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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF TIO_2 NANOPARTICLES.

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Abstract

Nanocrystalline semiconductor metal oxides have achieved great importance in world today. They may be defined as metal oxides with crystal size between 1 and 100 nm. TiO_2 nanoparticles have attracted significant interest of material scientists and physicists due to their special properties and have attained great importance in several technological applications such as photocatalysis, sensors, solar cells and memory devices. The TiO_2 nanoparticles have excellent antimicrobial activity against broadened spectrum of bacterial strains. In the present research work synthesis of TiO_2 nanoparticles has been carried on by Wet chemical method. The characterization of particles is carried out by XRD, TEM and SEM techniques. The stability of the nanoparticles is determined by TGA analysis. The importance and applications of these nanoparticles are presented and discussed in detail.

Introduction:-

Nanoparticles are particles between 1 and 100 nanometers in size.(1) Nanomaterials have high surface to volume ratio and thus have tremendous force in diffusion. The metallic nanoparticles are the most promising as they show good antibacterial properties due to their large surface area to volume ratios, which draw growing interest to researchers due to increasing microbial resistance against metal ions, antibiotics and the development of resistant strains.(2)

Nanocrystalline semiconductor TiO_2 particles are of particular interest due to their unique properties and several potential technological applications such as photocatalysis, antimicrobial, sensors, solar cells and memory devices. TiO_2 exists in three polymorphic phases: rutile (tetragonal density = 4.25 g/cm3), anatase (tetragonal, 3.894 g/cm3) and brookite (orthorhombic, 4.12 g/cm3).

TiO_2 nanoparticles are synthesized using various methods such as sulfate process , chloride process impregnation, coprecipitation , hydrothermal method , direct oxidation of $TiCl_4$, metal organic chemical vapor deposition method, etc. Sol-gel method is one of the most convenient ways to synthesize various metal oxides due to low cost, ease of fabrication and low processing temperatures. It is widely used to prepare TiO_2 for films, particles or monoliths.(3)
In general, the sol gel process involves the transition of a system from a liquid "sol" (mostly colloidal) into a solid "gel" phase. The homogeneity of the gels depends on the solubility of reagents in the solvent, the sequence of

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addition of reactants, the temperature and the pH. The precausors normally used for the synthesis and doping of nanoparticles are organic alkoxides, acetates or acetyl acetates as well as inorganic salts such as chlorides. Among the classes of solvents, alcohols are largely used.(4)

TiO_2 is stable in aqueous media and is tolerant of both acidic and alkaline solutions. It is inexpensive, recyclable, reusable and relatively simple to produce. It can also be synthesized in nanostructure form more readily than many other catalysts. (5)

TiO_2 nanoparticles (TiO_2-NPs), approximately less than 100 nm in diameter, have become a new generation of advanced materials due to their novel and interesting optical, dielectric, and photo-catalytic properties from size quantization.

Titanium dioxide (TiO_2) is a photo catalyst and widely utilized as a self-cleaning and self disinfecting material for surface coating in many applications, titanium dioxide has a more helpful role in our environmental purification due to its nontoxicity, photo induced super-hydrophobicity and antifogging effect.

Materials & methods:-

Synthesis of TiO_2 nanoparticles:-

20 ml Titanium chloride is mixed with 60 ml of 0.1N Ammonium hydroxide and this mixture is stirred for 48 h at room temperature. Titanium dioxide nanoparticles formation is indicated by change in color from purple to white colored solution. Solution was centrifuged and the precipitate was washed with distilled water and dried in isopropanol at RT. The samples were further subjected to antimicrobial activity and characterization by XRD. (Principles of Nanotechnology –Sulabha Kulkarni)(11)(13).

In this study, TiO_2 nanoparticles are synthesized by wet chemical method - a simple method under ambient atmosphere at room temperature, and well characterized by X-ray diffraction (XRD), SEM, TEM & TGA.

Characteristaion of TiO_2 nanoparticles:-

The structural properties of TiO_2 nanopartricles were recorded on ADV D8 Bruker X-ray powder diffractometer with Cu radiation, (λ=0.154nm in the range of 20-70^o.TEM images were observed on TECNAI FE12 TEM instrument operating at 120 kV using SIS imaging software (Hitatchi H-7500). The particles were dispersed in isopropanol and a drop of it was placed on formvar-coated copper grid followed by air drying. Scanning electron microscopy (ZEISS make) was used for morphology assessment. The sample was collected on a round cover glass (1.2 cm), washed with deionized water and dried in a desiccator at room temperature. The cover glass was then mounted on a SEM stub and coated with gold for SEM analysis. Thermal decomposition profile was recorded on Mettler Toledo TGA 851^e instrument in the temperature range 10 – 800^{0}C with a heating rate of 2°C.

Stability of TiO2 nanoparticles :

To test the stability of TiO_2 nanoparticles at different temperatures, the nanoparticles were placed at room temperature 25^{0}C, in Refrigerator at a temperature at 4^{o}C for 24h. Dental isolates D2, D4 were inoculated onto Nutrient agar plates and the wells were incorporated with TiO_2 nanoparticles placed at different temperatures.(10)

RESULTS AND DISCUSSION:-

Synthesis of TiO_2 nanoparticles:-

All the reagents used were of analytical grade and no further purification was done before use. The TiO_2 nanoparticles obtained from Titanium trichloride and ammonium hydroxide was washed with distilled water and dried in isopropanol. White amorphous powder formed indicated the formation of TiO_2 nanoparticles.(fig 1)

Fig 1:- Titanium dioxide nanoparticles

Characterisation of TiO2 nanoparticles :-

TiO_2 nanoparticles were characterized by using XRD which was performed at a scanning range of 2- $70^o(2\theta)$ using copper Kα radiation with a wavelength of 1.5406 A^o. Fig.2.2 shows the X-ray diffraction analysis of TiO_2 nanoparticles. The major peaks are indexed at 26^o, 36^o, 43^o, 54^o, 62.5^o, 71.5^o respectively. All the diffraction peaks of sample correspond to the characteristic hexagonal wurtzite structure of TiO_2 nanoparticles. Absence of any other peaks clearly indicates that no impurities are present. The diameter of the nanoparticles is calculated by Debye-Sherrer equation: $D = K\lambda/\beta \cos\theta$ where K is Sherrer constant, λ is the X-ray wavelength, β is the peak width at half maximum, and θ is the Bragg diffraction angle. The average crystallite size D was estimated to be around 26.6 nm using the Debye–Sherrer formula. (6)

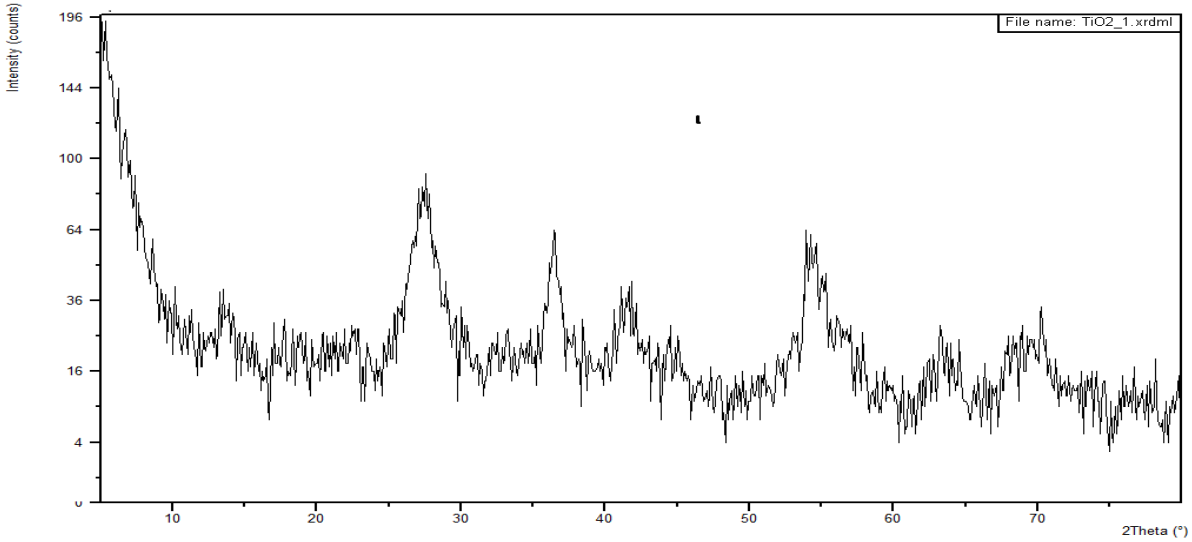

Fig 2.2 :- XRD graph of TiO2 nanoparticles

TEM & SEM Analysis:-

TEM images of sol-gel derived nanoparticles are shown in Fig. 3a. Clear spherical structures can be seen in the Fig. 3a having diameter ~ 20nm. Selected area diffraction is shown in Fig. 3a which clearly indicates that the TiO_2 nanoparticles are highly crystalline in nature. Selected area diffraction pattern of the nanoparticles indicates that the TiO_2 nanoparticles prepared via this route are crystalline in nature. Nanoparticles obtained in this case are adhering to one another. Agglomeration of nanoparticles is more in fig 3.b than the former one. As can be seen from the TEM image that the average particle size is ~ 20 nm, which is in agreement with the crystallite, size obtained from XRD.

The particles size measured by TEM were generally in agreement with those determined by XRD though some agglomeration is present due to high surface energy of the particles.(7,8)

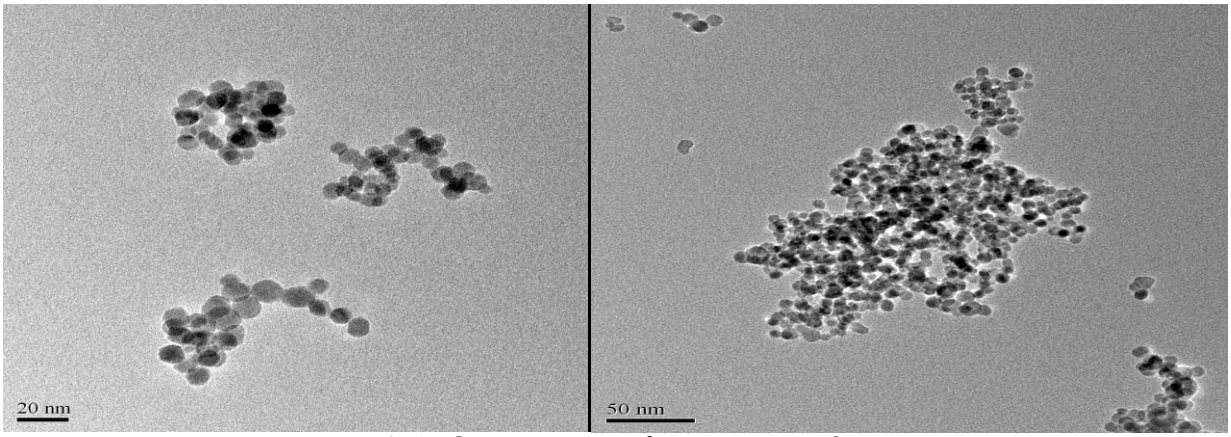

Fig 3a, b:- TEM image of TiO2 nanoparticle

SEM images of the TiO_2 nanoparticles prepared by wet chemical method is shown in Fig. 3.1 Fig. 3.1a,b shows the SEM image of nanoparticles. Clear nanostructures can be seen in figure. The SEM images of TiO_2 nanoparticles showed agglomeration(9) of particles. The nanoparticles seen by SEM image consist of a number of crystallites which are observed by TEM image.

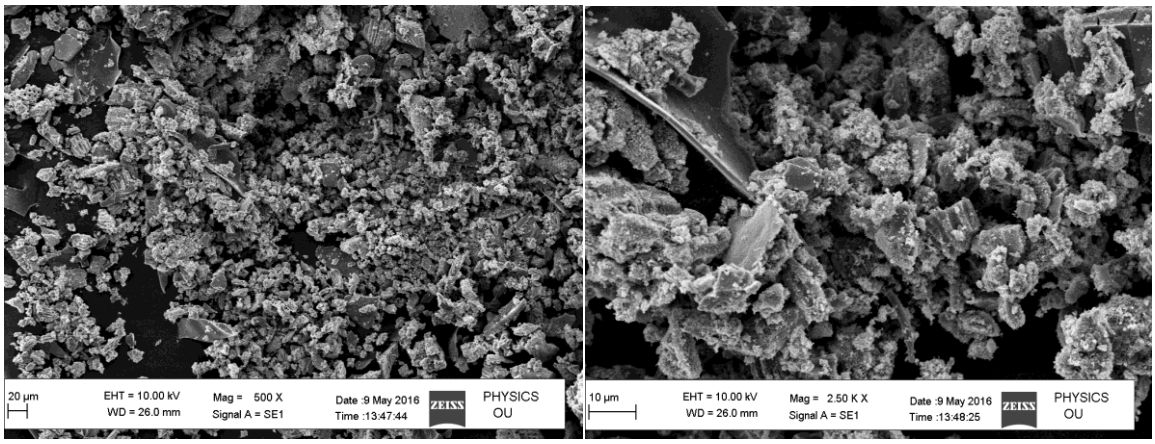

Fig 3.1 a, b:- SEM images of TiO2 nanoparticles

TGA Analysis of TiO2 nanoparticles:-

The TGA analysis of the nanoparticles as shown in fig. 3.2 indicate that there is significant weight loss at a temperature of 103.996 ^{0}C, 104 .14^{o}C and 104.29 ^{o}C of around 11 %. The mass loss at the following temperatures is due to loss of physically absorbed water molecules as well as loss of volatile impurities present if any. There is no significant weight loss observed at any other temperature showing that TiO_2 nanopartlcles are stable upto temperatures of 800^{0}C and can resist temperatures of 800^{0}C.

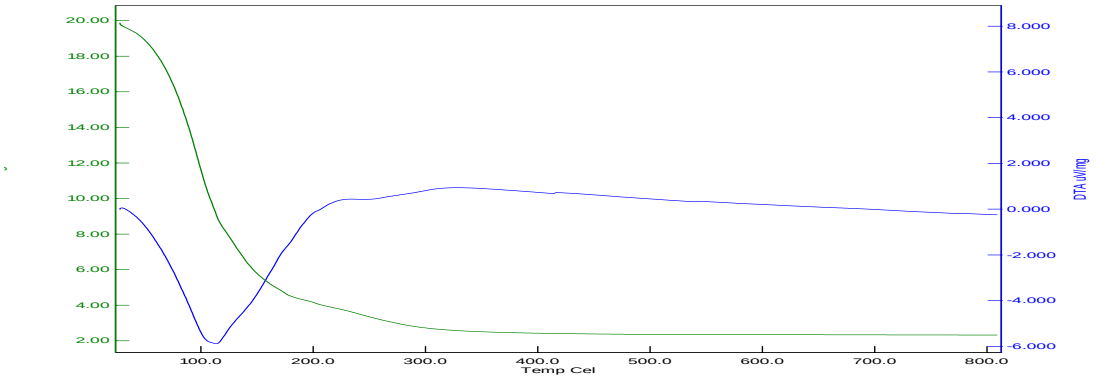

Fig 3.2:- TGA analysis of TiO_2nanoparticles

Activity of TiO_2 nanoparticles at different temperatures:-

The nanoparticles were stored in Refrigerator at a temperature of 4°C and at room temperature at around 25°C and the antibacterial activity of the nanoparticles placed at these temperatures were checked against dental plaque causing organisms.(12) Isopropanol was used as control..The results show that the nanoparticles placed in refrigirator were more effective with a zone diameter of 2.5cm followed by the one placed at room temperature.(fig 3.3) From this study we can infer that the nanoparticles stored in refrigirator are most effective compared to that of the one stored at room temperature.

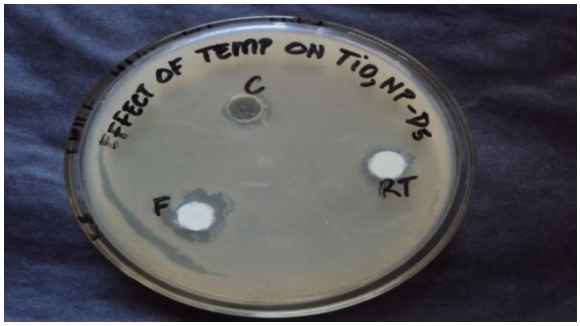

Fig 3.3:- Activity of TiO_2 nanoparticles at different temperatures

Comparitive Analysis of the antimicrobial activity of TiO_2 nanoparticles (syn) vs TiO2 nanoparticles (com):-

Antimicrobial activity of TiO_2 nanoparticles synthesized by wet chemical method (Principles of Sulbha Kulkarni) was checked against TiO_2 nanoparticles (commercial sample) P23 from ARCI. The zone diameter was 20 mm in case of TiO_2 nanoparticles synthesized by wet chemical method and 15 mm by the commercial TiO_2 nanoparticle in case of D23 dental isolate. It was observed that the TiO_2 nanoparticles which were synthesized by wet chemical method had fairly good activity compared to the commercial TiO2 nanoparticles.

Fig 3.4:- Comparitive analysis of antimicrobial activity of TiO_2 np (syn) vs TiO2 (comm.)

Conclusion:-

The TiO_2 nanoparticles synthesized by wet chemical method are amorphous in nature. The TiO_2 nanoparticles which were synthesized and were well characterized by XRD, TEM, SEM, TGA.. The characterization revealed that theTiO_2 nanoparticles synthesized were of nanosize and pure. The XRD studies showed that TiO_2 nanoparticles were having a size of 26.6 nm which was similar to TEM studies.TGA analysis showed that TiO_2 nanoparticles were stable upto temperatures of 800^0C. It also indicates that products of TiO_2 nanoparticles can be subjected to fairly high temperaratures since they are stable. The studies also showed that TiO_2 nanoparticles can be used as antimicrobial agents against dental plaque causing organisms.

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