SYNTHESIS AND CHARACTERISATION OF HYBIRD NANOPARTICALES

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ABSTRACT: The foremost aim of present investigation is to enhance the thermal properties of fluids with dispersion of various types of hybrid/nanoparticles which has capability to enhance heat transfer characteristics. Thermal conductivities of nano fluids are expected to be higher than common heat transfer fluids. The use of metal nanoparticles has not been intensely investigated for heat stransfer applications due to lack of stability. Research efforts have been equally put forth to enhance the operational performance of Heat transfer fluid (HTF) using hybrid/nanomaterials.The relatively high surface-to-volume ratio of nanomaterials enables them to effectively promote nucleation and heat transfer in PCM. The proper selection and preparation of nanomaterials paves way for attaining enhanced thermal conductivity, specific heat and heat transfer performance of hybrid nano fluids. Nanomaterials, prepared in different structures including nanotubes, nanorods, nanoparticles, etc., would exhibit distinct thermo-physical aspects when dispersed in heat transfer fluid (water). In this context various hybrid/nanoparticles are synthesized using sol gel method. Synthesized particles are to be tested for XRD, SEM to know crystalline structure, size, shape, etc. Here we present an experimental study on the effect of various hybrid/nanoparticles when dispersed in HTF which is generally water with varying mass percentages. Ultrasonication process needs to be performed during dispersion of nanoparticles in HTF for uniform dispersion and to avoid agglomeration. Then nano based fluids were characterized to study Physical, thermal properties. The nano fluids were tested using Fourier Transformer Infrared Spectrometry (FTIR) to check chemical compatibility between HTF and nano particles, later thermal conductivity analyzer was used to verify thermal conductivity of various hybrid/nano based fluids. Thermo gravimetric analyzer

(TGA) is used to check the commencement and end of decomposition temperature of various hybrid/nano basedfluids. like distribution of nanoparticles in HTF, melting and freezing temperatures, thermal conductivity, differential scanning calorimeter (DSC) equipment need to be used to check melting freezing characteristics of nano fluids. Later several experimentations are carried out using shell and tube type heat exchanger to study heat transfer characteristics of various hybrid/nano fluids. The results from various characterization techniques and experimentation should be analyzed for optimizing the nano particles type and proportions used in HTF for proposing in practical applications.

Keywords: -x-ray diffraction, field emission scanning electron microscope, ultrasonic vibrator**,** differential calorimeter(DSC)

INTRODUCTION

Nanomaterial's are keystones of Nano science and nanotechnology. A nanoparticle has capability to alter process wherein substance and outputs are produced therefore scale and character of performances that may obtain. Currently, so one can absolutely gain within the future. A nanometre is so small compare to millimetre - about lakh times smaller than men's hair. Nanomaterial's is concern due to the truth at this extent precise,, electric powered, houses develop. These eminent homes have the ability for unbelievable impact in vcrs, computers transistors etc, remedy, and various areas. Few Nano stuff arise absolutely however of specialized hobby are engineered Nano stuff that can be created for, getting used in different company outputs and access. They can be noticed in stuff like , hair care products, tennis ,badminton stain-resistant iteams, rubber industries, electronics instruments, further to various ordinary things, and are practiced in medicinal drug for reason of evaluation, imaging and drug shipping. Nano substance are making at the molecular degree to take favor in their minute size and unique structure which are commonly now not seen of their regular, bulk opposite numbers. Nanomaterial's have far extra ground place to volume ratio than their universal paperwork that could start to more chemical reactivity and alter their power., Approach of industrial stuff to be had currently could be very tremendous, ,

The history of nanomaterial emerged out right now after the huge knock while Nano stuff were used within past meteorites. Nature later superior various exclusive Nanostructures like ocean shells, and plenty of others. Nanoscaled smoke particles have been common during using fireplace by using the usage of early people. The medical story of nanomaterials but started out out loads later. The foremost scientific record is gold debris in 1857. Nano stuff catalysts have been studied for past 70 years. Nano substance have programs in the feild of Nano era, and presentations one in every of a type physical chemical developments from general chemical substances (i.E., silver nano, carbon nanotube,, photo catalyst, carbon nano, silica)., Nanostructured substances are divided as per seigal are 0 dimensional, 1 dimensional, 2 dimensional, 3 dimensional. Nano stuff are materials which might be categorize with the help of manner of an especially quality grain length or by a dimensionality limited to 50 nm. Nano stuff can be design with different modulation dimensionalities as described by Richard W. Siegel: zero ,one ,two three These stuff have built a great interest in past decades by advantage of their amazing mechanical, electrical, optical and magnetic qualities Approaches for making nanostructures There are numerous various process of making nanostructures: of course, macromolecules or nanoparticles or Bucky balls or nanotubes and so on can be processed artificially for certain distinct stuffs. They can also be classify by process depend on equilibrium or near-equilibrium thermodynamics such as technique of self-organization and self-assembly .these techniques, processed substance can be group into useful figures so that in end the substance can be used to a few application. 6.1.2. Mechanical grinding is a classic sample of ‘top down’ approach of processing of nanostuff, where the substance is arranged not by cluster assembly but by the structural break down of coarser-grained structures as the result of serious plastic deformation. This has become a famous way to make nanocrystalline substances because of its easiness, the approximately economical machinery needed, and the appropriateness to essentially the synthesis of all classes of materials. The main advantage generally quoted is the possibility for easily scaling up to tonnage quantities of material for various uses. correspondingly, the severe issues that are normally specified are; 1. Contamination from milling media and/or atmosphere, and 2. To unite the

powder product without debauch the nanocrystalline microstructure. In fact, the contamination issue is generally given as a sense to reject the mechanism, at least for few materials. Here we will check the mechanisms presently admit answerable for creation of nanocrystalline structures by mechanical attrition of single phase powders, mechanical alloying of different powders, and mechanical crystallisation of amorphous substances. The initial substance is prepared to make a soluble oxide and forms a sol in contact with H2O or dilute HCL. Deportation of water from the sol produce the gel, and the sol/gel transformation manage particle dimensions. Calcination of gel yields oxide. Sol-gel processing correlate to the water removing and condensation of alkoxide-related precursors like as Si(OEt) The reactions related in the sol-gel chemistry depend on elimination of water and condensation of metal alkoxides M(OR)z can be defined as follows: MOR + H2O → MOH + ROH (hydrolysis) MOH + ROM → M-O-M + ROH (condensation) Sol-gel approach of processing Nano stuff is prominent between chemists and is extensively selected to form oxide substance. The sol-gel approach can be described by a chain of various steps. Evolution of various blanced solvent mixtures of the alkoxide or solvated metal precursor. 2. Gelation leads from development of an oxide- or alcohol- bridged system (the gel) by a polycondensation reaction that leads in a sudden rise in the viscocity of the solvent mixture .fading of the gel (during which the polycondensation process

Last up to the gel convert into a thick mass, follow by shrinkage of gel network and removal of solvent from gel pores. Ostwald ripening (also linked to as coarsening, is the event by which tiny particles are used by big particles during the growth development) and phase conversion may arise simultaneously with syneresis. The fading process of gels can increased up to week and is censorious to oppose of cracks in gels that have been cast. 4. Dehydrate of the gel, when H2O and other volatile liquid are eliminated from the gel. This procedure is arduous due to essential variation in the arrangement of the gel

PROCESSING OF NANOMATERIAL

Sol-gel principal

This need development of manmade system by which creation of a colloidal suspense so thick mass of sol to build a system in a steady watery section (gel).

precursors processing present colloids made of normally a hard stuff enclosed with help of different active ligands. Initial stuff processed so make solvable oxide hence varieties bulky stucture in associate with H2O or HCL .elimination of watery stuff from sol gives gelly, and gel transformation manage particle shape and size. Oxide is yield via calcination

this technique indicate to addition of water and condensation of alkoxide-depend precursors like as Si(OEt) 4). reactions in sol-gel chemistry formost depend on addition of water and change of steel alkoxide gases form to liquid M(OR):

$$MOR + H2O \rightarrow MOH + ROH \text{ (hydrolysis)}$$

$$MOH + ROM \rightarrow M\text{-}O\text{-}M + ROH \text{ (condensation)}$$

This technique of processing nanoparticles is little famous between chemists and generally used to make oxide stuff. This technique generally divided in various steps

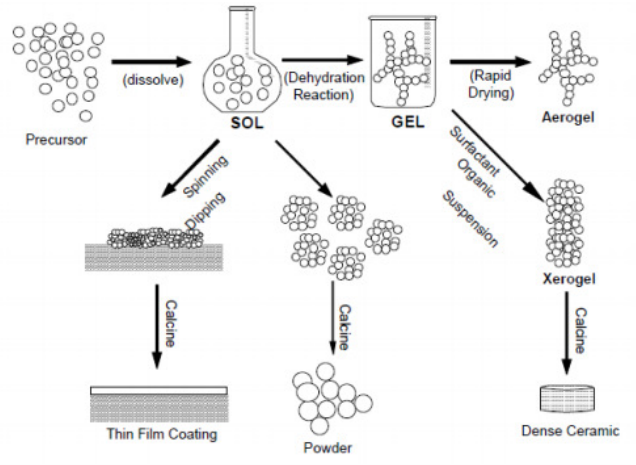


Fig. sol-gel chemistry of processing of nanostuff.

1. evolution of various steady liquids of alkoxide

2. gelly comes via evolution of oxide- or alcohol- bridged frame by poly condensation process which leads in sudden increment in viscosity of mixture

3. Declining of gel in which poly condensation process last until gelly change into a rigid stuff, lead to shrinkage of gel arrangement also removal of water from gel minute hloes. Ostwald (associate to as coarsening, is process in minute stuff are use by quit big particles growth period) phase conversion happen simultaneously along syneresis. fading era of gelly results to week so quite tough to avoidance of cleave in gelly which have to be make

4. dehydration of gelly, when H2O other volatile solvent are eliminated in structure.current technique quite difficult because of basic alteration in the arrangement of gel.

Due to heat evaporation if isolation occurs , product monolith is referred as aerogels. If liquid (H2O) take out in supercritical or close to it environment, output is jelly.

5. Dehydration, in which bound M-OH compounds are eliminated, against rehydration jelly is stabilized. It is due to calcining the output at temp up to 8000 C.

6in high temp (T>8000 C). decomposition and densification of gel occurs.

Minute holes of gel nearby break, resting common things are volatilized.basic things which following in sol-gel chemistry are proven below. Concern present synthesis technique occur because chance of processing non metal which is not organic stuff like glasses, ceramics or ceramic stuff in quite low tem in compare to

increment in temp gadget demanded through firing ceremics leading hurdles to stop in making A profitable bottom-up technique is manage tboom of particles then oppose fresh fashioned particles from agglomerating. struggles are provide the process are accomplished therefore no unseen reactant is remains on output and sure getting rid of any development aids which applicable in method . manufacturing quotes of these powders quite low by current technique. foremost gain anybody can get single sized Nano stuff via any bottom up principal.

XRD (X-Ray Diffraction)

The nuclear and subatomic shape of precise stones is figured by crystallography technique so atoms figure a low transmit X-beams to diffract in supream main headings. crystallographer transmit three dimension picture of thickness of electrons indoor gem by calculating the edges and power of these diffracted rays. By this electron thickness, prescribe place of the molecules in stone can be analyze,

The technique additionally describe the figure and capicity of different atoms, along vitamins, , proteins, like DNA.

central technique for specifying nuclear model of fresh materials to identifying stuff look comparative by help of first-rate study beam important stone frame likewise define irregular digital fields of a stuff.



Fig: XRD

FESEM

This microscope work with electron instead of light. These electrons are delivering through path of an area emission origin. The body is scanned aid through of electrons like to a zig-zag manner this microscope is applied to visualize quite small topographic small print on the area or whole or fractioned stuff. Researchers in chem, physics, bio with this approach to like to The capacity to study smaller-area contamination spots at electron accelerating voltages likeminded with electricity dispersive spectroscopy (EDS).

- Great -quality, low-voltage snap shots with minute electrical charging of samples (voltages ranging 0.5 to 30 kilovolts).
- Semiconductor gadget cross part inspection for gate widths, gate oxides, movie thicknesses, and making details
- Advanced film width and structure uniformity determination
- Small infection feature geometry and elemental composition measurement



Fig: FESEM

DSC (Differential Scanning Calorimeter)

This could be a heat analysing approach which brilliant within live of thermal wanted to adjust the atmosphere of linked diploma case and quotation is figure out in aspect of temp. each the instance and reference are obtained at a pretty similar temperature in direction of test. basic half, temp scenario this probe is made with foremost reason specimen holder temp rasing straight as factor of your period. Hint take a like to be at want to linked degree all-over distinguish heat oppose over the capacity of temp to be filtered.

present strategy is that once sample sense a country alter as an specimen stage developments , only for heath requisite to stream there to than the relevance lift on each at an identical temperature. whether or not or not less or additional heath want to circulation to the occasion depends upon whether or not the procedure is exoergic or endoergic.



Fig:4.8DSC

The mechanism of ultra-sonication was performed continuously for 3 hours. With the help mechanical stirring and ultra-sonication is to split down the
Collected mass of nanoparticles and to control the suspension stable for a more
Time. The period for magnetic stirring and ultra-sonication was fixed by conducting the tests for the various time periods and looking the Sedimentation time of nanoparticles in the nano solvents with and without the
addition of surfactant by keeping them in 10ml glass jar for
22 hours to 7 days.

Energy Dispersive X-Ray study; (EDX), explicit as EDS or EDAX, is given x-ray method accustomed build the essential proportion of materials. Usance embody stuff and material inspection , troubleshooting, DE formulation, and more.

This systems are connect to research appliance (Scanning research (SEM) or Transmission study(TEM)) devices where the imaging effectiveness of the scientific devices finds the specimen of interest. The input produces by this technique inspect contains spectra displaying spike such as weather formulation up being proportion of the sample being checked. Basic mapping of a specimen and image test are potential.

In a multi-technique strategy EDX becomes more strong, greatly in inspection and industrial rhetorical science research. The technique are often qualitative, half-quantitative, quantitative and mutually supply appropriate distribution of elements via mapping. This principal is non-destructive and specimens of interest are normally study in situ with small or no or no specimen making.

RESULTS AND DISCUSSIONS

Methodology

- Studying the key parameters of precipitation method which effect the size and shape of hybrid Nano particles.
- Synthesis of hybrid nano particles
- Characterization of hybrid nano particles
    - Crystalline of material (X-Ray Diffraction)
    - Size and shape (Field Emission Scanning Electron Microscope)

- Average particle size (particle Size Analyzer)

- Dispersing hybrid nano particles with varying mass percentage from 0.2 to 1 with the increment of 0.2 wt% of Heat Transfer Fluid (Water). - (Ultrasonicator)

Raw materials required for synthesis

- Zinc Acetate
- Titanium Oxide
- Sodium Dodecylbenzen Sulfonate (SDBS)
- Aluminum Isopropoxide
- Aluminum Nitrate Nanohydrate

Raw materials procured for the project

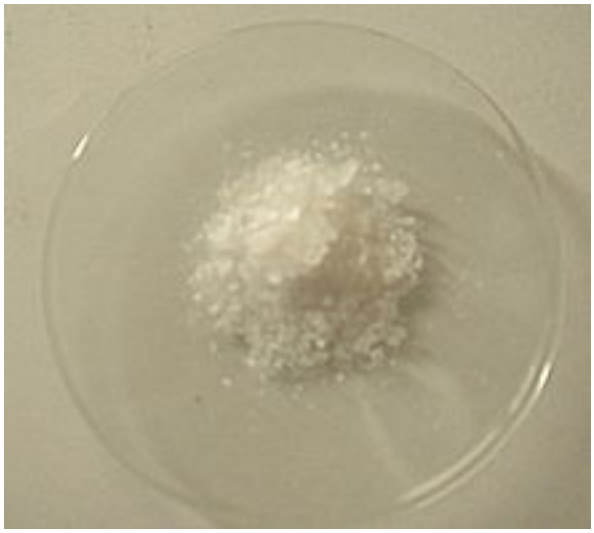


Zinc Acetate



Sodium dodecylbenzen sulfonate

Sodium dodecylbenzen sulfonate is used as reducing agent to reduce the precursor size.



Silver nitrate

Synthesis process of silver nano particles



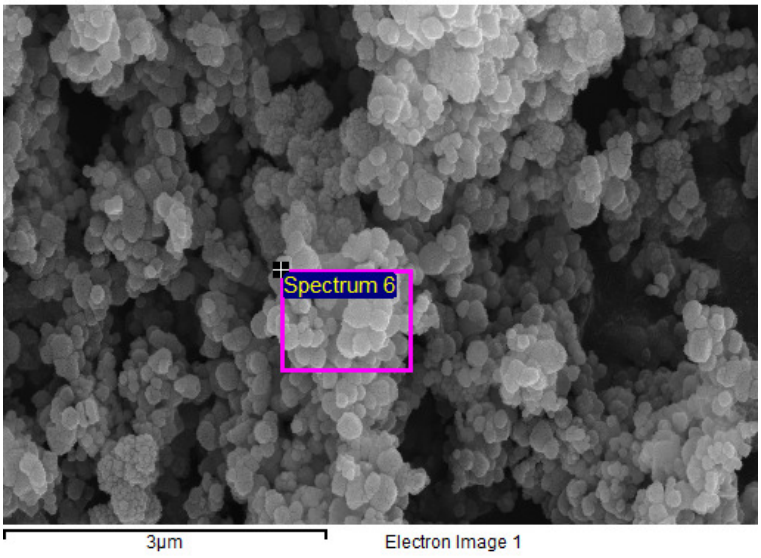


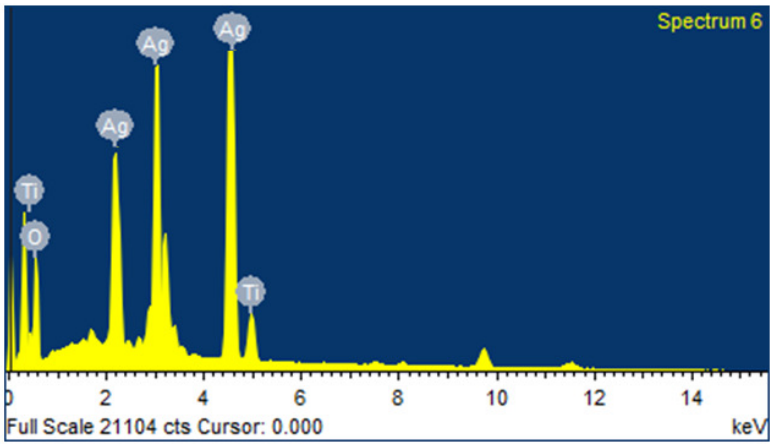
During addition of stabilizing agent and reducing agent, the stirring of precursor should be at 350 rpm with moderate temperature of 50 degree. These stabilizing agent and reducing agent should be added drop by drop slowly.



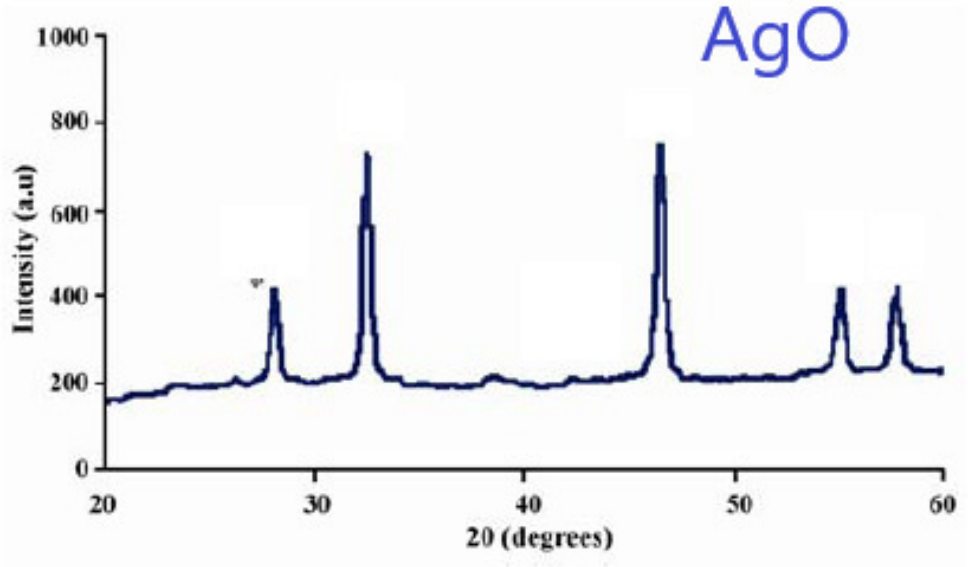
AgO nano powder

EDAX of silver nano particles





XRD of silver nanoparticles



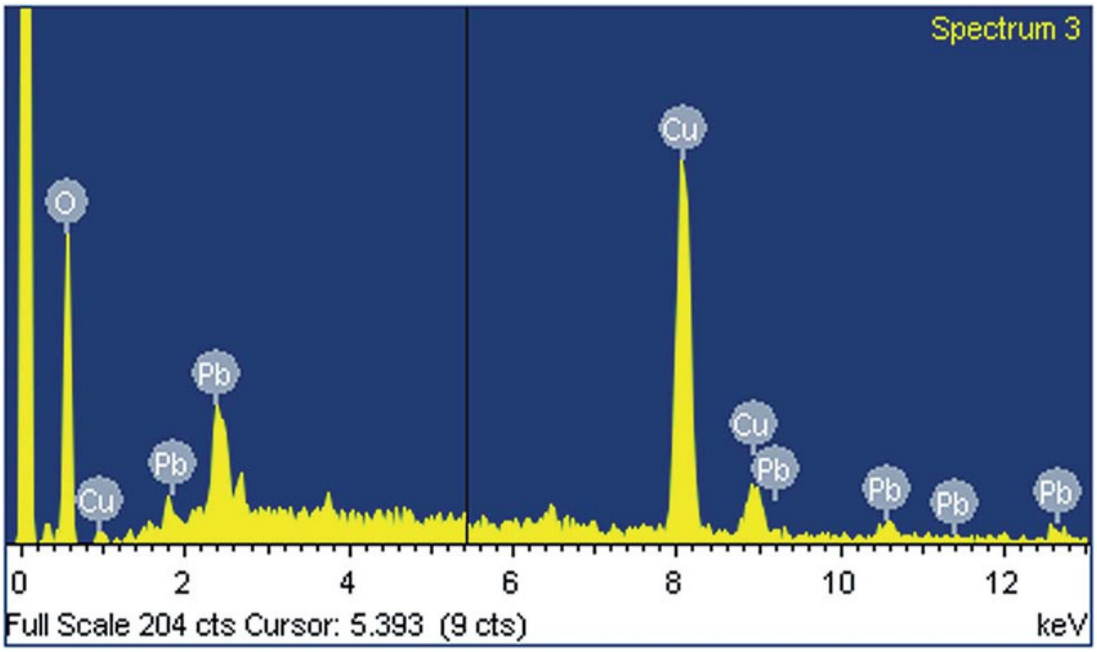
Synthesis process of ZnO nano particles

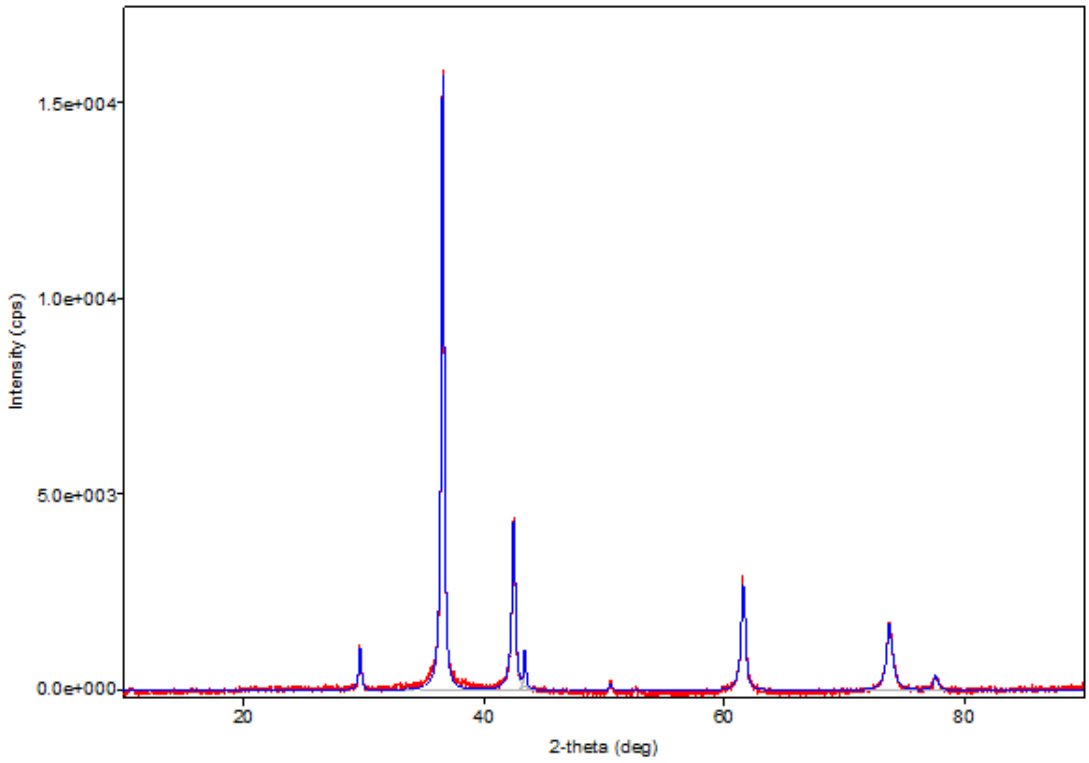




Copper oxide nanoparticles

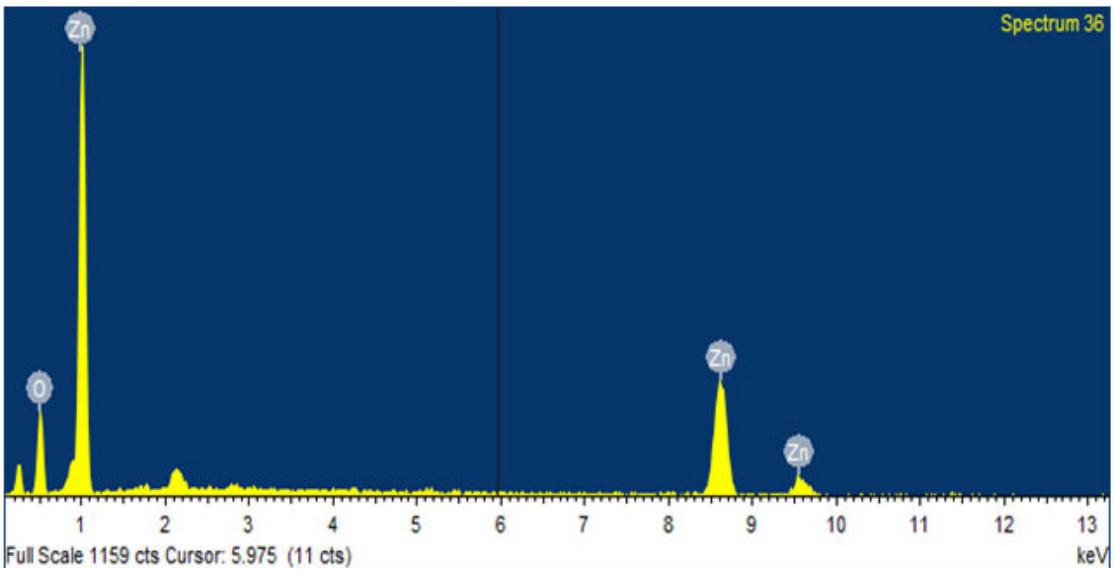
EDAX of Copper oxide nano





XRD of ZnO nanoparticles

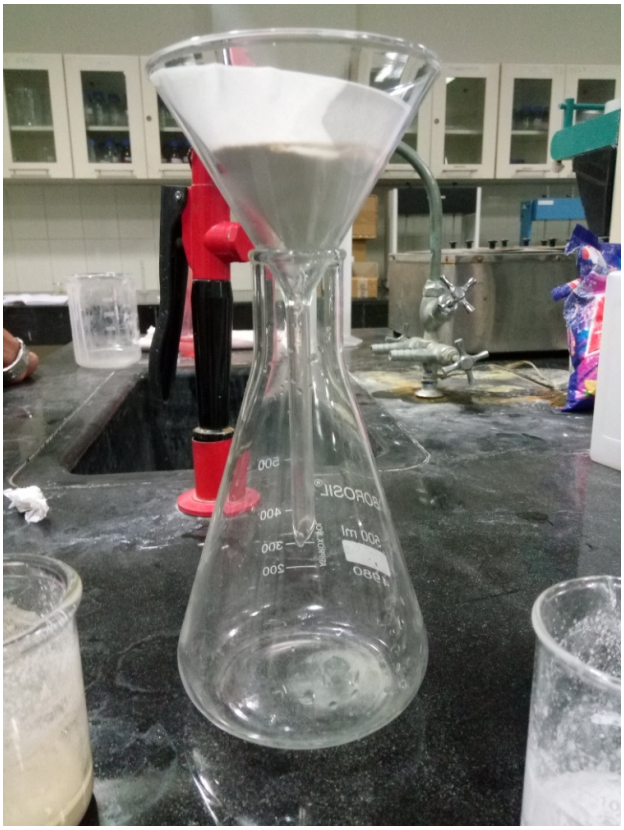
Figure one represents the X-ray diffraction pattern of ZnO nanopowder. a certain line broadening of the XRD peaks indicates that the ready material carries with it particles in nanoscale vary. From this XRD patterns analysis, we tend to determined peak intensity, position and dimension, full-width at half-maximum (FWHM) information.



EDAX of ZnO nanoparticles

Synthesis of TiO2 nanoparticles

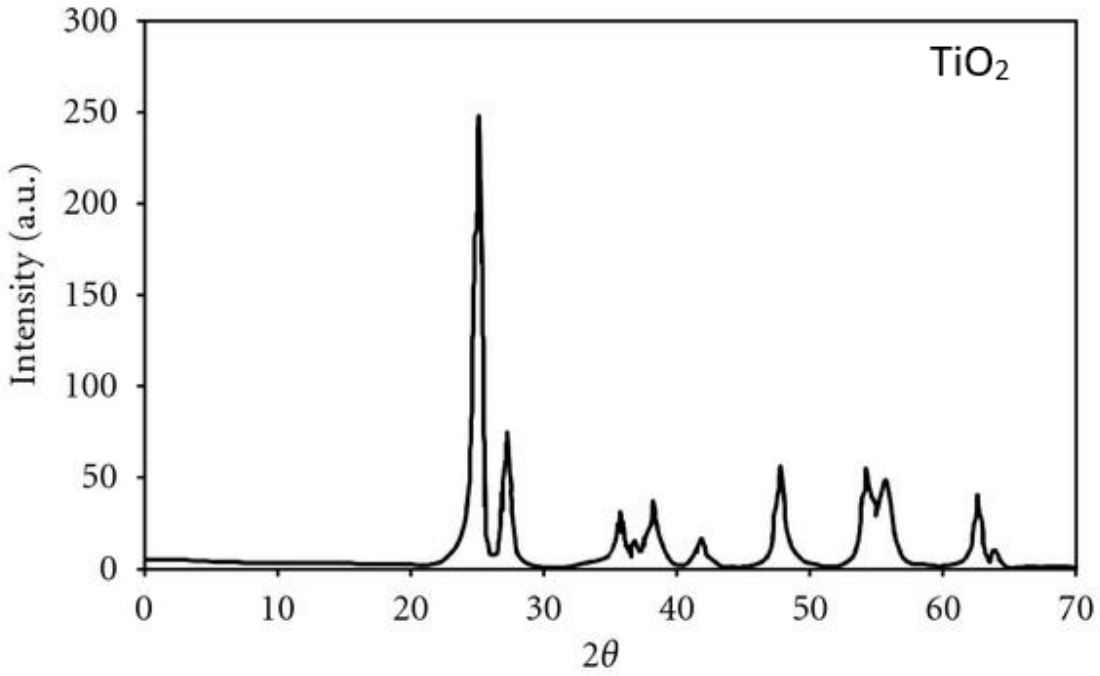




Filtering of nanoparticles



TiO2 nano powder



XRD of TiO2

The XRD pattern Figure affirms the presence of TiO2 nanoparticles. The prominent peaks have been in comparison with JCPDS facts and the peaks received inside the pattern coincides well with the literature. The depth of the peak is high and the width of (a hundred and one) aircraft at becomes slender. The sharpness of the peak uncovers that the samples own a great crystalline nature. The sturdy diffraction height round suggests TiO2 is in anatase segment. The spacious diffraction peak is not located in the patter

Ultrasonicator for dispersing nanoparticles in water



The combination of Silver and zinc oxide nanoparticles of equal proportion were considered in dispersing in water. The main intention is to

Heat transfer characteristics of Ag-ZnO dispersed in water

Nano particles type	Volume Fraction (%)	Density Water	Density NP	% of nanoparticles added	Weight of nanoparticles (gm)	Heat Transfer coefficient	Thermal Conductivity	Reynolds number

Ag-ZnO	0.0001	1000	6310	0.2%	0.0631	2353.048543	0.6588	4993.096
Ag-ZnO	0.0005	1000	6310	0.4%	0.315502	4096.89547	0.698	9986.193
Ag-ZnO	0.00075	1000	6310	0.8%	0.473254	5666.669588	0.718	14979.29
Ag-ZnO	0.001	1000	6310	1%	0.631006	2353.048543	0.728	4993.096

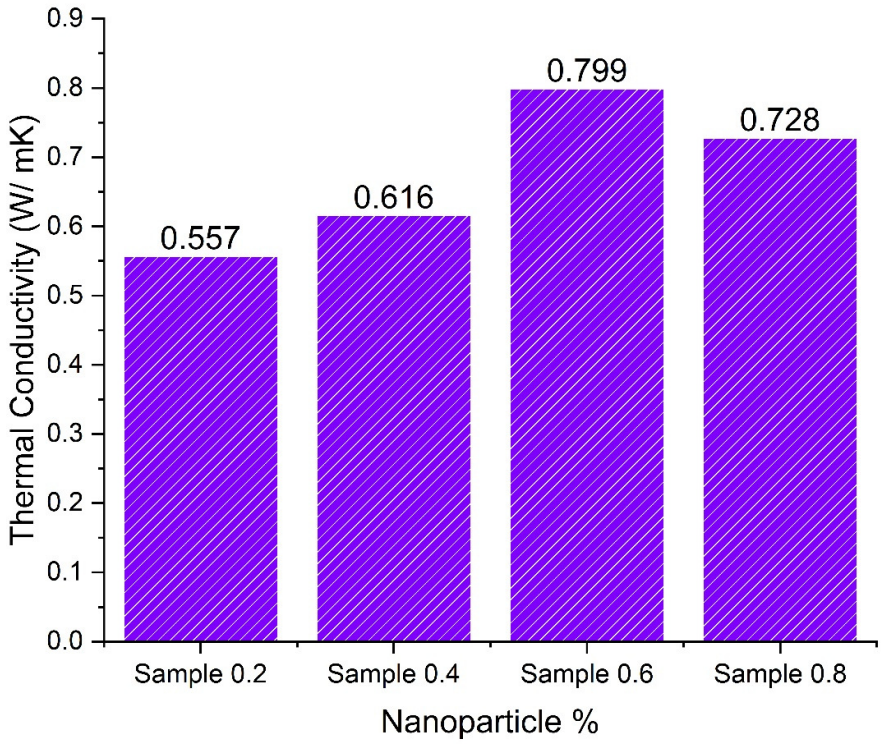
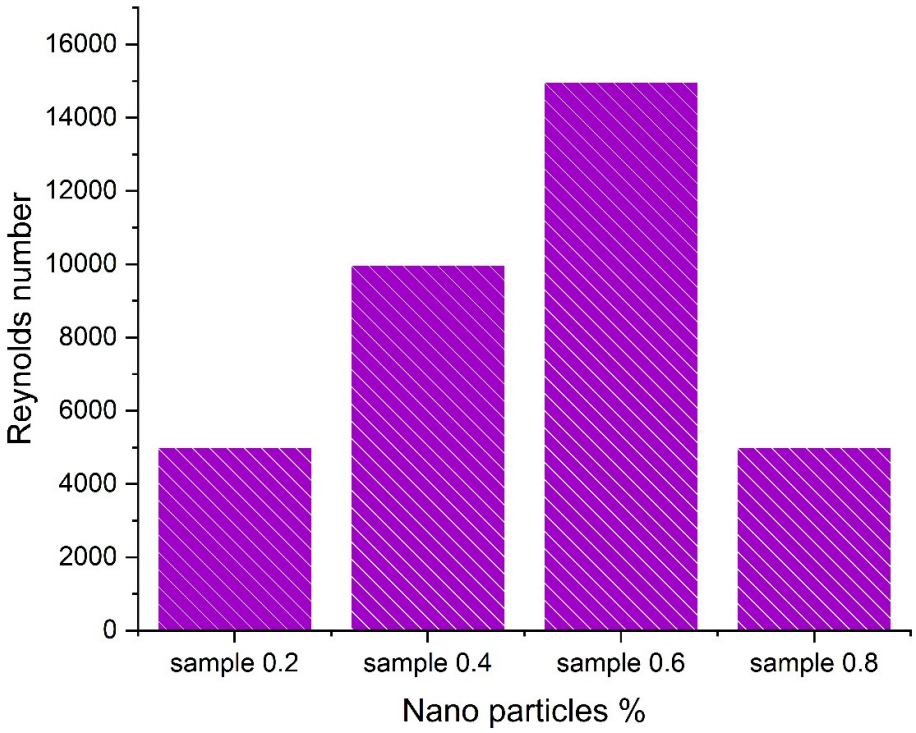


Fig. Ag-TiO2 dispersed nano fluid thermal conductivity



Heat transfer characteristics of Ag-TiO2 dispersed in water

Nanoparticles type	Density Water	Density NP	Weight of water	% of nanoparticles added	Weight of nanoparticles (gm)	Heat Transfer coefficient	Thermal Conductivity	Reynolds number
$AgTiO_2$	1000	5600	0.05	0.2	0.00028	2325.426847	0.557	4993.096
$AgTiO_2$	1000	5600	0.05	0.4	0.0014	4048.803304	0.616	9986.193
$AgTiO_2$	1000	5600	0.05	0.6	0.0021	5600.150338	0.799	14979.29
$AgTiO_2$	1000	5600	0.05	0.8	0.0028	2325.426847	0.728	4993.096

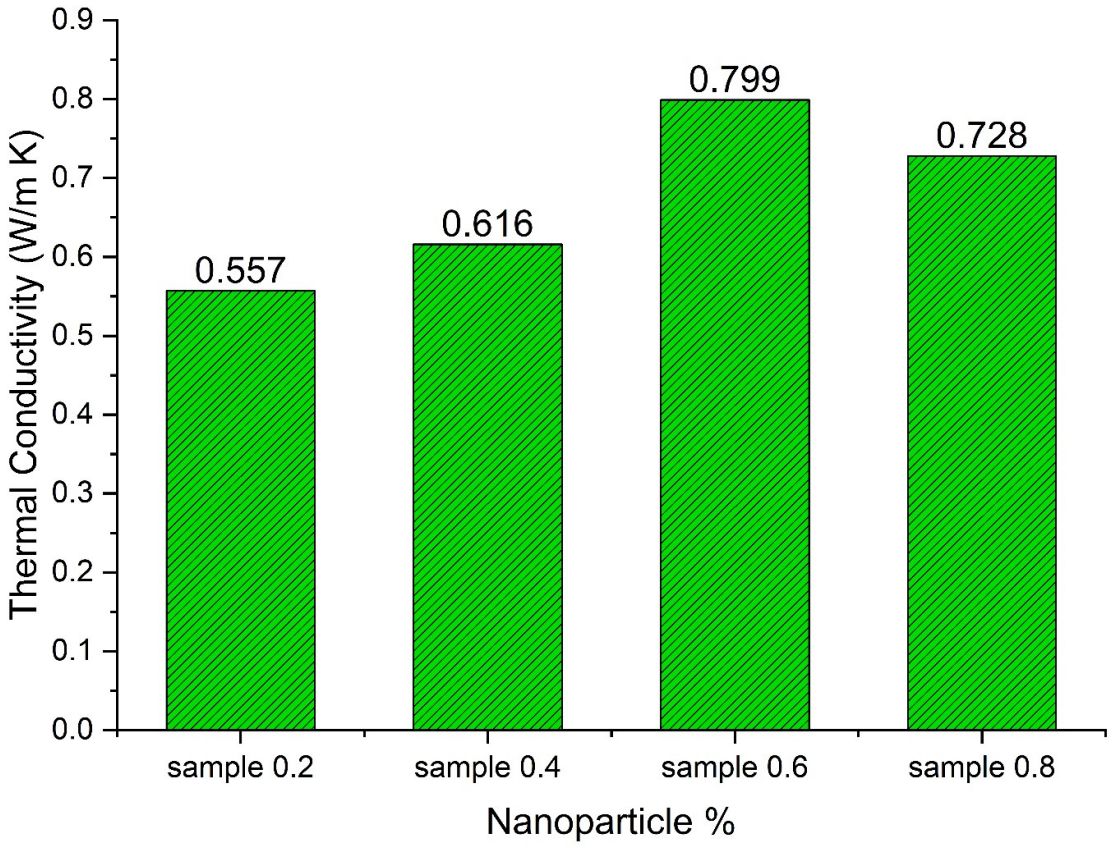

Thermal Conductivity (W/m K)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
0.557
0.616
0.799
0.728
sample 0.2
sample 0.4
sample 0.6
sample 0.8
Nanoparticle %

Fig.

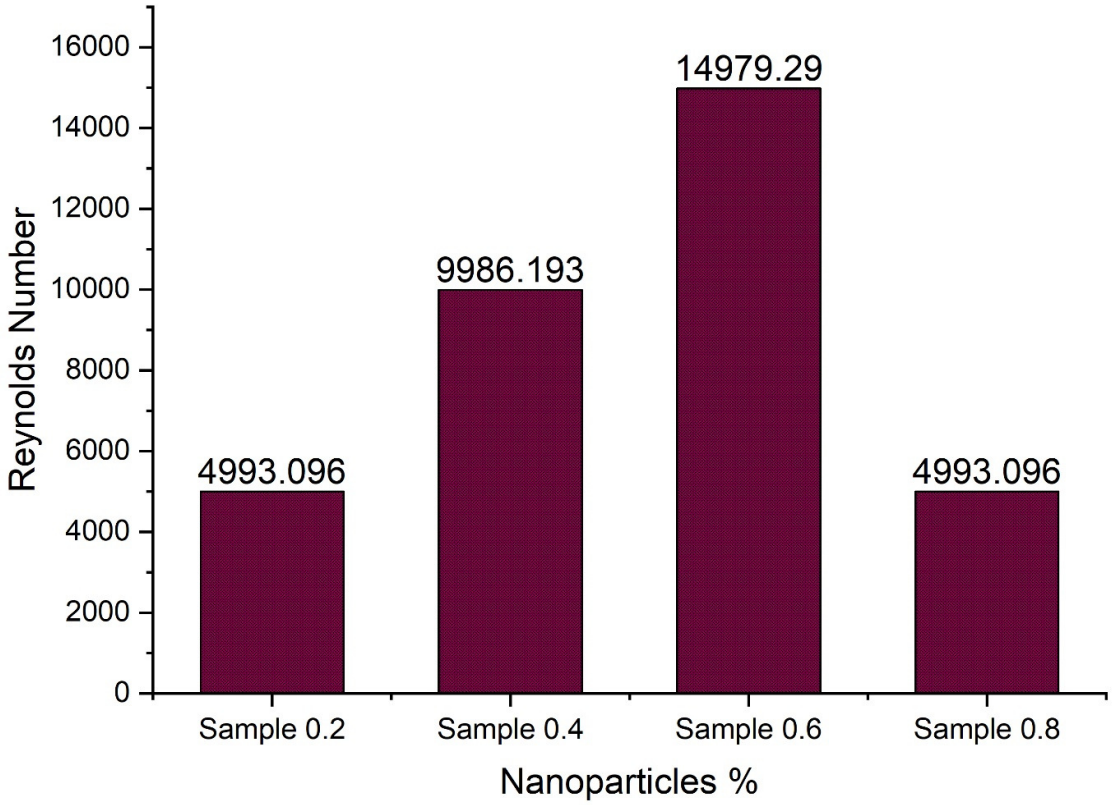


Fig.

RESULTS AND DISCUSSIONS

the consequences non inheritable from the scale of thermal conduction of silver-water nanofluids suggests that the size of the nanoparticles affects the thermal conduction of nanofluids. Decrease long of the nanoparticles showed higher sweetening of thermal conduction of nanofluids. because the extent fraction of nanoparticles can increase, the thermal conduction of nanofluids can increase. A most sweetening of forty dissembling within the thermal conduction has been dead the employment of nanofluids Ag-ZnO containing zero.6 % volume concentration of twenty nm silver nanoparticles. Also, it's miles obtrusive that there exists a strong temperature result on thermal conduction with the tested nanofluid at elevated Ag-TiO2 The thermal conduction of nanofluid can increase from twenty nine you have to thirty ninth. From the planning at on the firmness of silver-zincoxide water nanofluids, it's understood that lower within the length of nanoparticles ends in higher stability of nanofluids. Contrary to the decrease within the thermal conduction sweetening with the addition of nano, nano inclusion additionally improves the soundness of nanofluids. Also, boom within

the notice of wetting agent presents higher stability of nanofluids. it's found that the soundness of nanofluids is additional at lower hydrogen ion concentration fee of nanofluids. letter capability of nanofluids will increase with the addition of wetting agent. Also, average particle length (hydrodynamic length) decreases with the addition of wetting agent. It indicates that additional advantageous stability of nanofluids may be received with the addition of wetting agent. From the geological phenomenon approach, and size of hydrogen ion concentration, letter ability and customary particle length of nanofluids, it is confirmed that addition of nano inclusions enhances the stableness of silver nanofluids. Also, nanoparticles shows fairly higher balance fluid. However, the usage of SDS as wetting agent in nanofluids causes additional producing of froth which might additionally impede the fluid float at intervals the pipe strains of warmth exchangers or another thermal engineering devices. As silver - titania nanofluids with Mass fraction is viable with terribly less decrease in thermal conduction sweetening but advanced bodily stability. From the compelled convective heat transfer examine with silver- oxide water nanofluids, it's miles obvious that the addition of silver nanoparticles in water can increase the convective heat switch constant of the fluid. the warmth switch constant of nanofluid augments with the expansion in Reynolds amount of the fluid. Also, the warmth switch constant enhances with the rise within the concentration of nanoparticles in water. The most upgrades of twenty-two.1, forty four.7 and sixty 5.three % square measure received within the heat switch constant of silver-nanofluids for zero.01, 0.05 and nil.1 % extent attention of silver titania nanoparticles severally compared to water at Reynolds sort of 21000. The sweetening of warmth switch constant is perceptibly large within the turbulent location and fewer in depth within the laminal location. Similarly, the sweetening is slight within the transition location from laminal to turbulent regimes. Because the thermal conduction sweetening magnitude relation is higher compared thereto of the other temperature established homes that embrace density, consistency and heat ability. it's over that a lot of of the transport homes the thermal conduction sweetening plays a primary role in heat switch sweetening.

CONCLUSIONS

1. The sol gel procedure used for various hybrid nano particles were in good agreement. The particles were found to be in nano level.
2. The FESEM results suggest that the particles were in spherical profile with smooth surface.
3. The XRD also confirms that the particles are highly crystalline in nature.
4. The particles dispersed in water were enhancing the thermal conductivity both Ag-TiO2 and Ag-ZnO. The thermal conductivity enhancement is mainly due to increased surface to volume ratio.
5. These nanoparticles were act as an heat carriers transferring form one location to other location.

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