, where there is a lack of time for laundering in severe conditions. Also in the world of business, self-cleaning and water repellent textiles are very helpful for preventing unwelcome stains on clothes. Protection of the body from the harmful Ultra Violet portion of sunlight is another important area. Many scientists have been working on self-cleaning, water repellent and Ultra Violet-blocking textiles.

6.1 Preparation of Zinc Oxide coated cotton cloth

For textile applications, not only is Zinc Oxide biologically compatible, but also nanostructured Zinc Oxide coatings are more air-permeable and efficient as Ultra Violet-blockers compared with their bulk counterparts. Therefore, Zinc Oxide nanostructures have become very attractive as Ultra Violet-protective textile coatings. For instance, hydrothermally grown Zinc Oxide nanoparticles in SiO_2-coated cotton fabric showed excellent Ultra Violet-blocking properties. Synthesis of Zinc Oxide nanoparticles elsewhere through a homogeneous phase reaction at high temperatures followed by their deposition on cotton and wool fabrics resulted in significant improvement in Ultra Violet-absorbing activity.

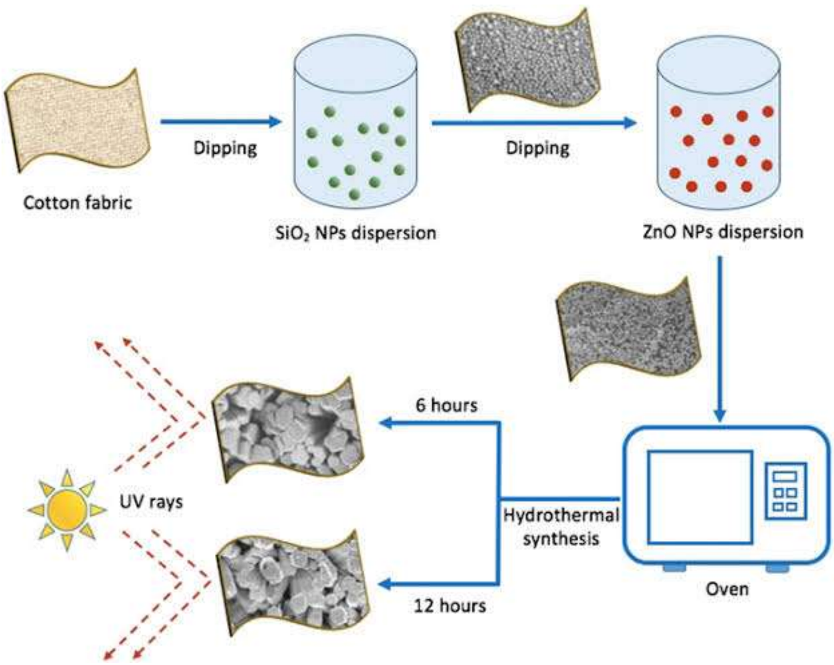

Fig 6(c) Schematic diagram of hydrothermal Zinc Oxide coating process

In the above process Zinc Oxide nanowires were grown on cotton fabric by to impart self-cleaning, and UltraViolet blocking properties. The Zinc Oxide nanowires were grown by a microwave-assisted hydrothermal method. Silica and Zinc Oxide nanoparticles are dissolved completely in separate beakers and both are in dispersion medium. The cotton cloth was then dipped into the Silica dispersion and then into the Zinc Oxide dispersion. After dipping the nanoparticles of both the medium gets attached to the cotton cloth, it was subjected to a oven and hydrothermal synthesis was takes place. Finally the cotton cloth was coated with Zinc Oxide nanoparticles in the form of nanowires or stem.

7. CONCLUSIONS

Zinc Oxide nanoparticles have been successfully synthesized by simple Sonochemical Intensification method. The prepared Zinc Oxide nanoparticles were spherical in shape and were characterized using X Ray Diffraction, Ultra Violet-Visible absorption, Fourier Transform -Infra Red and Scanning Electron Microscopy techniques. The average particle size was found to be 58.3 nm and 100-200 nm obtained from Scanning Electron Microscopy measurement for Zinc Oxide nano particles dried at 80℃. Then the Zinc Oxide nanoparticles was planned for an application on preparing a auto-cleaning and Ultraviolet wetting textile by the process of Hydrothermal synthesis and a Zinc Oxide nanoparticles coated cotton cloth was planned to prepare because Zinc Oxide nanoparticles offer tremendous potential in future applications of electronic and magneto–electric devices. Also, maybe, applied for photocatalysis, gas sensing, biomedical device and sun screen applications. The method has a high yield and can be used for large scale synthesis of Zinc Oxide nano particles.

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