Optimized Synthesis and Comprehensive Characterization of Zirconium Oxide Nanoparticles via Co-Precipitation Method

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ABSTRACT

The crystalline zirconium oxide (ZrO_2-KN) nanoparticles were synthesized using an optimized content of zirconium oxide with varying KOH concentrations via the co-precipitation method. The thermal history of the precursor was carefully analyzed through various measurements. The as-prepared samples were characterized to ensure structural, functional, morphological, and compositional properties using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and X-ray photoelectron spectroscopy (XPS). The monoclinic structure with space group $P2_1/c$ was confirmed from XRD (JCPDS 89-9066). The Zr–O stretching vibration and Zr–O_2–Zr bending vibration were identified through FTIR analysis. The well-dispersed particles with spherical morphology were confirmed by SEM analysis. The oxidation states of Zr-KN, O, and C were verified using XPS analysis. Furthermore, atomic force microscopy (AFM) analysis was used to study the nanomechanical properties of the nanoparticles, improving nano-formulation.

Keywords: Zirconium oxide, XPS, AFM, Nano formulation, Co-precipitation method

1.INTRODUCTION

Nano materials are considered as intermediate between classical molecular scale and micron sized entities. The synthesised of nano materials with structural stability are great importance with unique physical and chemical properties in comparison with those of their bulk counterparts, and their properties based on quantum size effect and high surface area [1-3]. Recently, many studies performed on the oxide material such as TiO_2, Al_2O_3, ZnO and ZrO2-KN etc., among these, Zirconia is very fascinating material in current technology. It has been renowned as a high-eminence in energy storage due to its thermal, chemical stability and outstanding mechanical properties such as high strength and crack robustness, high

melting point, low thermal conductivity, high coefficient of thermal expansion, high resistance to rust and high fracture toughness. ZrO2-KN is always a very significant ceramic material. Reduction of the dimensionality from two to one (quantum wires) and to zero should lead to further improvements [4]. The carriers are confined in a very small volume and population inversion occurs more easily, leading to lower threshold currents for lasing. Furthermore, the emission wavelength can tuned by simply varying the dimensions of the dot (or well). The main difficulty is to ensure that the dots comprising a device are uniformly sized. Quantum wires have been used in a wide range of applications, including nanoelectronics, photonics, and sensing. In nanoelectronics, quantum wires can be used to improve the performance of transistors and interconnects [5-8]. In photonics, quantum wires can be used to confine light in Nano scale waveguide, leading to enhanced light-matter interactions. In sensing, quantum wires can be used to detect small changes in the electrical or optical properties of the material, leading to highly sensitive sensors.

2.CO- PRECIPITATION METHOD:

Chemical precipitation involves the addition of chemical reagents, followed by the separation of the precipitated solids from the solution. Precipitation is the creation of a solid in a solution or inside another solid during a chemical reaction or by diffusion in a solid. When the reaction occurs in a liquid solution, the solid formed is called the 'precipitate'. The chemical that causes the solid to form is called the 'precipitant'. Without sufficient force of gravity to bring the solid particles together, the precipitate remains in suspension [9]. This method involves dissolving a salt precursor (chloride, nitrate, etc.) in water (or other solvent) to precipitate the oxo-hydroxide form with the help of a base. Very often, control of size and chemical homogeneity in the case of mixed- metal oxides are difficult to achieve. However, the use of surfactants, sonochemical methods, and high-gravity reactive precipitation appear as novel and viable alternatives to optimize the resulting solid morphological characteristics.

3.RESULTS AND DISCUSSION

3.1 UV STUDIES

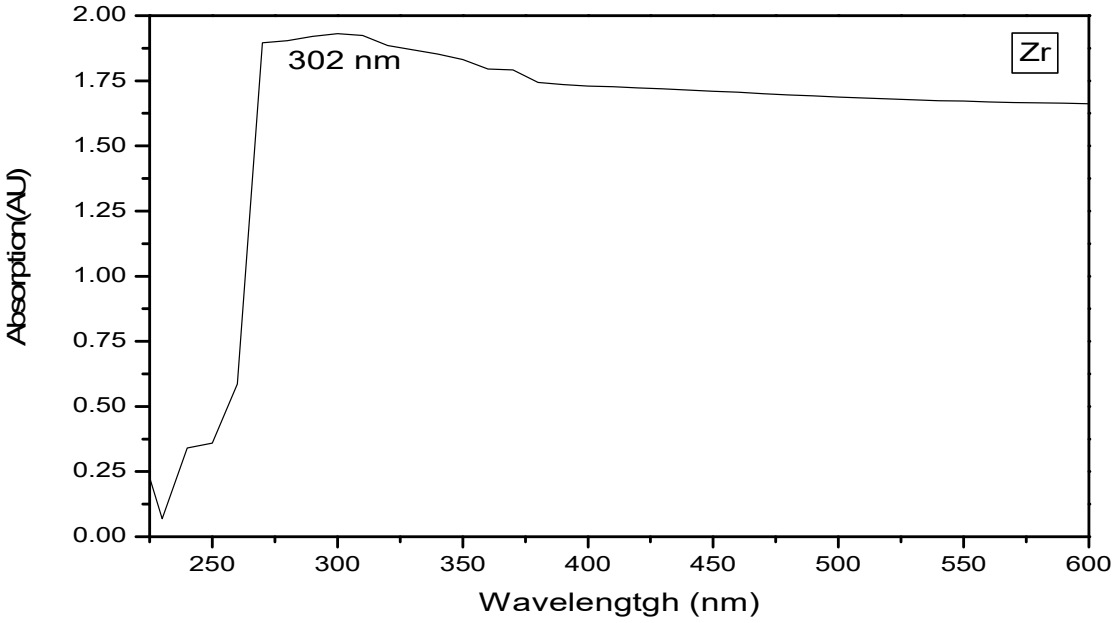


Figure.1.OPTICAL ABSORPTION SPECTRUM

The UV-Vis reflectance spectroscopy is used to study the optical properties of the synthesized nanoparticles. ZrO2-KN nanoparticles were suspended in water and kept in ultrasonication for 5 minutes. The Absorption spectra of ZrO2-KN nanoparticles was recorded using UV-Vis spectrophotometer over the wavelength range 200-800 nm. The UV-Vis spectrum is shown in Figure 1, From this spectrum, it has been inferred that ZrO2-KN nanoparticles has sufficient transmission in the entire visible and IR region. The absorption peak was found in the wavelength of 302 nm. This can be assigned to the intrinsic band gap of ZrO2-KN due to the electron transition from the valence band to the conduction band [10]. The band gap energy E_g is an important property as it determines their applications in electronics.In crystalline materials the E_g value is directly obtained from the absorption peak in the UV-Vis region. The energy band gap of the ZrO2-KN was calculated using the Planck's equation. The energy band gap of the synthesized, ZrO2-KN nanoparticles was calculated using the Planck's equation.

$$E_g = hc/\lambda.$$

The corresponding band gap of the synthesized, ZrO nanoparticles was found to be from 4.1 to 5.69 respectively for the temperature ranges from 452ºC to 500ºC. It's a luminescent Material with good optical transparency [11].

3.2 FTIR

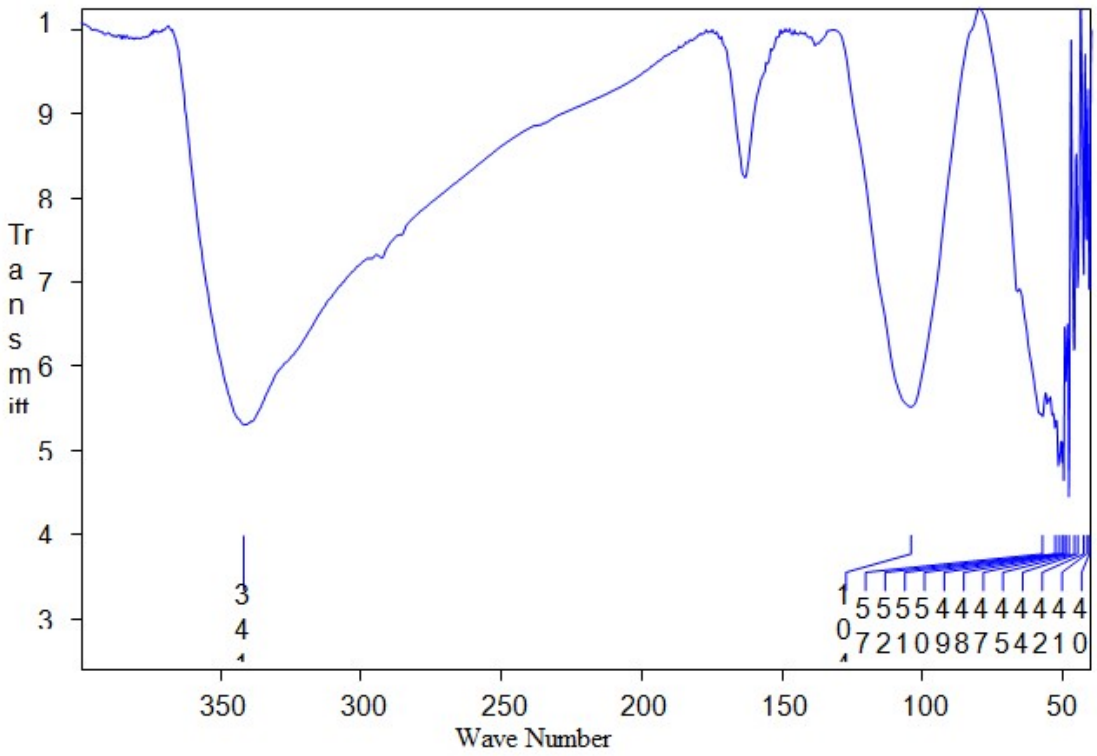


FIG.2.FTIR spectra of ZrO2-KN

Fourier transform infrared spectroscopy was used to detect the presence of functional groups adsorbed on the surface of synthesized nanoparticles during precipitation process. The FTIR spectrum of the synthesized ZrO2-KN nanoparticles was recorded in the range 500 - 4000 cm^{-1} using KBr pellets and the spectrum has been shown in Figure 2. The transmittance peak at 3416.12 cm^{-1}corresponds to the O-H stretching vibration.

The peak at 1742.18 cm^{-1}, 1708.64 cm^{-1} is attributed to C=O stretching vibrations. The absorption peak at 1676.52 cm^{-1} is due to -C=C- stretching vibrations. The absorption peak at 1040.02 cm is assigned to C-C stretching vibration. The storng bands at 415.48 cm-405.23 cm-1 corresponds to stretching vibration.

3.3 XRD

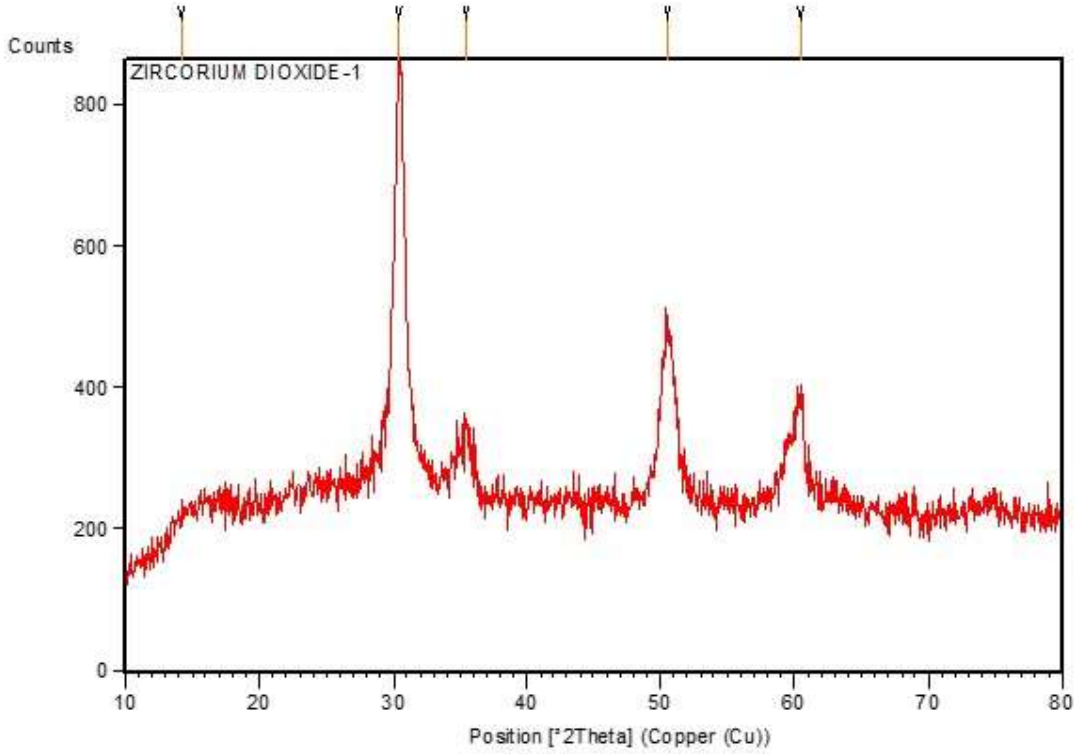


Fig.3 XRD Graph of ZrO2-KN at 450°C

X-ray diffraction is a non-destructive and analytical method for identification and quantitative analysis of various crystalline forms of nanoparticles, also known as phases of the compound present in the samples.All the diffraction pattern peaks indexed to monoclinic ZrO2-KN(JCPDS:89-9066). The average size of the particle was calculated using the Debye Scherrer formula

$D = K\lambda / \beta\cos\theta$ D = the particle size,
k = a constant of (0.9), λ = the X-ray wavelength (1.54 Å),
θ = the Bragg's angle in radians, β = the full width at half maximum of the θ peak used.

The average size of the synthesized ZrO2-KN nanoparticle were calculated to be 40.89 nm, 22.70 nm, 31.38 nm respectively. FWHM increases with decrease the crystalline size [12]. The crystalline size decreases in a linear manner and is shown in Figure. For the ZrO2-KN nanoparticles it starts from 35nm to 40nm, The nanoparticles reported from 36nm to 13nm for 450°C.

3.4 SEM

It is important to know the exact nature of nanoparticles formed and this can be deducted from the SEM image of the sample. The morphology, structure and size of the samples can be investigated by SEM. Figure .4 shows that the ZrO2-KN nanoparticles were spherical in shape. The size of the particle were calculated by drawing a line in the image and then dividing the length of the line by the number of grains and the average size was calculated to be 1µm to 200nm for the ZrO2-KN nanoparticles respectively for the temperatures 450ºC. Also observed two kinds of particle morphologies small and secondary particles hexagonal like size approximately 0 .42 micrometer . It can be concluded that the catalyst played a pivotal role in the surface morphologies of the ZrO2-KN powder.

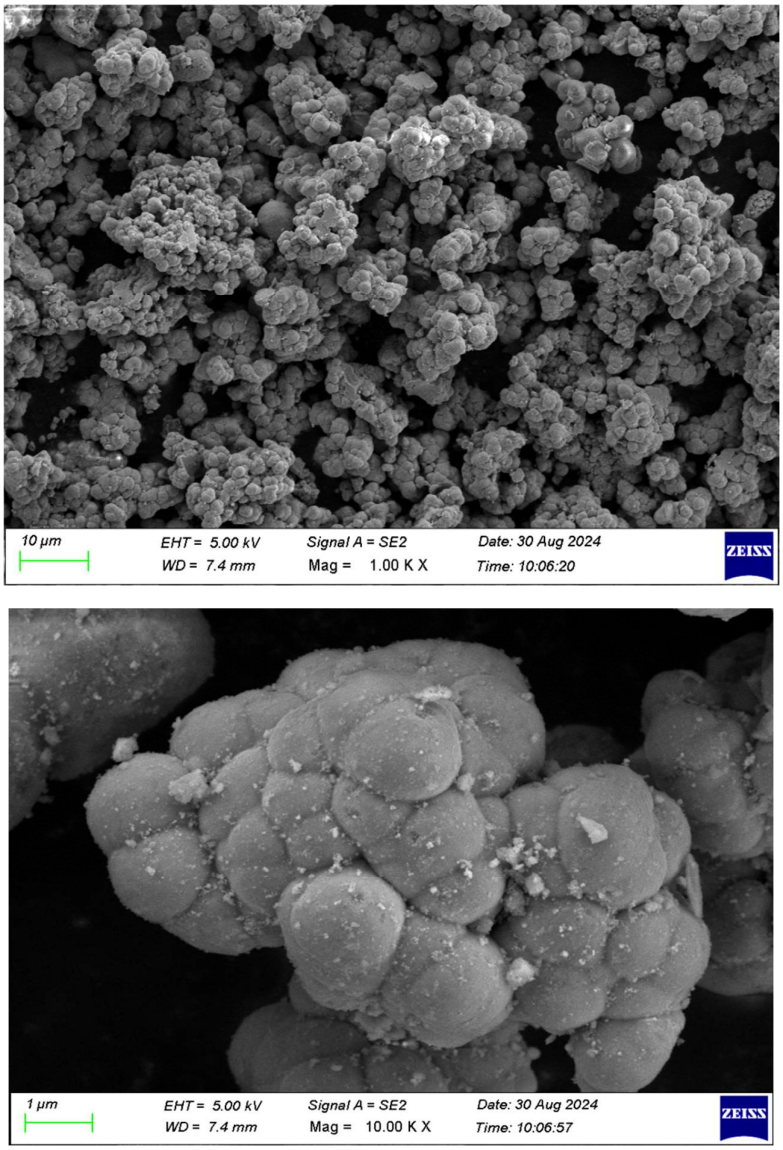
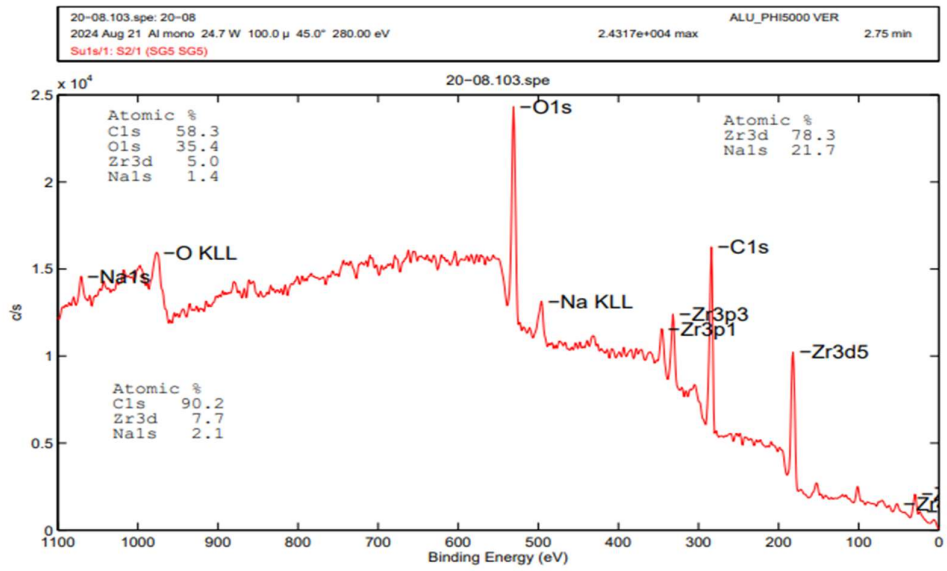
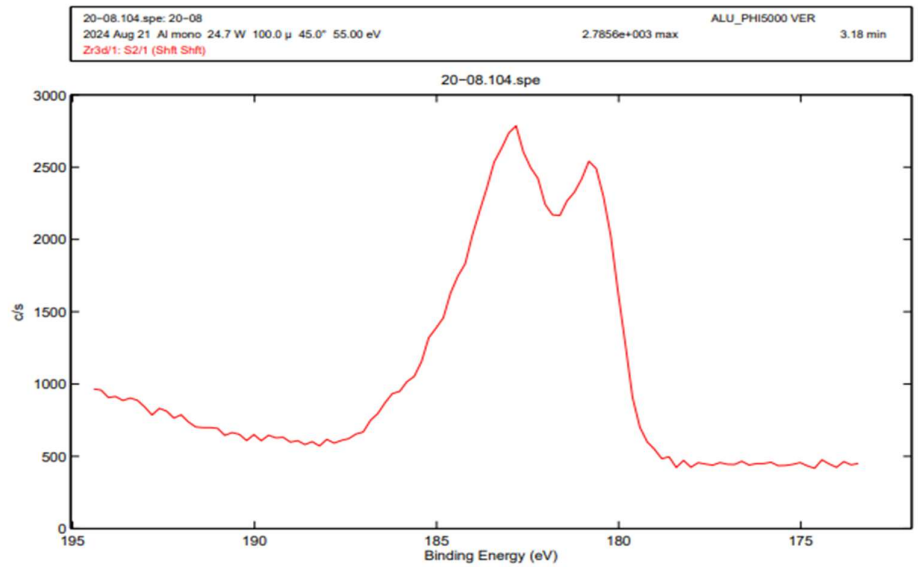


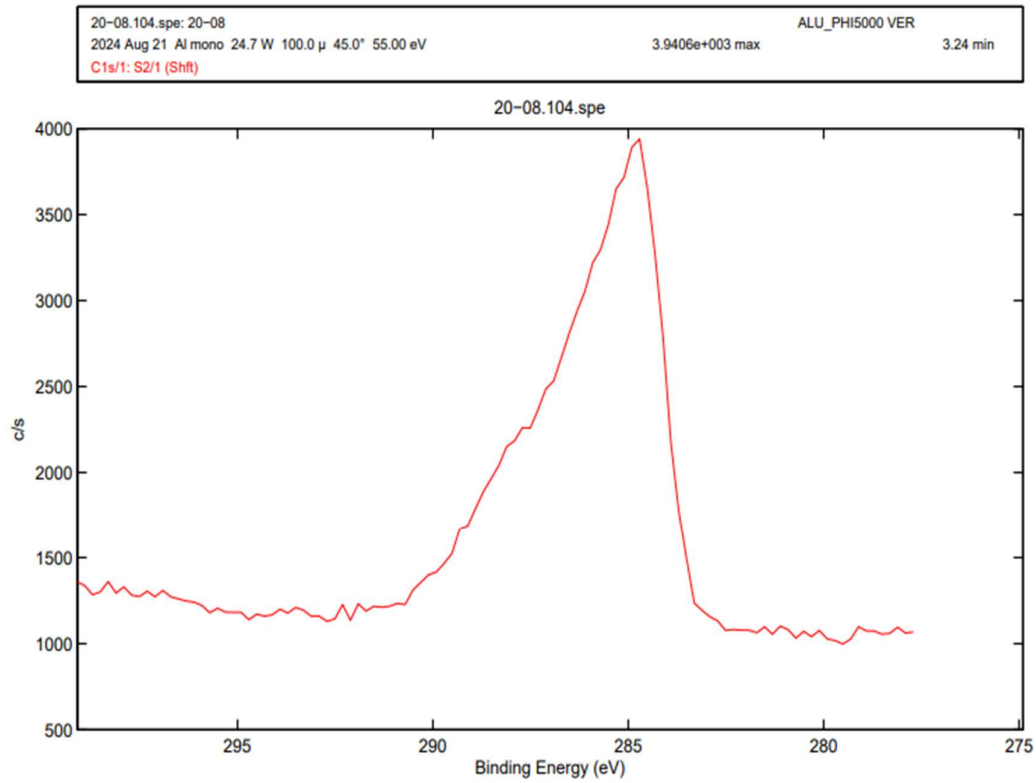
FIG.4.SEM image of ZrO2-KN

3.5 XPS

The chemical valence state of ZrO2-KN nanoparticles was investigated by XPS analysis. The high resolution XPS spectra of wide Zr_3d_5,O1s,C1s,Na1 score levels of ZrO2-KN were exemplified and the wide range scanning on the surface of samples. The doublet binding energy around 280 ev of satellite peak for ZrO2-KN correspond to spin orbit coupling [13].



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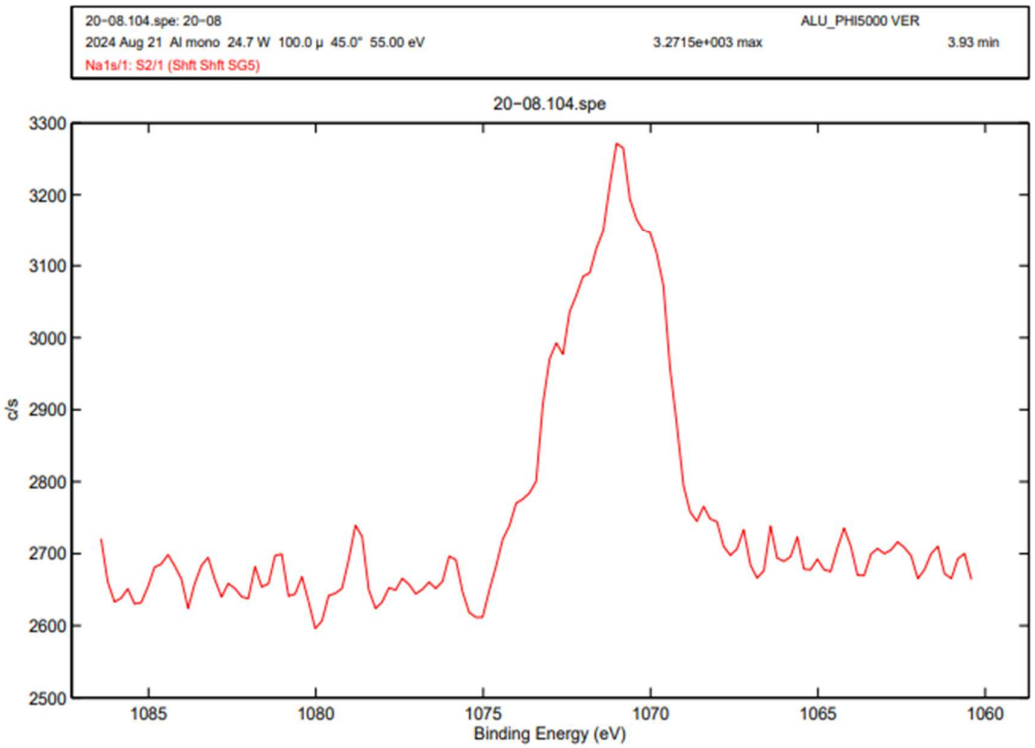
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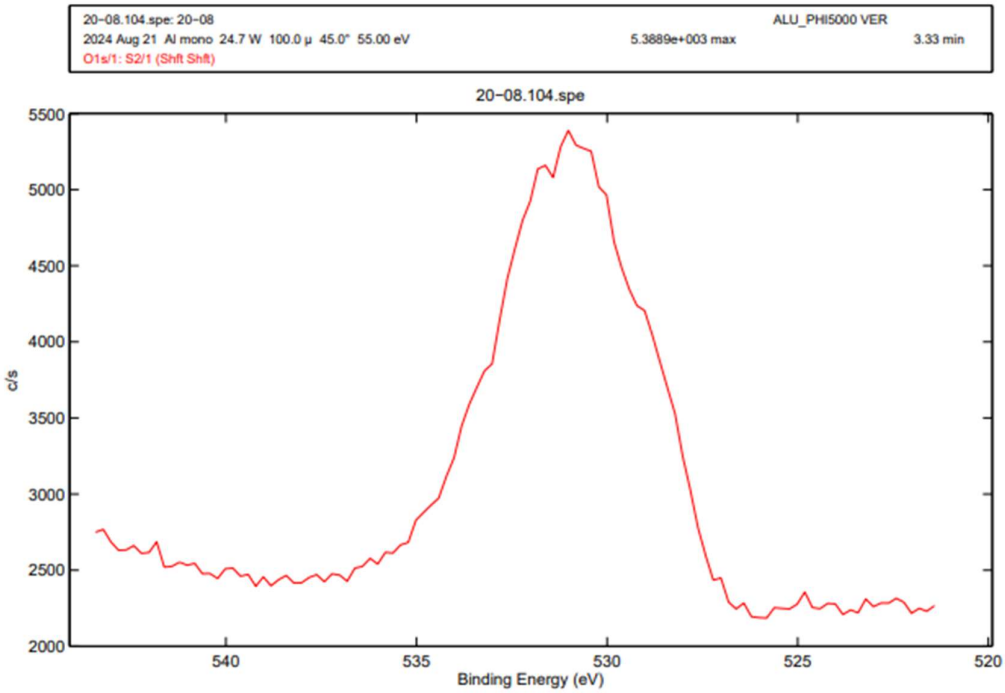
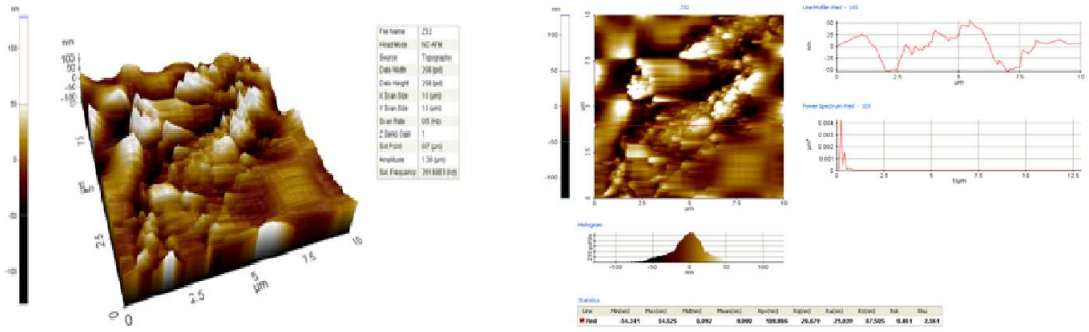


FIG.5.XPS spectra of ZrO2-KN

3.6 AFM



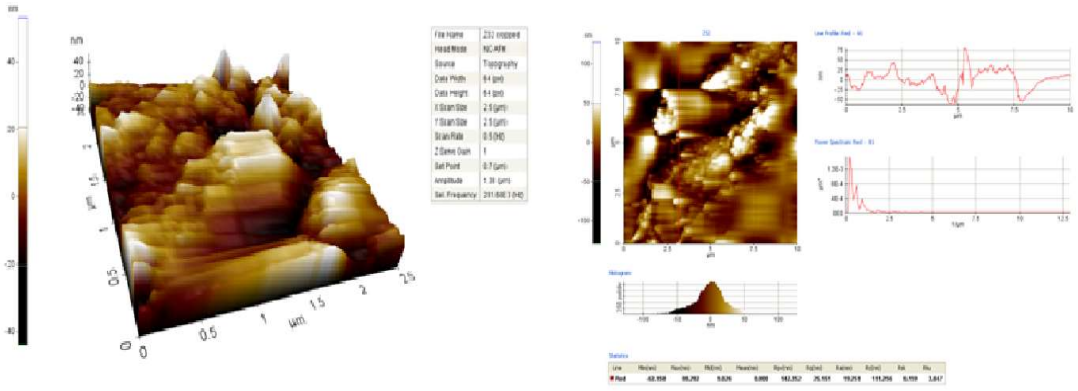


FIG.6.AFM surface image for ZrO2-KN

Figure shows the results obtained from AFM images along with area of analysis of ZrO2-KN powder without any doping agent [14,15].The information gives the surface topography of the sample, the increased roughness on either side can be correlated to changes in the structure of ZrO2-KN as a function of annealing temperature. Effect of annealing temperature on the mean height and median height were also examined and it was found that phase transformation and size of metallic nanoparticles.

4. Conclusion

ZrO2-KN nano particles was synthesised by co- precipitation method. The UV –V is analysis shows that the excitonic peak of ZrO2-KN nano particles occurs at 302 nm which shows that there is a strong blue shift for the synthesized nano particles and for ZrO2-KN nano particles the peak occurs for 450^0C respectively. The FTIR spectrum shows the existence of functional groups in the synthesized ZrO2-KN nano particles. The XRD results reveals that the ZrO2-KN has monoclinic and spherical shape structure and the average size was calculated. The SEM image results shows that the synthesized ZrO2-KN nanoparticles were spherical in shape. The average size was calculated to be 1µm to 200nm respectively. The high resolution XPS spectra of wide Zr_3d_5,O1s,C1s,Na1 score levels of ZrO2-KN were exemplified and the wide range scanning on the surface of samples. AFM results the mean height and median height were also examined and it was found that phase transformation and size of metallic nanoparticles.

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