Preparation of Zinc oxide Nanoparticles and its Characterization Using Scanning Electron Microscopy (SEM),Energy – Dispersive X-ray pectroscopy(EDAX) and Fourier Transform Infrared Spectroscopy (FT-IR)

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Abstract

A Shinny white powder Zinc oxide which has unique physical and chemical properties has wide range of applications used in paints, lotions on skin, food, electronics, biological and pharmaceuticals. A regular intake is needed to store it in the body. This paper holds the basic method of how to prepare the zinc oxide nanoparticles combining with the organic compound in an ancient and conventional method. The first part of the paper comprises of the structure seen before the process (structure of Zinc oxide).The next part is the structure obtained after the process gets completed (structure of Zinc oxide with the addend). The Studies are well analyzed by SEM, EDAX and FT-IR. There is a difference seen in the transmittance and in the absorbance level. The morphological study is done using SEM. Further the band gap is investigated by the results obtained.

Keywords: Zinc oxide, addend, EDAX, SEM, FT-IR.

1. Introduction

Richard Feynman in 1959 gave a talk mentioning "there is plenty of room at the bottom". His idea was to bring about the n-number of combinations, the spaces, the structures and its major properties in the micro state, which are unseen or not discussed to the scientific world. The micro state is the real image of the macro state. The invention of Scanning Electron Microscopy (SEM) in the year 1981, gave a new face lift to the emerging photographic methods to study the surface morphology of tiny materials. Nano Science and Nano Technology has brought a revolution in the modern science and engineering. The combination of Zinc oxide nanoparticles with the organic compounds depicts the various sizes and shapes when they form. In recent years the application of Zinc oxide in various fields has influenced its performance among the family of elements putting it front. The optical, mechanical, chemical property of Zinc oxide brings forward its high chemical and mechanical stability too. Broad range of absorption is exhibited by Zinc oxide nanoparticles.

Structures are predominantly obtained in one dimensions 1D, two dimensions 2D and three dimensions 3D. Structures like needles, helixes, nanorods, ribbons, belts, wires which come under one dimensional category are obtained from zinc oxide. The nanoparticles of Zinc oxide are seen in the form of Nano pellets, Nano-sheets, Nano plates, rectangular logs, spherical spheroids [1]. In terms of production of Nano Zinc oxide (nZnO) it takes the third place next to Nano Silicon dioxide (nSiO_2) and Nano Titanium dioxide (nTiO_2). The presence of Nano materials in the scientific market gives a platform to think about being an agent towards antibacterial activity. Zinc oxide is commonly added in sunscreens, coatings and paints to absorb UV light.

Products depending on bacteria, fungi, plants and yeasts always result in the production of nanoparticles excellently [2].

2. **Experimental**

a. Materials used

Preparation of Nano particles is focused here in simple manner. Zinc oxide powder (size in microns), water (Polar Solvent), magnetic stirrer.

b. Sample Preparation [A]
(Reduction of size of the Zinc powder from microns to Nano particle size)

Zinc powder is taken and then dried in shade for more than an hour, this is done to make the powder dry. Water is taken as the polar solvent, the dried zinc powder is mixed with water. This mixture is stirred for more than five hours. This is done to make the solute get mixed with the solvent. The obtained mixture is poured in plane pane and it is allowed to dry in shade. This mixture takes its own time to get dried up, nearly for more than ten days. Water content gets reduced due to evaporation. The need for the polar solvent is, it breaks the bulk segments in the zinc powder to smaller units. Obviously the size gets reduced. Here the tank water is used rather than distilled water. This leads to the presence of few other elements seen in the results.

c. Sample Preparation [B]
(Sample is prepared using leaf extract)

Leaves of lemon tree are taken, and then it is cleaned by washing it with water. Then the leaves are dropped into a bowl containing water. This system is exposed to heat, on boiling this the color of the solution gets changed. This clearly tells the presence of Nano particles. As we use the leaves of lemon tree it is obviously yellow in color (leaves are green but the extract obtained is yellow in color).Hence we get the solution the leaf extract. Zinc powder is now mixed with the extract and stirred well for nearly five hours. As we get the mixed up solution, this is kept for drying state. To obtain this, the mixed up solution is poured into plane pane. This system is kept in a shady region for the water to get evaporated. It takes more than ten days for evaporation. Finally the sample is obtained[4].

3. **Measurements**

Scanning Electron Microscopy (SEM) images of Zinc oxide powder nanoparticles and Zinc oxide with leaf extract sediment is taken. Energy dispersive X-ray spectroscopy EDAX from TSL Ametek for samples A, B is taken.
The FTIR study for the samples A,B is taken.

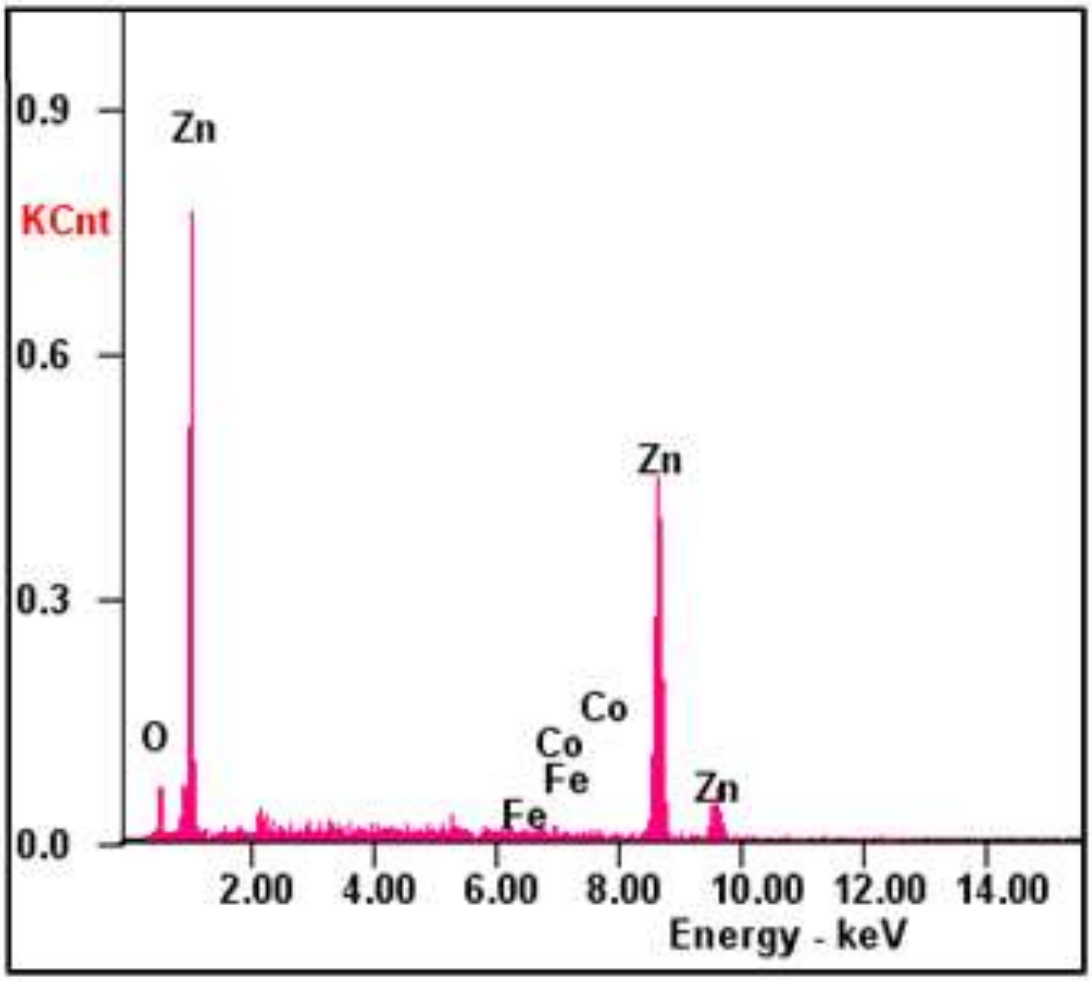
Sample A - - zinc oxide , Sample B – zinc oxide mixed with lemon extract

4. **Results and Discussions**

EDAX and Scanning Electron Microscopy (SEM) image of Zinc oxide powder is taken. Fig. 1 and Fig. 2 reveal the EDAX parameters of the samples A,B. The energy-dispersive X-ray spectrometer spectrum of zinc oxide nanoparticles is taken, that is the sample A. Whereas the study of EDAX tells us about the composition that is seen in the sample. The elemental composition is quite understood by having this result. Fig. 3 and Fig. 4 reveals the SEM image of the samples A,B shows a difference, there are some cracks seen on the surface of the Nano particles B, but the cracks are not seen on the surface of Nano particles sample A. This could be the reason that the biomolecules have drastically shown an impact on the surface of the sample B. The organic substance that is obtained from the leaf extract has brought these cracks on the sample B whereas we don't see it on sample A. The energy dispersive X-ray spectrometer spectrum shows the composition of elements that is seen in the samples. As tap water is used the presence of Co and Fe are seen in the figure. 1.

4.1 **EDAX ANALYSIS**

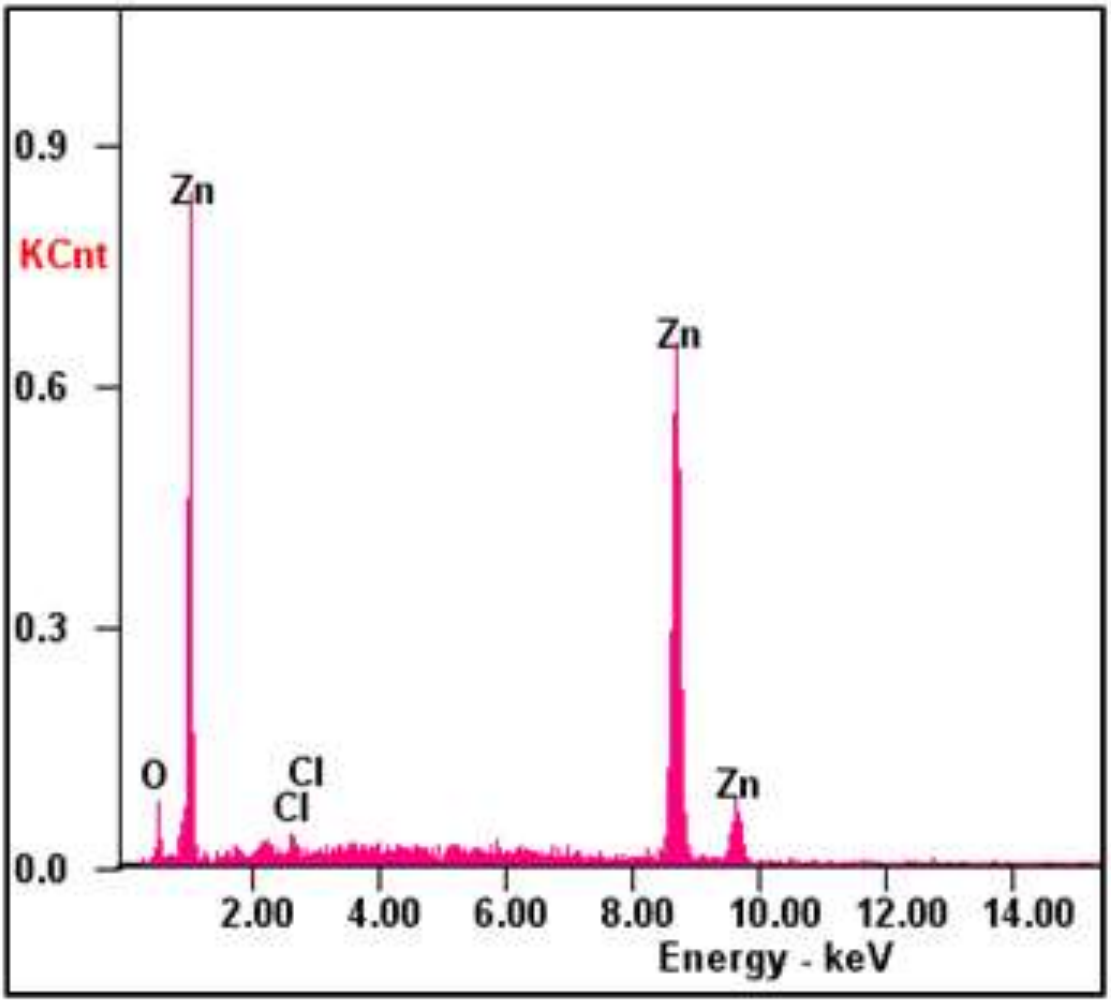
EDAX as we know it shows the composition of the elements in the sample, here the



Element	*Wt%*	*At%*
OK	14.87	41.58
FeK	00.84	00.67
CoK	00.80	00.61
ZnK	83.49	57.14
Matrix	Correction	ZAF

Fig.1 - **Energy-dispersive X-ray spectrometer spectrum of zinc oxide nanoparticles**

elements shown in the table has the preceding letter K . This letter K implies to the K shell, whereas the electrons from the K shell gets knocked out when the sample is exposed to the incoming electrons (which is the bombarding electron). As there occurs vacancy getting formed in the K shell the neighbouring electrons from the shells L, M,N may jump into the K shell to occupy the spaces[3-4].



Element	*Wt%*	*At%*
OK	10.96	33.20
ClK	01.24	01.70
ZnK	87.80	65.11
Matrix	Correction	ZAF

Fig.2 - **Energy-dispersive X-ray spectrometer spectrum of zinc oxide mixed with lemon extract nanoparticles**

The movement of the electrons from the neighbouring shells to the K shell will release the X-rays. As this process happens at the regular rate, we get the peaks seen in the graph for that element from that particular shell. At% is the atomic percentage which tells about the number of atoms in terms of percentage[5]. Whereas the Wt % is the weight percentage which tells the function of weight of the atoms. On adding, we get the total to be 100%, this gives the clear picture of the composition of the elements in the sample.

4.2 **SEM ANALYSIS**

The Sample A,B are clearly analyzed using SEM. The cracks caused by the organic compound

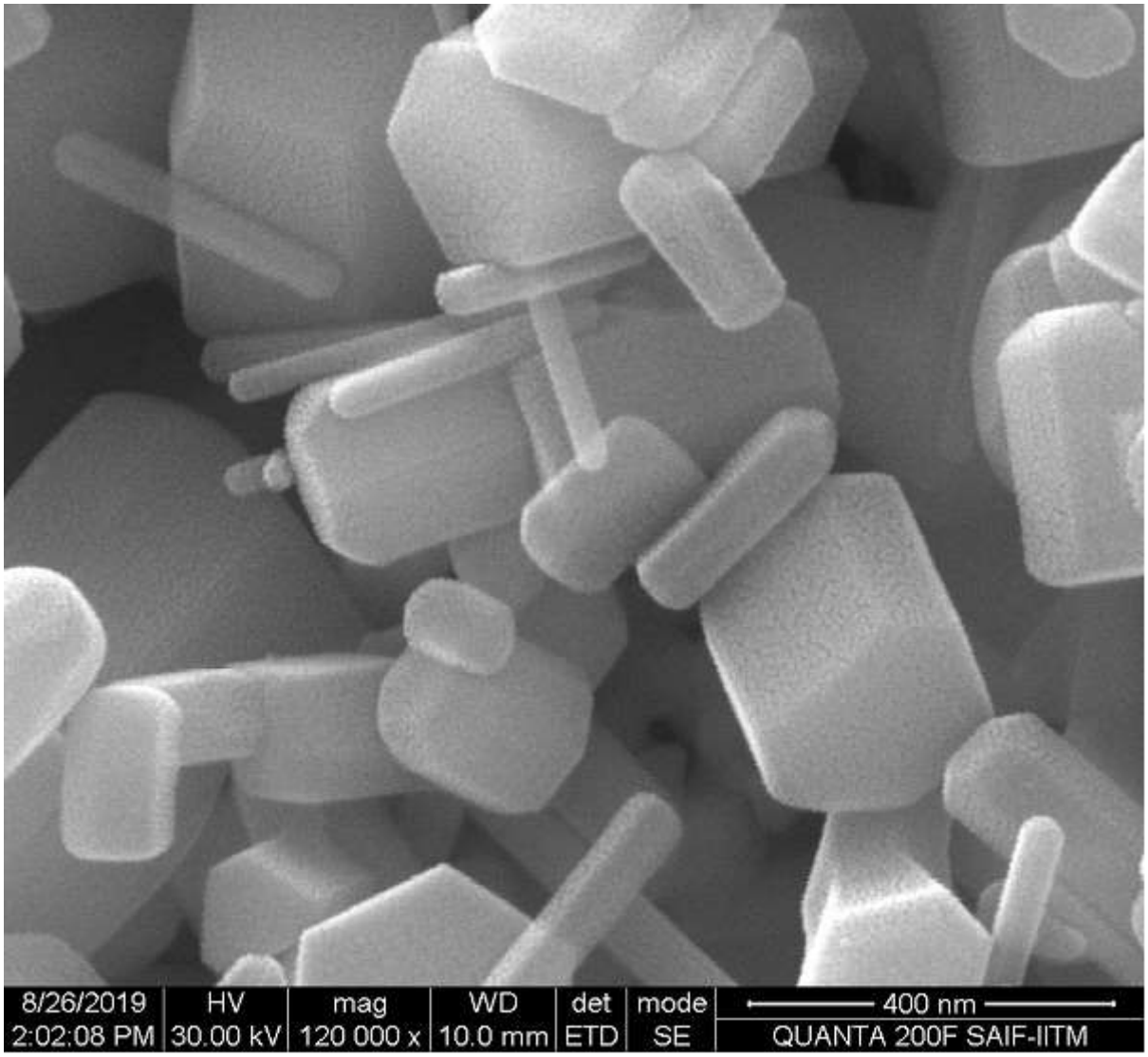


Fig. 3 - **SEM- image of zinc oxide nano particles**

is seen in the SEM image of the sample B. Whereas we don't find the legible cracks on sample A.The shapes shown in the picture gives us a clear idea of the structures, solid forms taking the rectangular, hexagons, cuboid and few pellets shapes[6-7].

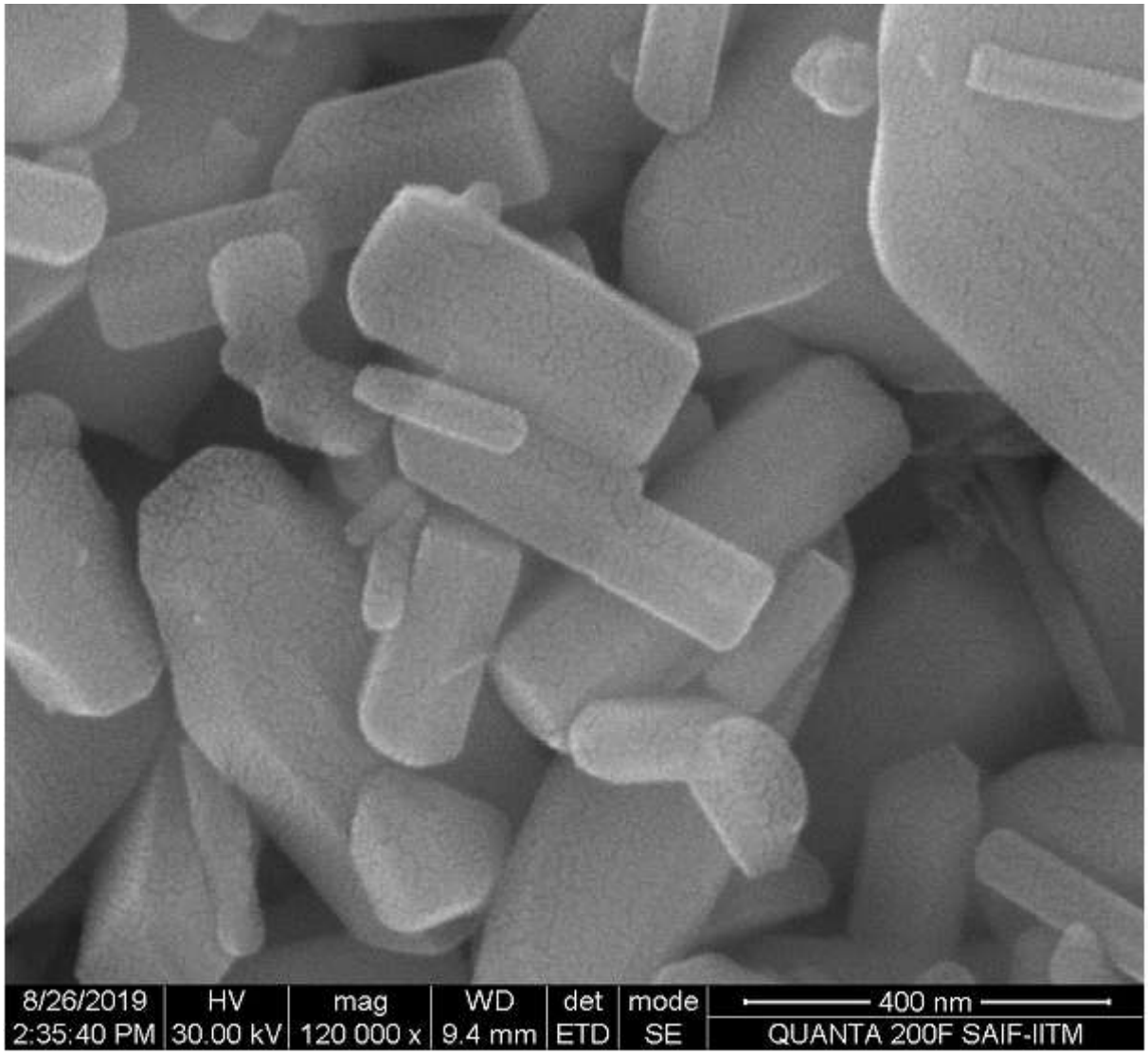


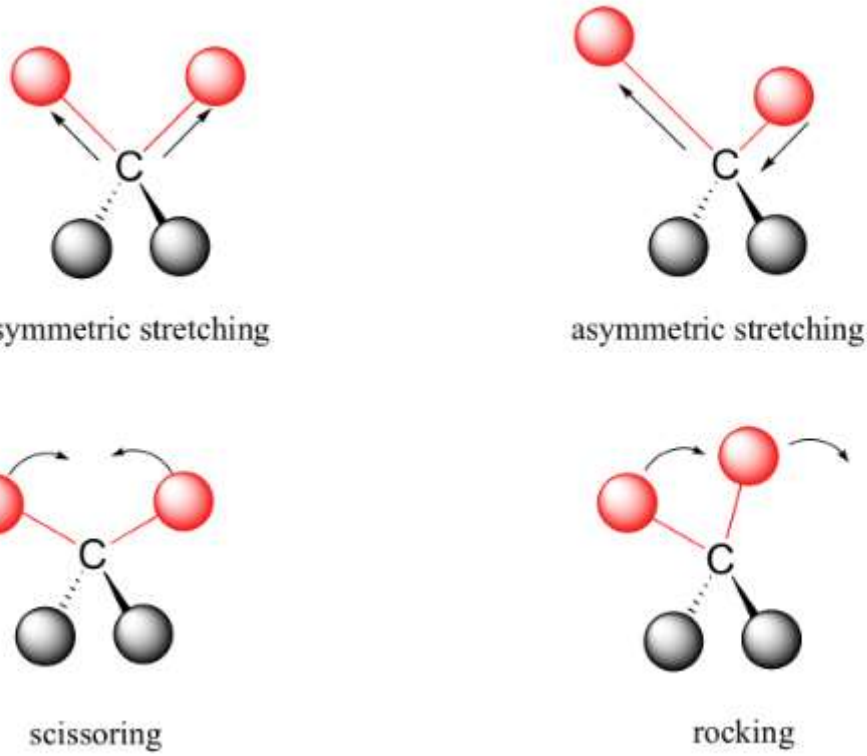
Fig.4 - SEM-image of Zinc oxide mixed with lemon extract nanoparticles

Both the samples are taken in the 400 nm magnification, this is done to have a comparative study of the samples with no deviation.

4.3 **FTIR ANALYSIS**

The peaks shows the occurance of metal oxide and we know that ZnO is a metal oxide.The major peaks are to be seen below 1000 cm^{-1} to ensure that ZnO is a metal oxide. The spectrometer (Bruker) is used to find out the FT-IR analysis. We got nearly eight peaks for the sample A, this sample is an undoped or pure sample.

The obtained peaks fall in the range of 560.87 cm^{-1} to 3709.97 cm^{-1}. Stretching and Bending concepts are seen in the results. It is a known fact that it is easy to bend a bond rather than to stretch or on the other hand to compress[3].



The energy of vibrations seen in the molecular region is more over quantized than it is continuous, this put forth that a molecule can stretch or else bend at only at certain allowed frequencies. Here the molecule when exposed to electromagnetic radiation, if there occurs a matching with the vibrational modes, then it will absorb the energy from the radiation and jump to a higher vibrational energy state.

The amplitude will increase but the vibrational frequency will still remain the same. On having a note the difference in energy between the two vibrational states is fundamentally equal to the energy associated with the wavelength of the radiation that was absorbed. Figure -5 shows the undoped zinc oxide that is the Sample- A, whereas the Figure - 6 shows the organic doped material zinc oxide with lemon extract that is the Sample B.

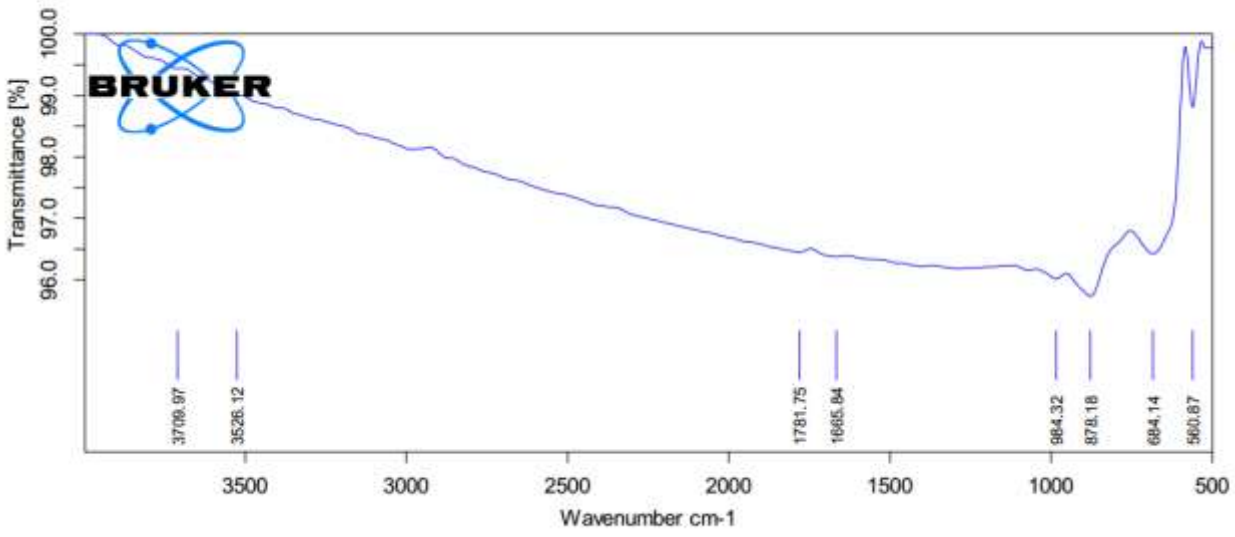


Fig. 5 - **Fourier transform infrared spectroscopy spectrum of zinc oxide nanoparticles**

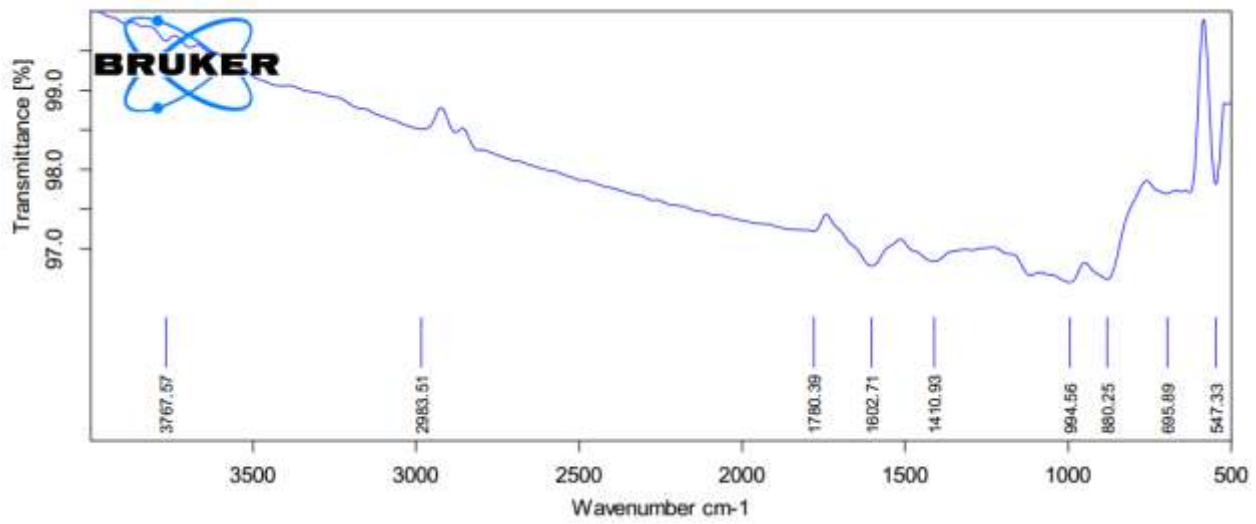


Fig. 6 - **Fourier transform infrared spectroscopy spectrum of zinc oxide mixed with lemon extract**

Fourier transform infrared spectroscopy spectrum of zinc oxide nanoparticles

S.NO	ABSORPTION PEAKS OBTAINED FOR ZnO NANO PARTICLES (cm-1)	STANDARD ABSORPTION FREQUENCY RANGE (cm-1)	APPREANCE	GROUP
1	560.87	600-500	STRONG	C-I stretching
2	684.14	730-665	STRONG	C=C bending
3	878.18	900-860	STRONG	C-H bending
4	984.32	1000-650	STRONG	C-H bending
5	1665.84	1675-1665	WEAK	C=C stretching
6	1781.75	1800-1770	STRONG	C=O stretching
7	3526.12	3550-3200	STRONG,BROAD	O-H stretching
8	3709.97	4000-3000	MEDIUM,SHARP	O-H stretching

Fourier transform infrared spectroscopy spectrum of zinc oxide mixed with lemon extract nanoparticles

S.NO	ABSORPTION PEAKS OBTAINED FOR ZnO NANO PARTICLES (cm-1)	STANDARD ABSORPTION FREQUENCY RANGE (cm-1)	APPREANCE	GROUP
1	547.33	690-515	STRONG	C-Br stretching
2	695.89	730-665	STRONG	C=C bending
3	880.25	900-700	STRONG	C-H bending
4	994.56	995-985	STRONG	C=C bending
5	1410.93	1440-1395	MEDIUM	O-H bending
6	1602.71	1650-1580	MEDIUM	N-H bending
7	1780.39	1800-1770	STRONG	C=O stretching
8	2983.51	3000-2840	MEDIUM	C-H stretching
9	3767.57	4000-3000	MEDIUM,SHARP	O-H stretching

5. CONCLUSION

By the simplest method the Zinc Oxide nanoparticles are prepared (this method can also be called as a slow evaporation method). The pure Zinc Oxide is sample A and the sample B is Zinc Oxide mixed with Lemon extract an organic compound. Study of EDAX with the samples have clearly shown us the composition of the elements present in the sample. Whereas the SEM image shows the minor cracks that is seen on the sample B due to the additive lemon extract. The FTIR study reveals us that peaks gets heaped out at a point 2983.1 wavenumber cm^{-1} whereas the peak sinks out at a point 1602.71 wavenumber cm^{-1}, this is highlighted in sample B on comparison with sample A .As discussed these occur due to the bonding vibration caused by the radiation which is applied on the sample.

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