



# Morphological and Optical Analysis of Tungsten Oxide Nanosheets for Gas Sensing Application

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## ABSTRACT

*Tungsten oxide (WO<sub>3</sub>) is a widely recognized transition metal oxide known for its stability and non-toxicity in nanoparticle form. Its low cost and simple synthesis procedure make it a promising contender for many applications. In the present work, WO<sub>3</sub> nanoparticles have been synthesized via a one-step hydrothermal route. X-ray diffraction pattern demonstrated the formation of a single phase of high crystallinity tungsten trioxide. Crystallite size and strain were used to explain the structural defects and formation mechanism of WO<sub>3</sub>. UV-visible spectroscopy was used to investigate the optical characteristics, and the single phases of WO<sub>3</sub> showed a band gap of 2.5 eV. Field Emission Scanning electron microscope (FESEM) images demonstrated the morphological properties of material. As a result, tungsten trioxide was found to be more environmentally sensitive than pre-transition-metal oxides.*

**Keywords:** Tungsten Oxide, Nanomaterial, Nanosheets, Hydrothermal

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## INTRODUCTION

The general consensus around nanoparticles and nanotechnology has been evolving over the previous few years, and the position may have changed. The current work focuses on tungsten oxide which is well-known metal for gas sensing applications. It has exceptional optical features, specifically the ability to change colour depending on its oxidation state [1]. This feature has led to its employment in a variety of sectors and gained attention across various research domains due to its versatility in electrochromic devices (such as displays and smart screens), where its transparency can change in response to an applied voltage, as well as in energy storage, catalysis and gas sensing. [2], [3], [4].

Over the past 15 years, the adaptable nature of WO<sub>3</sub> has led to its utilization in a range of applications [5]. Research into photoelectrochemical cells for water

splitting began in 1976, and more recent studies have investigated their potential application in dye-sensitized solar cells, batteries, and sensor technology [6]. Moreover, WO<sub>3</sub> has gathered significant interest for sensing applications due to its role as a prominent n-type gas-sensing material, proving instrumental in detecting various hazardous gases [7].

The structural characteristics and gas-sensing behaviour of nanocrystalline WO<sub>3</sub> powders synthesized through a sol-gel process using tungstic acid [8]. A comparative study of electrochromic tungsten oxide films prepared via spin-coating and dip-coating techniques, evaluating their structural, morphological, optical, and electrochemical properties [9, 10]. Still, there remains a significant research gap concerning the tungsten oxide for gas sensing purposes [11], [12].

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Extensive research has been conducted on tungsten oxide nanoparticles and their flat structures, such as 2D nanosheets or thin films, primarily due to their remarkable surface area-to-volume ratio. Two-dimensional nanosheets, owing to their substantial surface area and relatively small volume, are particularly well-suited for gas sensing applications [13], [14]. Various methods, including sol-gel synthesis, hydrothermal approaches, and chemical vapor deposition, can be employed to produce nanostructured  $\text{WO}_3$  materials [15], [16], [17].

Tungsten oxide has been extensively researched for its ability to transition reversibly between a colourless state and a blue colour when subjected to alternating small positive and negative voltages [18], [19]. The stoichiometry of  $\text{WO}_3$  can change significantly, causing oxygen vacancies to occur in its crystal structure [20]. These vacancies can have an effect on its electrical and electrochemical characteristics and are useful in some applications like gas sensing, photodetectors, solar cells, etc. Hydrated versions of  $\text{WO}_3$  exist, such as  $\text{H}_x\text{WO}_3$  (where  $x$  is a variable) [21]. These hydrates can have unique properties and are useful in applications such as gas detection [22], [23].

Recently, there has been significant interest in tungsten-oxide-based materials due to their capability to absorb near-infrared (NIR) [24], [25], [26] light effectively and their efficient conversion of light into heat. Moreover, these materials possess an intriguing oxygen defect structure and exhibit strong local surface plasma resonance (LSPR) [27], [28], [29] resulting in robust photo absorption across a broad range of NIR wavelengths. Previously, a range of light-absorbing nanomaterials, including noble metals, polymeric materials, and other inorganic nanomaterials, have been investigated for their potential use in photothermal therapy for cancer treatment [30].

Even  $\text{WO}_3$  has been utilized as an anode material due to its high theoretical capacity, low cost, and environmentally friendly nature [31]. Its main limitation, however, lies in its low electrical conductivity, a challenge that has been addressed with  $\text{WO}_{3-x}$  materials. The research focused on the development of high-performance mesoporous  $\text{WO}_{3-x}$  anodes using a hard template approach to enhance electrical conductivity. The resulting material exhibited an impressive reversible capacity of  $748 \text{ mAh g}^{-1}$  and a

high volumetric capacity of  $1,500 \text{ mAh cm}^{-3}$  compared to bulk  $\text{WO}_{3-x}$ .

In recent years, the quest for efficient gas sensing materials has intensified due to their crucial role in various industrial [32], environmental [33], and biomedical applications [34]. Among the myriad of materials explored, tungsten oxide ( $\text{WO}_3$ ) has emerged as a promising candidate owing to its unique properties such as high surface area, excellent chemical stability, and sensitivity to various gas species [35],[36].

Nanoscale morphological and optical properties play a significant role in determining the performance of gas sensing materials [37], [38]. Tungsten oxide nanosheets, with their two-dimensional structure, offer a large surface-to-volume ratio and enhanced gas interaction capabilities, making them particularly attractive for gas sensing applications [39], [40].

This paper presents a comprehensive study on the morphological and optical characteristics of tungsten oxide nanosheets and their implications for gas sensing. We investigate the synthesis methods, and examine the structural properties, surface morphology [41], and optical properties of  $\text{WO}_3$  nanosheets using various analytical techniques such as XRD, UV-Visible spectroscopy and FESEM [42], [43].

The novelty of this study lies in its exploration of an unaddressed research gap, specifically analysed the morphological and optical properties of the synthesized material [44]. Nanoparticles of  $\text{WO}_3$  doped materials were synthesized using the hydrothermal method and subsequently evaluated for their potential application in gas sensors. The orthorhombic structure of  $\text{WO}_3$  has been presented in Fig. 1.

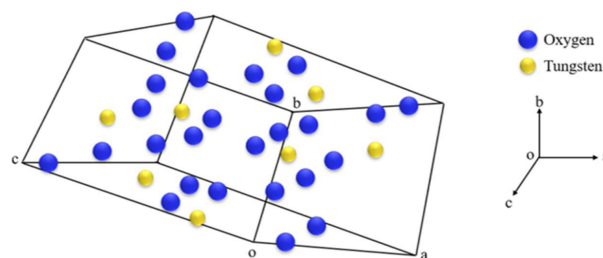


Fig. 1. 3D model of orthorhombic  $\text{WO}_3$

## EXPERIMENTAL SECTION

### Chemical Reagents

During the synthesis process, analytical reagent (AR) grade chemicals and reagents (as detailed in Table

1) were used without further purification. Absolute ethanol and deionized (DI) water were employed for cleaning and washing procedures. Unless otherwise stated, all solutions were prepared using DI water.

**Table 1.** Chemicals utilized in the synthesis process

Chemical Name	Chemical Formula	Manufacturing Company	Purity
Sodium Tungstate	$\text{Na}_2\text{WO}_4$	M/s Thomas Baker	98%
Oxalic Acid	$\text{HO}_2\text{C}=\text{CO}_2\text{H}$	M/s Loba Chemie	99%
Nitric Acid	$\text{HNO}_3$	M/s CDH Ltd	98%

## Synthesis

The single-step hydrothermal method was employed to synthesize tungsten oxide nanosheets. To prepare a single phase of  $\text{WO}_3$ , a solution of  $\text{Na}_2\text{WO}_4$  (0.096 M) was initially prepared in 30 ml DI water and stirred for 15 minutes. Afterwards, the mixture was agitated for a few minutes with the addition of 5ml  $\text{HNO}_3$ . A 0.249 M solution of oxalic acid was prepared in 20 mL of deionized (DI) water and stirred for 30 minutes. This solution was then added dropwise to the pre-prepared solution under continuous stirring. The resulting mixture was stirred further for one hour at room temperature. Subsequently, the precursor solution was transferred into a Teflon-lined hydrothermal autoclave reactor and heated to  $220^\circ\text{C}$  for 24 hours in a hot air oven. It was then left overnight to cool naturally. The solution was later centrifuged and washed with DI water and ethanol several times. Finally, the precipitates were collected and dried in a vacuum oven at  $60^\circ\text{C}$ . The sample was then grounded to obtain the nanoparticles of  $\text{WO}_3$ . The powder was then calcinated in the presence of oxygen for an hour at  $500^\circ\text{C}$  with a heating rate of  $1^\circ/\text{min}$ .

## RESULTS AND DISCUSSION

### X-Ray Diffraction

The XRD pattern of the synthesized powder sample was recorded within the  $2\theta$  range of  $10^\circ$  to  $80^\circ$ . This technique is used to characterize the structural properties of materials, including tungsten oxide ( $\text{WO}_3$ ) nanosheets, which are essential for understanding their behaviour in gas sensing applications [45], [46].

XRD involves bombarding a sample with X-rays and measuring the intensity and angle of the diffracted X-rays. This data provides information about the crystal

structure, phase composition, lattice parameters, and crystallinity of the material [47].

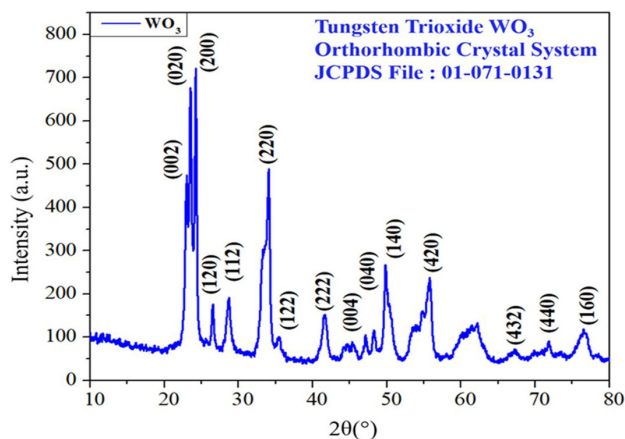
For our study on the morphological and optical analysis of tungsten oxide nanosheets, XRD serves as a crucial characterization technique. By analysing the diffraction patterns obtained from  $\text{WO}_3$  nanosheets, we can identify the crystal phases present, assess their purity, and determine their crystalline structure.

The peaks of the synthesized sample were analysed and indexed using the JCPDS file 01-071-0131, showing a good match with  $\text{WO}_3$ . The synthesized  $\text{WO}_3$  exhibited an orthorhombic crystal structure corresponding to specific (hkl) planes, given as (002), (020), (200), (120), (112), (220), (122), (222), (004), (040), (140), (420), (432), (440) and (160) as shown in Fig. 2.

The JCPDS file specifies that crystal structure of  $\text{WO}_3$  as being in the space group Pmnb which means that  $\text{WO}_3$  crystal structure has an orthorhombic symmetry with specific translational and rotational elements [48].

The Pmnb space group specifies the following symmetry elements, where 'P' defines a primitive lattice, 'm' represents the Mirror planes perpendicular to the a-axis, 'n' defines the Mirror planes perpendicular to the b-axis and 'b' illustrates the Glide planes perpendicular to the c-axis. In  $\text{WO}_3$ , tungsten atoms are located at the corners and edges of distorted octahedra formed by oxygen atoms. Each tungsten atom is surrounded by six oxygen atoms, and each oxygen atom is shared between two octahedra [49].

This arrangement results in an orthorhombic crystal structure where the lattice parameters are unequal. Typically, in  $\text{WO}_3$ , the lattice parameters are  $a \neq b \neq c$ , and the angles between the lattice vectors are  $90^\circ$ .



**Fig. 2.** XRD pattern of  $\text{WO}_3$

### Lattice Parameters, Crystallite Size and Strain

Lattice parameters are the lengths of the edges and the angles between them that define the unit cell of a crystal lattice. In other words, they describe the geometric arrangement of atoms in a crystal structure.

The crystallite size corresponds to the size of the smallest crystal in, a single crystal in powder form. Debye Scherrer formula provided in equation-1 has often been used to determine the size of crystallites. The Debye Scherrer formula is given as,

$$d = K \frac{\lambda}{\beta \cos \theta} \quad \dots(1)$$

Here,  $d$  represents the average diameter of crystallite, value of shape constant ( $k$ ) is 0.9,  $\lambda$  stands for radiation wavelength (1.54 Å),  $\beta$  refers to full width at half maxima (FWHM),  $\theta$  is Bragg's angle of respective peaks [50], [51]. The average of analysed peaks was determined and the obtained crystallite size of  $\text{WO}_3$  was approx. 23 nm. This signifies the average size of individual crystalline domains. Smaller crystallite sizes can enhance surface area, potentially improving reactivity or electrical properties, particularly in catalysis or semiconductor applications [52], [53]. The lattice parameters for as synthesized sample were calculated using the equation-2.

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \quad \dots(2)$$

The inter planer spacing is denoted by  $d$ , with  $(hkl)$  representing the Miller indices, while  $a$ ,  $b$ , and  $c$  correspond to the lattice sides. Table 2 illustrates the differences between the lattice parameters from the JCPDS file and the calculated values.

**Table 2.** Lattice parameters of the as-synthesized sample

Parameters	Calculated Value for $\text{WO}_3$	JCPDS Value for $\text{WO}_3$
$a$ (Å)	7.37	7.34
$b$ (Å)	7.57	7.57
$c$ (Å)	7.34	7.75

The deviated value of lattice parameter represents the presence of micro strain. The Williamson Hall (W-H) Plot which is a graphical tool commonly used to calculate the strain and crystallite size, for describing the microstructural characteristics of crystalline materials and which applied the following equation,

$$\beta \cos \theta = (\epsilon \times 4 \sin \theta) + K\lambda / D \quad \dots(3)$$

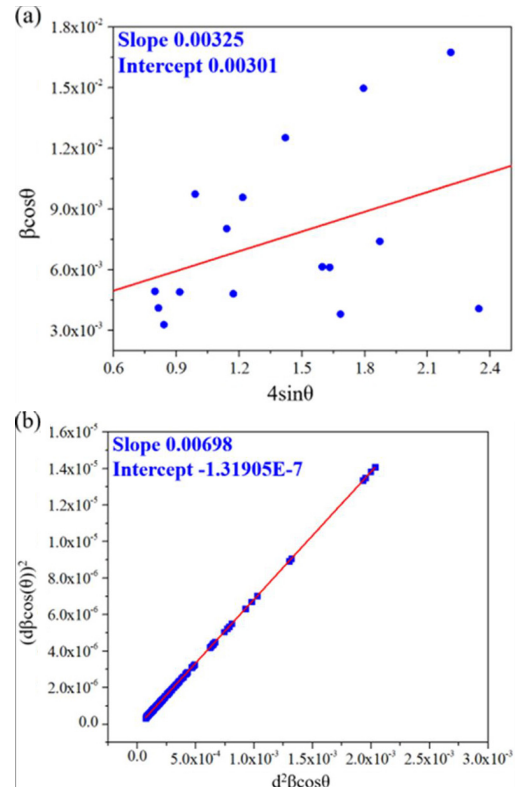
Here,  $\epsilon$  indicates the micro strain in the as synthesised material [54], [55]. The W-H plot is in between  $(4 \sin \theta)$  and  $(\beta \cos \theta)$ , for  $\text{WO}_3$  the slope and intercept are 0.00325 and 0.00301 respectively observed, as shown in Fig. 3(a). The crystallite size and strain obtained by using the W-H Plot is 46.04 nm and 0.00325 respectively.

The Size Strain (S-S) plot is also used as it offers precise and unbiased measurements of crystallite size and strain [56]. S-S plot accounts for multiple transitions in the material, including direct and indirect transitions, while the W-H plot typically assumes a single type of transition [57], [58].

The S-S plot often yields a more linear relationship between the absorption coefficient squared ( $\alpha^2$ ) and the photon energy and even requires less data processing. The equation 4 is the formula that was applied [59].

$$(d\beta \cos \theta)^2 = K\lambda/D (d^2\beta \cos \theta) + \epsilon^2/4 \quad \dots(4)$$

The S-S plot was obtained between  $(d^2\beta \cos \theta)$  and  $(d\beta \cos \theta)^2$  using which the slope is 0.00698 and intercept equals to -1.31905E-7 as observed in Fig. 3(b). The crystallite size and strain calculated by S-S Plot are 19.85 nm and 0.00229 respectively.



**Fig. 3.** (a) W-H plot of the synthesized sample, (b) S-S plot of the prepared sample

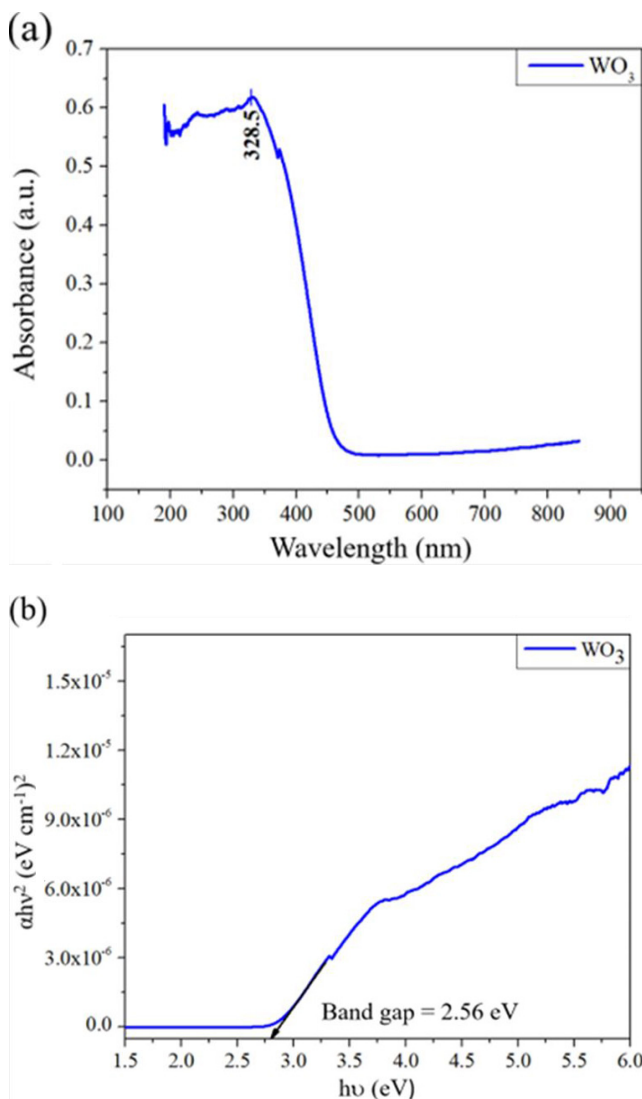


## UV-Visible Spectroscopy

Ultraviolet-visible spectroscopy, is a widely used analytical technique to study the electronic transitions that occur in molecules.

UV-Vis spectroscopy detects the absorption of ultraviolet (UV) or visible light by molecules. When molecules absorb UV or visible light, their electrons transition from lower energy states to higher ones [60]. The difference in energy between these states matches the absorbed light's energy, appearing as absorption bands in the UV-Vis spectrum.

This technique was employed for as synthesized sample with wavelength ranging from 190 to 850 nm. The maximum absorption peaks were seen in the UV region between 190 nm and 450 nm.



**Fig. 4.** (a) Absorption spectra of WO<sub>3</sub>, (b) Tauc Plot of the synthesized sample

In the UV region, molecules undergo electronic transitions, where electrons are excited from lower energy levels to higher ones. This transition results in absorption of UV light, leading to characteristic absorption spectra [61]. For as synthesized material, 328.5 was the highest absorption peak as shown in Fig. 4(a).

UV analysis represents the optical bandgap, for which we employed the Tauc plot [62], [63]. Tauc plot defines the bandgap energy ( $E_g$ ) of a material. The Tauc plot is a graphical representation of a semiconductor material's absorption edge, typically plotted against specific parameters,  $(\alpha h\nu)^2$  versus the photon energy ( $h\nu$ ). The equation used for this,

$$\alpha h\nu = A[h\nu - E_g]^{1/n} \quad \dots(5)$$

Here,  $\alpha$  denotes the absorption coefficient,  $A$  is proportionality constant,  $E_g$  represents the bandgap and the nature of transition represented by  $n$  exponent. Using this formula, the bandgap of WO<sub>3</sub> was determined to be 2.56 eV, as shown in the Fig. 4(b). The bandgap of 2.56 eV was optimum for a number of applications [64], [65].

In the optoelectronic devices like photodetectors and sensors, the bandgap may provide a suitable balance between their sensitivity to visible light and electrical conductivity [66].

The refractive index is a helpful parameter for characterising and identifying compounds. It determines how light propagates through a material. It affects phenomena such as reflection, refraction, and dispersion. Understanding the refractive index is crucial for designing optical components like lenses, prisms, and fibres. Refractive index can serve as a characteristic property for identifying materials. Different substances have distinct refractive indices, allowing for their identification through techniques like refractometry or ellipsometry.

The effective refractive index of a medium can vary when a gas with a different refractive index than the medium in the vicinity interacts with it [67], [68], [69]. The variation of refractive index can detect the presence of specific gas and used for gas sensors. The material's Refractive Index is also calculated using the equation-6.

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{E_g / 20} \quad \dots(6)$$

Here,  $n$  denotes the refractive index, while  $E_g$  represents the corresponding band gap of the sample [70]. As determined earlier the band gap of  $\text{WO}_3$  is 2.56 eV, the as synthesized material  $\text{WO}_3$  has a refractive index of 2.53.

### Morphological Analysis

FESEM (Field-Emission Scanning Electron Microscopy) characterization involves using a high-resolution electron microscope to examine the surface morphology and structure of materials at the nanoscale [71]. It provides detailed images of the sample's surface, revealing features such as size, shape, and distribution of particles or structures [72].

FESEM images can also reveal surface features such as roughness, porosity, and the presence of defects or crystalline facets [73]. These surface characteristics can influence the material's properties and applications.

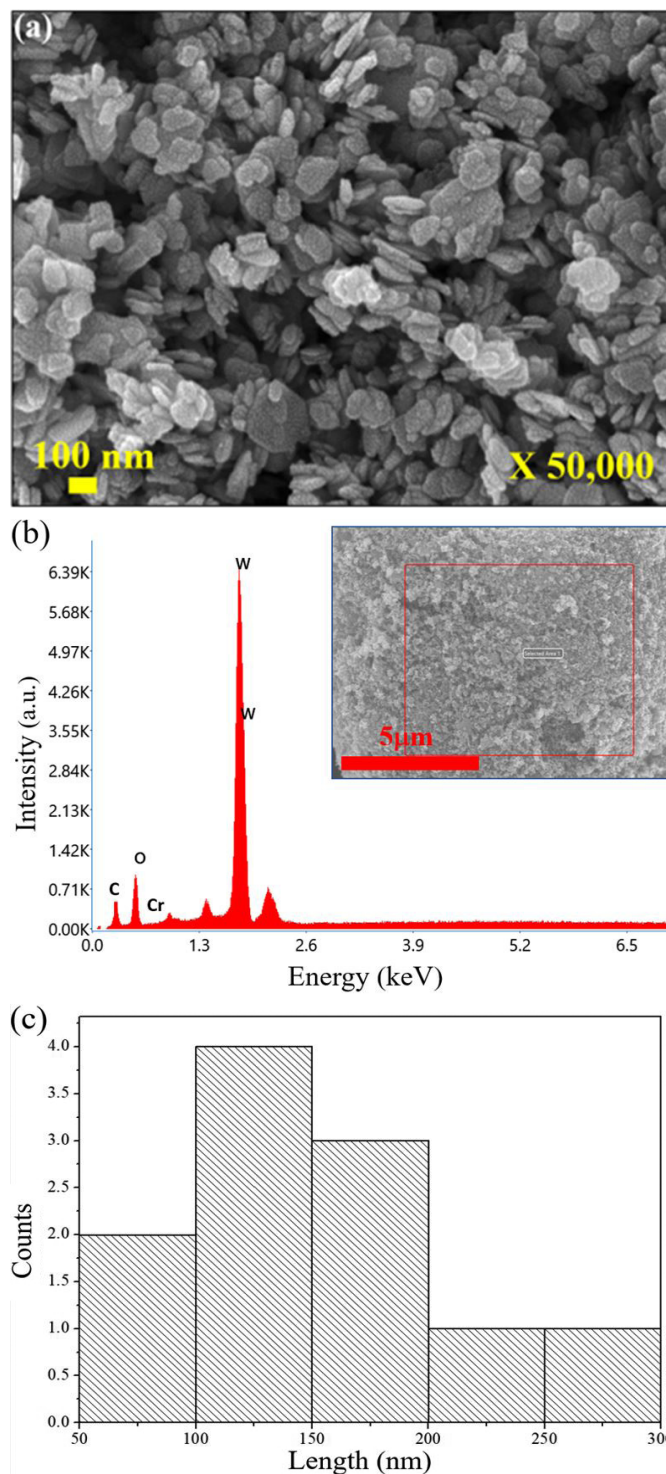
The image can indicate whether the  $\text{WO}_3$  nanoparticles are well-dispersed or aggregated. Aggregation can affect the material's properties, such as its reactivity and optical behaviour [74]. Depending on the imaging conditions and the quality of the sample, FESEM images may provide insights into the crystalline structure of  $\text{WO}_3$ . This can be further confirmed by complementary techniques such as X-ray diffraction (XRD).

FESEM technique provides a significant experimental approach used for studying the surface properties and composition of materials at the microscopic level. Depending on the synthesis approach and conditions employed,  $\text{WO}_3$  can have a variety of morphologies [75].

The nanoflower like morphology can be seen through hydrothermal route but during this process metal oxides may contain the volatile impurities such as water, which indicated the presence of acidic environment and promotes the nucleation and growth. To remove these volatile elements the calcination process is frequently used [76]. The calcination transforms the morphology of tungsten trioxide into the 2D Nanosheets. These tend to be thin, flat constructions with a high aspect ratio, resembling paper sheets within range of 100 nm as shown in Fig. 5(a).

Energy Dispersive X-ray Spectroscopy (EDX) spectra provide information about the elemental composition

of a sample [77]. When a material is exposed to high-energy electrons or X-rays, it emits characteristic X-rays with energy levels that correspond to the specific elements within the sample. It identifies the elements present in the sample by detecting characteristic X-rays emitted when the electrons of atoms in the sample are



**Fig. 5.** (a) FESEM Image of the tungsten oxide, (b) EDX Spectra of corresponding area, (c) Particle Size Distribution

excited and transition to lower energy levels [78]. It gives an indication of the relative abundance of each element in the sample by measuring the intensity of the characteristic X-rays emitted.

In conjunction with field emission scanning electron microscopy (FESEM), EDX can provide information on the spatial distribution of elements, showing where specific elements are concentrated in the sample [79].

Fig. 5(b) shows that the tungsten (W) and oxygen (O) are present in the sample. Furthermore, the sample contains a particularly high concentration of tungsten. The signals attributed to carbon and copper in the EDX spectra originated from the carbon coating on the copper grid of electron microscope.

The observed nanosheets exhibited particle sizes ranging from 100 nm to 150 nm, as illustrated in Fig. 5(c). Their high aspect ratio contributed to enhanced material reactivity. They are hexagonal, rectangular 2D shape nanosheets. Even 2D  $\text{WO}_3$  nanosheets frequently have outstanding structural and optical characteristics, making them valuable for sensors and photocatalysis.

## CONCLUSION

The current work emphasis on the structural and optical study of  $\text{WO}_3$  for application in gas sensors. The synthesis process used for  $\text{WO}_3$  was the hydrothermal method. XRD analysis of the as-synthesized powder sample revealed that the material's crystal structure has orthorhombic symmetry. The formation of 2D nanosheets demonstrated by the FESEM technique. The band gap obtained from the tauc plot is 2.56 eV, within the region of wide bandgap semiconductors with a refractive index of 2.53. The Debye-Scherrer method yielded a crystallite size of 23.15 nm. The crystallite size and strain values calculated from the W-H Plot were 46.04 nm and 0.00325, respectively. Whereas, the S-S plot showed crystallite size and strain of 19.85 nm and 0.00229, respectively. Because of its varied qualities,  $\text{WO}_3$  can be applied to a wide range of situations.  $\text{WO}_3$  is widely explored for photocatalytic properties and has been proved useful for environmental cleanup and water splitting for the production of hydrogen.  $\text{WO}_3$  has also been explored for gas sensing applications. When exposed to reducing gases, such as hydrogen, its broad bandgap allows the generation of charge carriers in the crystal lattice, or oxygen vacancies in

the crystal lattice when exposed to oxidizing gases like ozone. It can therefore be utilised in gas sensors to identify different gases, such as hydrogen, ozone ( $\text{O}_3$ ) and nitrogen dioxide ( $\text{NO}_2$ ). Thus, the future prospect is to use the as-synthesized  $\text{WO}_3$  nanosheets for gas sensing applications due to its high aspect ratio and advantageous band gap.

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