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Review on Eco-friendly Green Synthesis Methods of Tungsten Oxide Nanoparticles

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Abstract

Synthesis of tungsten oxide nanoparticles by alternative method has been use of environmentally friendly green methods that involve natural materials like plants, bacteria, fungi, solvothermal, microwave-assisted, biosynthesis, sonochemical, polysaccharides, seaweed, plant-derived materials, biodegradable polymers, and algae using effective green chemistry to create nanoparticles has sparked a lot of attention lately due to its environmental friendly, simplicity, affordability, and clean technology involve no use of any dangerous chemicals and produces no impurities or wastes. Plant extracts have garnered the greatest attention among these bio-entities due to their special natural qualities that enable them in a single production step to stabilize and decrease metal nanoparticles. The changes and future prospects of green synthesis methods using plant extracts, solvothermal, microwave-assisted, biosynthesis, and sonochemical processes for tungsten oxide nanoparticles are reviewed in this paper.

Keywords: Eco-friendly, Green Synthesis, Tungsten Oxide, Nanoparticles

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Introduction

Recent years have seen an acceleration of nanoparticle research due to their wide range of applications. Furthermore, the development of nanotechnology has had a significant impact on materials engineering by offering chances to improve and broaden applications through the creation of better materials. [1, 2]. The significance of metal nanoparticles stems from their nanoscale size and high surface atom proportion, which result in high surface areas [3, 4]. The particles' characteristics at this small scale differ greatly from those of their bulk counterparts [5, 6]. Because of their intriguing characteristics, Nanoparticles have been the focus of a lot of research because of their structural shape, the "bottom-up" method and the "top-down" technique are the two ways to synthesize nanoparticles. By decreasing their size, the top-down approach isolates the resulting nanoparticles from the bulk substance into tiny parts [7]. Tungsten oxides have been the focus of extensive investigation for a long time because of their fascinating physical properties and possible chemical applications. [8]. As a transition metal oxide semiconductor, tungsten oxide (WO_3) has shown promise as a material for a number of uses, including photocatalysis, smart windows, gas sensors, and electrochromic devices [9,10]. The application of chemistry to stop environmental degradation and preserve human health is known as "green chemistry" is the process of creating new products and methods or altering current ones in order to reduce or eliminate the usage and production of dangerous materials, therefore minimizing harm and the threat to the environment and public health [11, 12]. It is important to apply green chemistry in order to minimize the usage and manufacturing of dangerous materials, in this sense, the design of green synthesis techniques takes into account twelve fundamental ideas in green chemistry [13, 14]. One of the biggest obstacles to reducing the use of hazardous materials is the creation of nanostructured materials, non-toxic reagents, corrosive acids such as (HCl, HNO_3, or H_2SO_4) [15–19]. The manufacture of nanoparticles using ecologically friendly protocols that use microorganisms or plant extracts instead of traditional physical and chemical processes has gained popularity in recent years, in this instance, the reaction is typically finished in a matter of minutes by simply mixing the extract with a room-temperature solution of the metal precursor [20]. Plants, algae, fungi, bacteria, diatoms, and human cells all biologically create NPs as green synthesis pathways available for

nanoparticles, plants are the most favored [21,22]. The pace of formation of the nanoparticles and other aspects are known to be affected by stirring duration, temperature, pH of solution, type of plant extract, metal salt concentration and concentration of plant extract [23]. This approach involve no complicated steps and is thought to be more cost-effective, energy-efficient, and environmentally friendly [24]. In this study instead of squandering time on the ideal solution synthesis method, it will assist researchers in devoting their attention to the development and preparation of tungsten oxide nanoparticle approaches, because they won't have to produce the materials in several methods, upcoming researchers will be able to produce nanomaterials in simple and inexpensive ways, as this publication provides green methods for preparing tungsten oxide nanoparticles.

1. Synthesis of tungsten oxide nanoparticles by plant extract methods.

1.1 Orange juice solution [25].

Orange juice was used as a stabilizing and reducing agent during the environment friendly production of spherically-shaped tungsten trioxide (SS-TTO) nanoparticles. In the beginning, 20 milliliters of double-distilled water were used to dissolve 2 grams of ammonium tungstate. After addition of 10 mL of fresh orange juice to this mixture while swirling constantly, a greenish-yellow suspension was produced. After that, some of the liquid in this mixture was evaporated. The leftover material was then dried at 120 °C and calcined for an hour at 500 °C to produce the final nanoparticles.

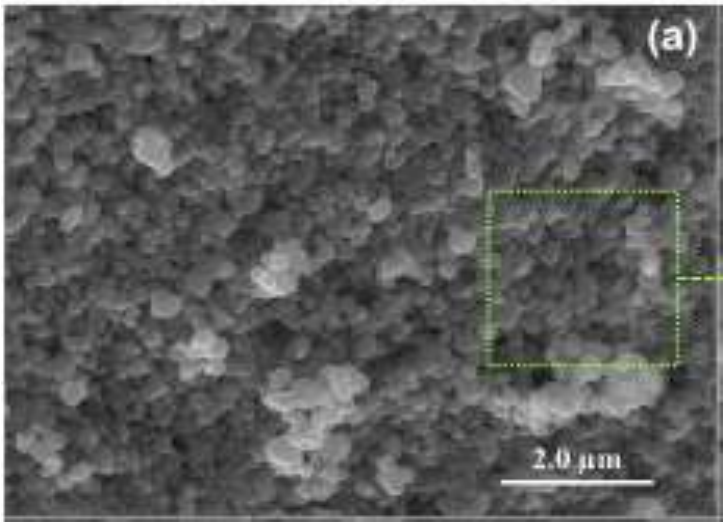
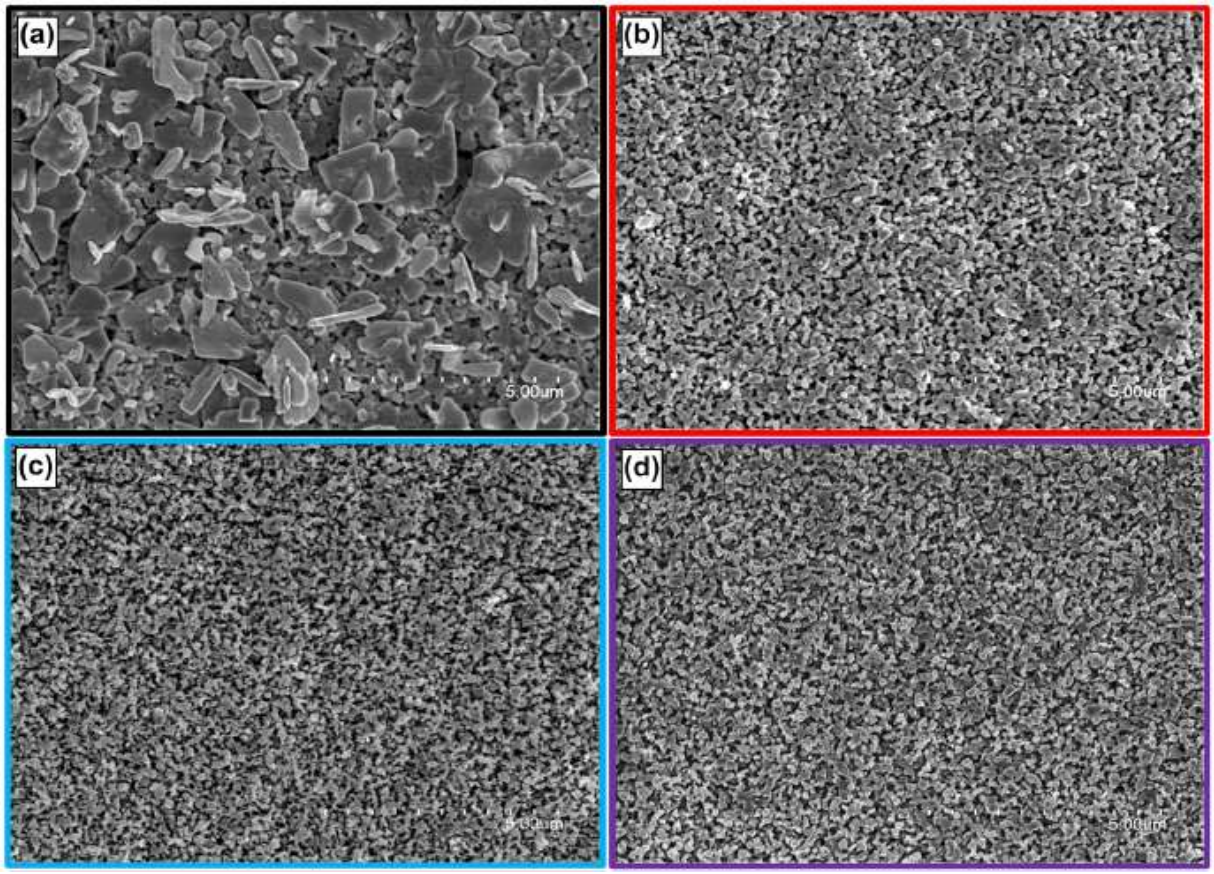


Figure 1: SEM images of the SS-TTO nanoparticles **[25].**

1.2 Melia azedarach leaves extract [26].

A random sample of M. azedarach leaves was taken from a number of mature trees in the area around the University of Valencia's Burjassot Campus in Spain. After being cleaned with tap and distilled water, fresh leaves were allowed to air dried indoors for a week at room temperature. 15 g of dried leaves and 150 mL of D.W. were combined at 60 ºC to create the extracts. For twenty minutes, the infusion was kept at that temperature. To prepare the various reaction media, the solution was cooled and filtered. Tungsten foils (1,3 cm^2 of exposed surface) were anodized for 4 hours at a cell potential of 20 V to create WO_3 nanostructures. The cathode of the cell was platinum foilA 1.5 M methanesulfonic acid (MSA) aqueous solution was combined with different molar concentrations. Azedarach leaf extract (ranging from 0% to 10%) and heated to 50°C in order to carry out the anodization process.



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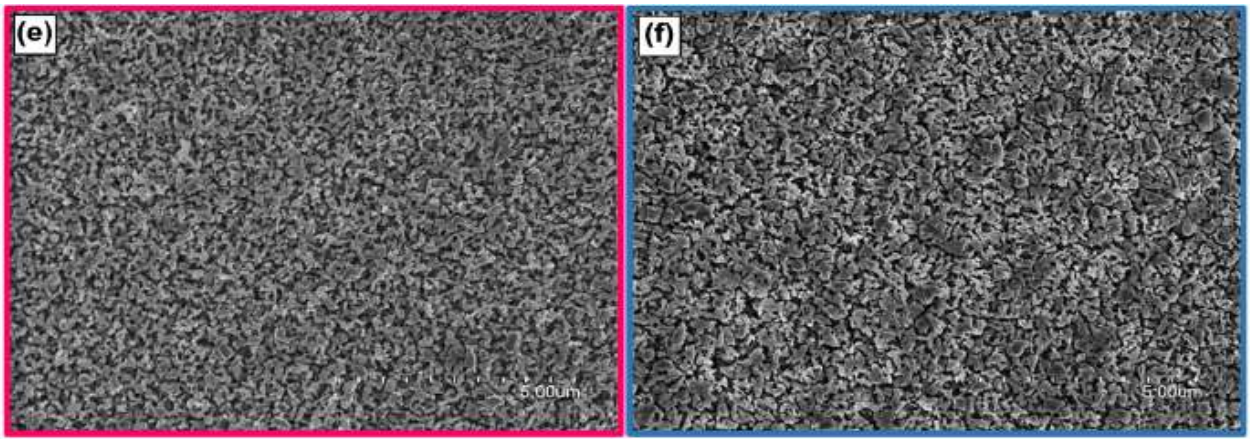

Figure 2: FESEM images (a) of the blank sample (b) 1%, (c) 3%, (d) 5%, (e) 7% and (f) 10% of extract **[26].**

1.3 Rhamnus Prinoides Leaf Extract [27].

After taxonomically identified the freshly collected *Rhamnus prinoides (gesho) leaves*, the leaves were kept at room temperature after being cleaned with both tap and distilled water. Left in the shade for two weeks until the moisture was completely dried, Then move to after leaves, the leaves were roughly crushed, placed into a clear plastic bag, sealed, and kept fresh until needed. 30 g of weighed *Rhamnus prinoide*s leaves were placed for an hour at 50 ^{o}C in 500 mL of distilled water, at room temp., pH of 5.9 the aqueous extract was filtered using *Whatman No. 1* filter paper. On the other hand, 82.5 grams of salt and 500 milliliters of distilled water were combined, stirred constantly and heated slowly until the salt was evenly mixed, to create a 0.25 M solution of Na_2WO_4. A standard synthesis process involved gradually adding 250 mL of Rhamnus prinoides aqueous leaf extract to 250 mL of sodium tungsten precursor solution (0.25 M). The mixture was continuously stirred with a magnetic stirrer while being gradually heated to 100 ^{o}C for one hour, or until the color changed from brown to greenish. After that, A greenish precipitate of $WO_3.nH_2O$ was produced by gradually adding 5 mL of 30% HCl to the liquid while stirring continuously, following each centrifugation at 3500 rpm for 5 minutes, allowing the liquid cool to ambient temperature, distilled water was used three times to clean it and remove any leftover impurities, After being gathered in a petri plate, the green precipitate was dried at 100 degrees Celsius in a

microwave oven. After that, the powder was annealed under air for two hours at 600 ºC, the resultant light yellow powder was appropriately packed for additional analysis and use.

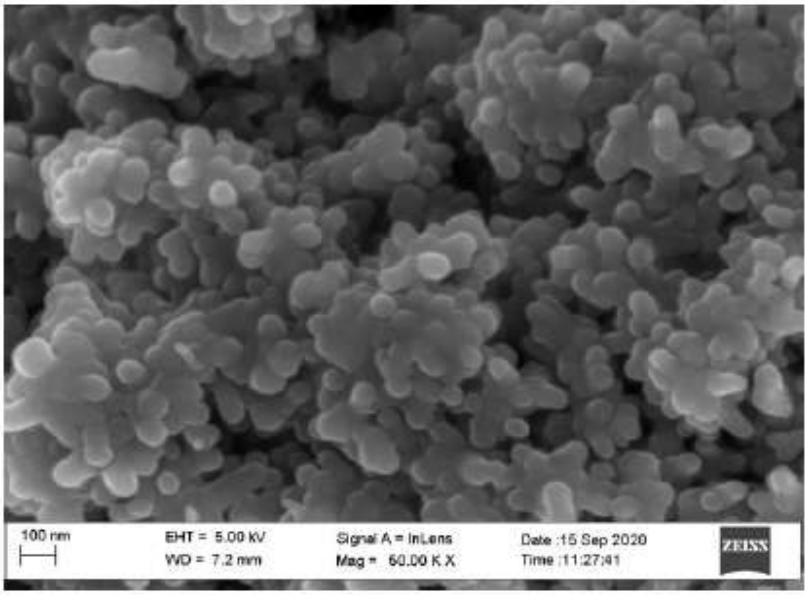

Figure 3: SEM images of biosynthesized WO_3 nanoparticles **[27].**

1.4 Moringa oleifera Lam. [28].

The extract of *Moringa oleifera* was used to create nanoparticles biologically using green approach. The leaves were first thoroughly cleaned using distilled water (14). A conical flask containing 100 milliliters of autoclaved distilled water and 1 milligram of tungsten oxide was placed on a magnetic stirrer. 50 grams of leaves were accurately weighed and ground in a mortar and pestle with distilled water for 15 to 20 minutes. These ground leaves were centrifuged for five minutes at 10,000 rpm at 4ºC. Following centrifugation, the supernatant was gathered and transferred to the flask holding the tungsten oxide solution. After being left overnight, the solution's color changed from light brown to dark brown, signifying the production and reduction of tungsten nanoparticles.

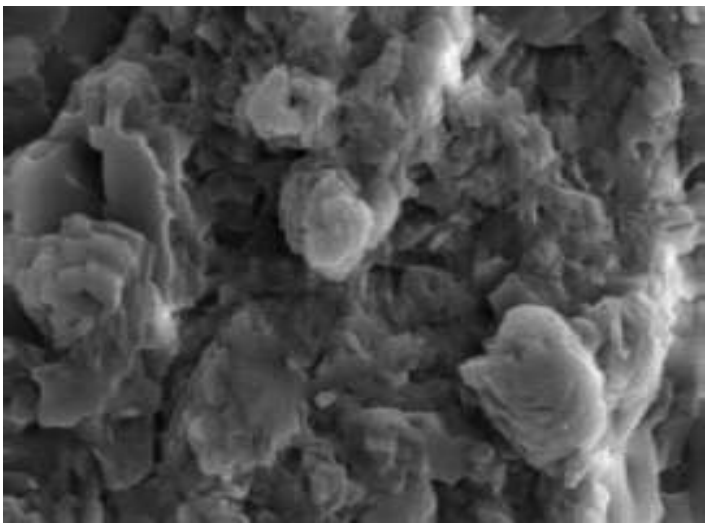

Figure 4: The SEM of tungsten oxide nanoparticles synthesized **[28].**

1.5 Psidium Guajava leaves extract [29].

Twenty milliliters of deionized water were used to create a 10−2 M solution of sodium tungstate dihydrate acetate monohydrate, which was then agitated for ten minutes at room temperature. A solution of sodium tungstate dihydrate was then gradually supplemented with 40 ml of Psidium Guajava leaf extract. Then, the mixture was placed for 4 h at 60 °C while being constantly stirred. Then, the mixture was placed on a heated plate to harden and remove any traces of water. The material was subsequently calcined at 400 °C for four hours in a furnace to produce the black WO_3 NRs.

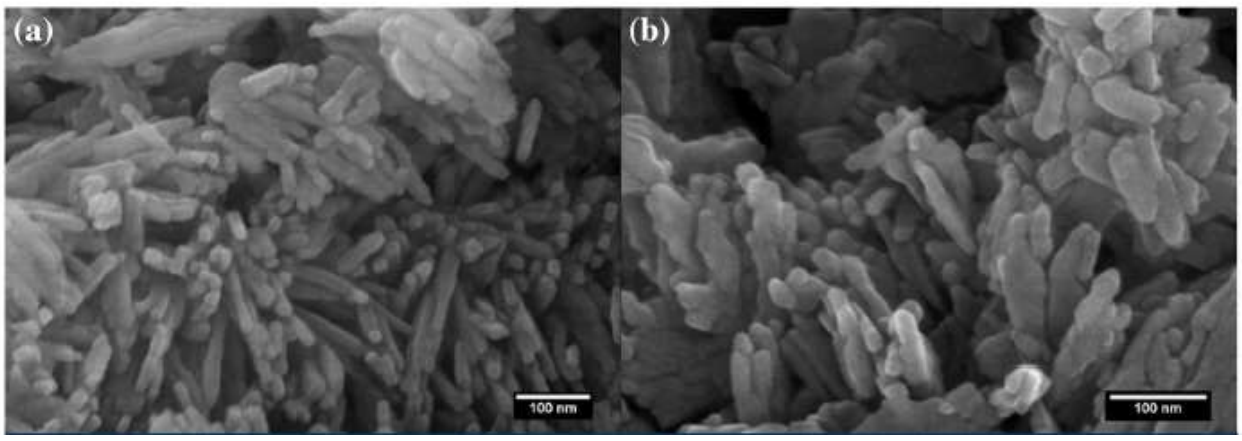

Figure 5: a and **b** FE-SEM images of synthesized WO_3 NRs **[29].**

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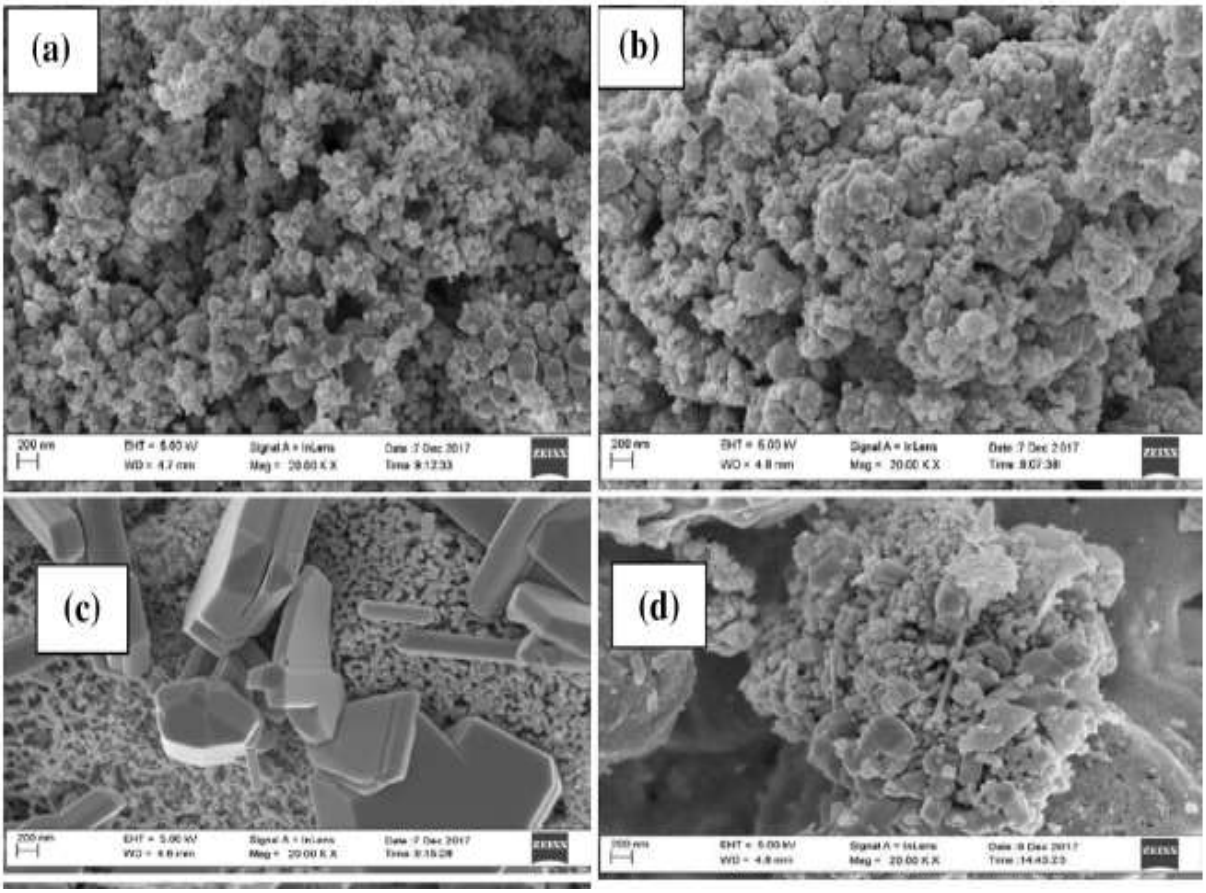
1.6 Spondias mombin aqueous extract: [30].

A 10 cm^3 volume of Spondias mombin aqueous leaf extract was gradually added to 100 ml of 0.06 M of $(NH_4)_{10}(H_2W_{12}O_{42})\cdot 4H_2O$ solution. For around thirty minutes, the mixture was continuously stirred at 150 rpm while being gradually heated to 120 °C. The pH was then carefully adjusted to an acidic range of 1 to 4 by adding drops of 10% HNO_3, and raised to a range of 7 to 13 by adding 0.5 M NH_4OH. For 30 minutes, the solution was continuously swirled. After letting the resultant solution for 24 hours, using decantation and washings to get rid of any contaminants or remaining aqueous extract, the white precipitate was separated, white precipitates formed and the changed from rusty brown to yellow. After applying the conditions, a white precipitate was produced, which was then dried for six hours at 80 °C in a moisture extractor. An air-filled furnace was used to anneal the resultant WO_3 for two hours at 550 °C.



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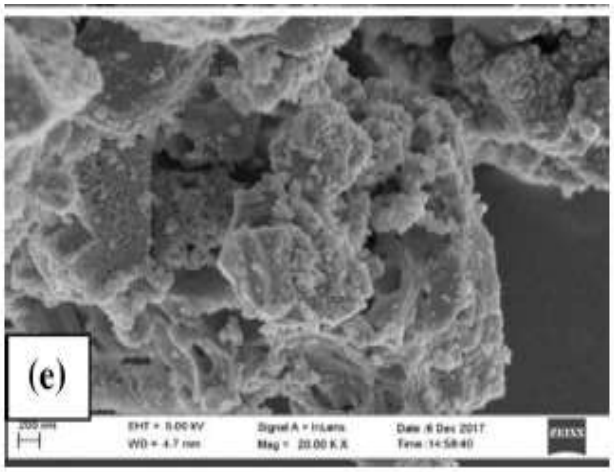
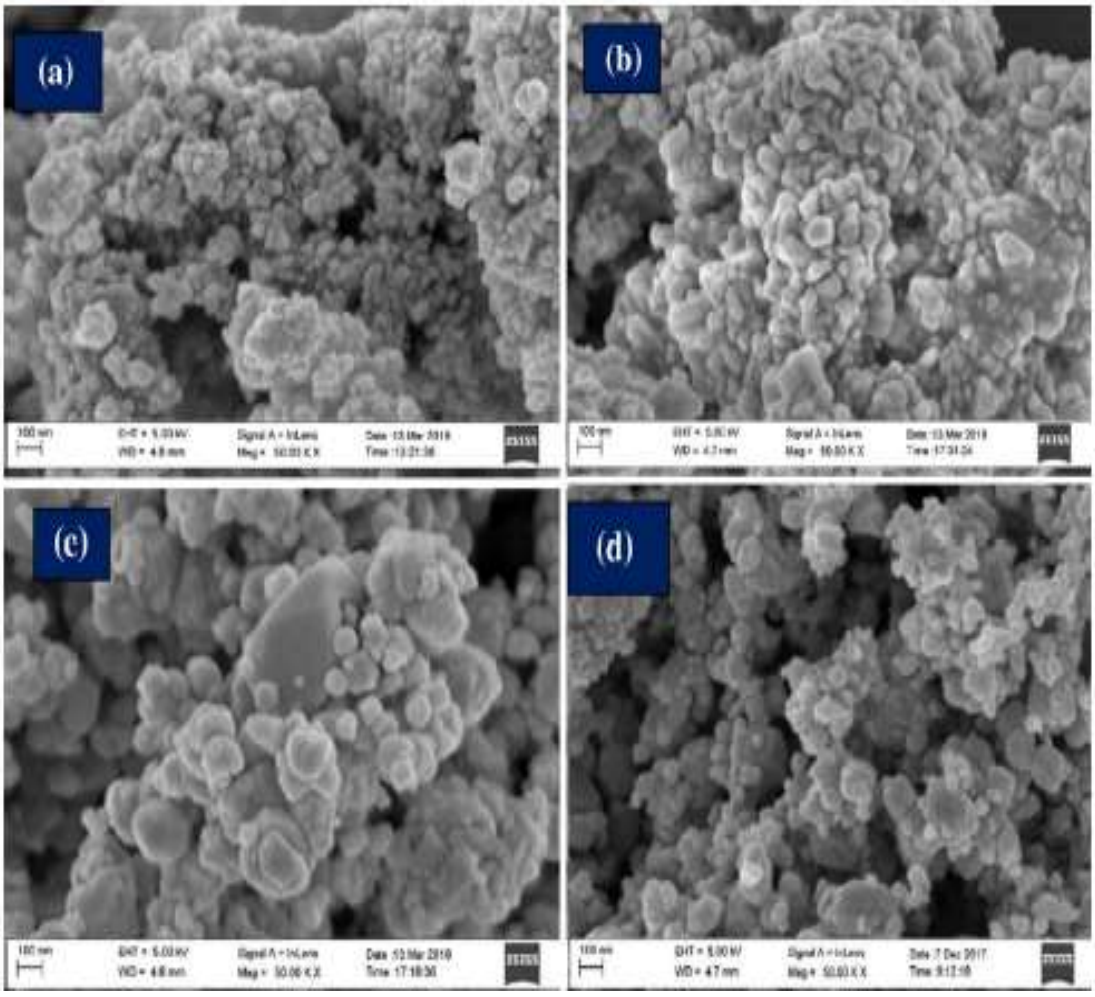


Figure 6: HR-SEM images of synthesized WO_3 NRs at different pH **a** pH 1, **b** pH 4, **c** pH 7, **d** pH 10, and **e** pH 13 **[30].**



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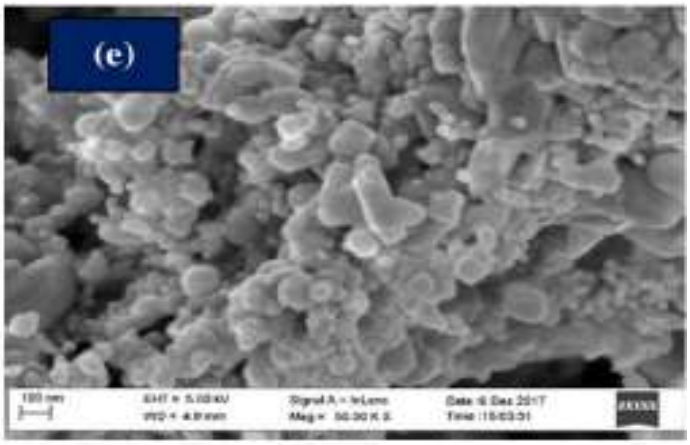
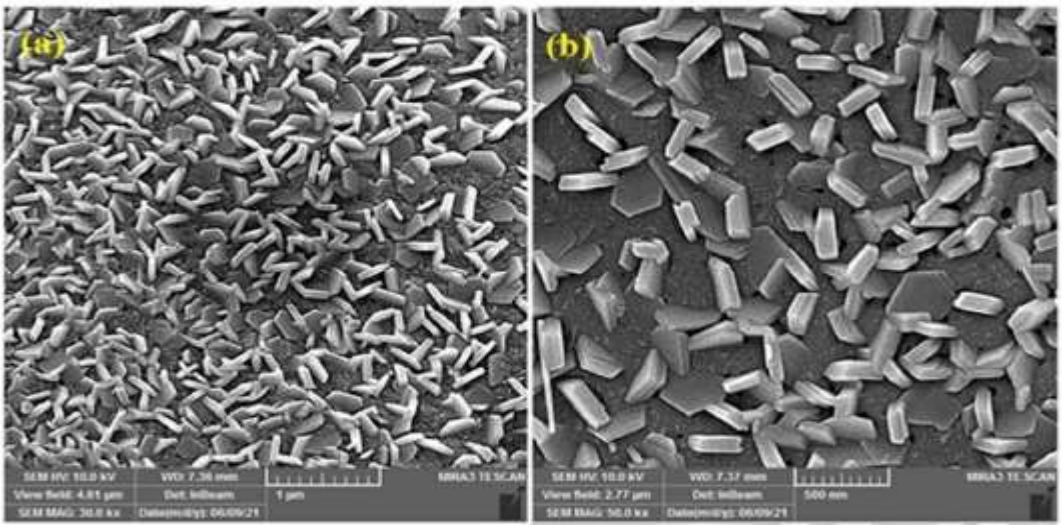


Figure 7: HR-SEM images of synthesized WO_3 NRs at 2h, **a** 250 °C, **b** 350 °C, **c** 450 °C, **d** 550 °C, and **e** 650 °C and pH 1 **[30].**

1.7 Gelatin extract [31].

Using a magnetic stirrer, 3.30 g of sodium tungstate dihydrate (Na_2WO_4. $2H_2O$) dissolved in 100 mL of distilled water for one hour to start the WO_3-NS preparation process. The sodium tungstate solution was then completely dissolved at room temperature, and 20 mL of the gelatin solution was added to create a homogenous solution. HCl (2.0 M) was used to acidify the resultant solution until its pH was 1.5. In order to get the desired yellow gel, the produced mixture was stirred for (16 h). After the pollutants were cleaned three times using ethanol and distilled water, the gel was baked for sixteen hours at 60°C to dry it out. Following the curing procedure, the resulting gel was calcined for two hours at different temperatures of 450, 650, and 850◦C to finish creating WO_3-NS with colors of black, green, and yellow.



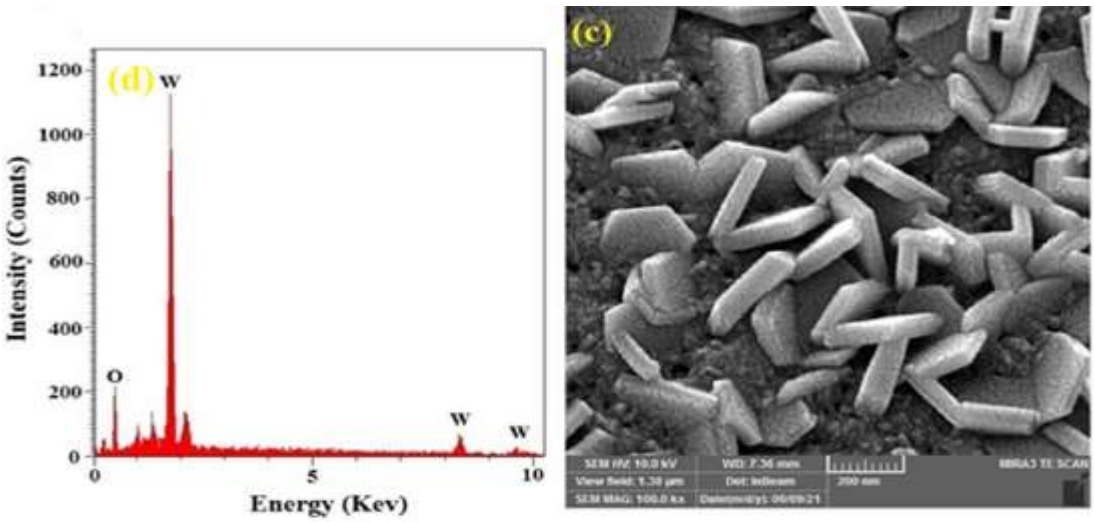


Figure: 8 (a–c) The FE-SEM images of WO_3-NS (d) EDX at 650 ◦C **[31].**

1.8 (vitamin C) [32].

The following procedure was used to create WO_3 nanopowders using the precipitation technique in an aqueous vitamin C solution: A 20-minute room temperature stirring period was applied to a 1 M aqueous solution of *vitamin C*, Tungsten hexachloride WCl_6 dissolved in 50 ml of H_2O, stirred for 30 minutes and at room temp. Next, *vitamin C* was added to the mixture as a pH-controlling, green capping, and reducing agent. The precursor solution (WCl_6/H_2O) was mixed for five hours while a 1 M solution was added dropwise until the pH level reached 2.

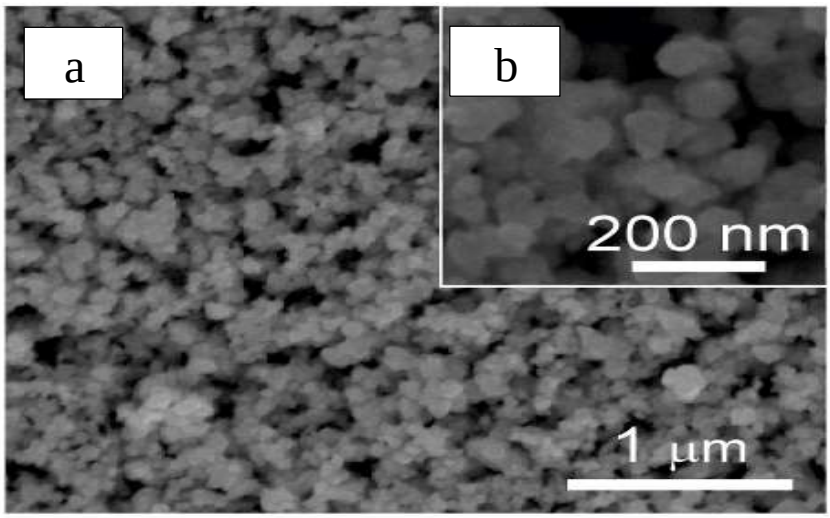


Figure 9: FESEM images of synthesized WO_3 powders: (a) and (b) in vitamin C **[32].**

2. Synthesis tungsten oxide nanoparticles by solvothermal methods.

2.1 Stainless steel autoclave [33].

40 mL (0.025 M) of WCl6 solution in ethanol was put into a 50 mL Teflon-lined stainless steel autoclave, and it was let to stand for 1h., and then heated to 200°C in a muffle furnace to produce WO_3 nanowires. The autoclave was then held for varying amounts of time three, six, and twelve hours. At last, the furnace reached room temperature by natural cooling. A pale blue precipitate was produced following centrifugation, ethanol and distilled water washing, and four hours of drying at 60°C.

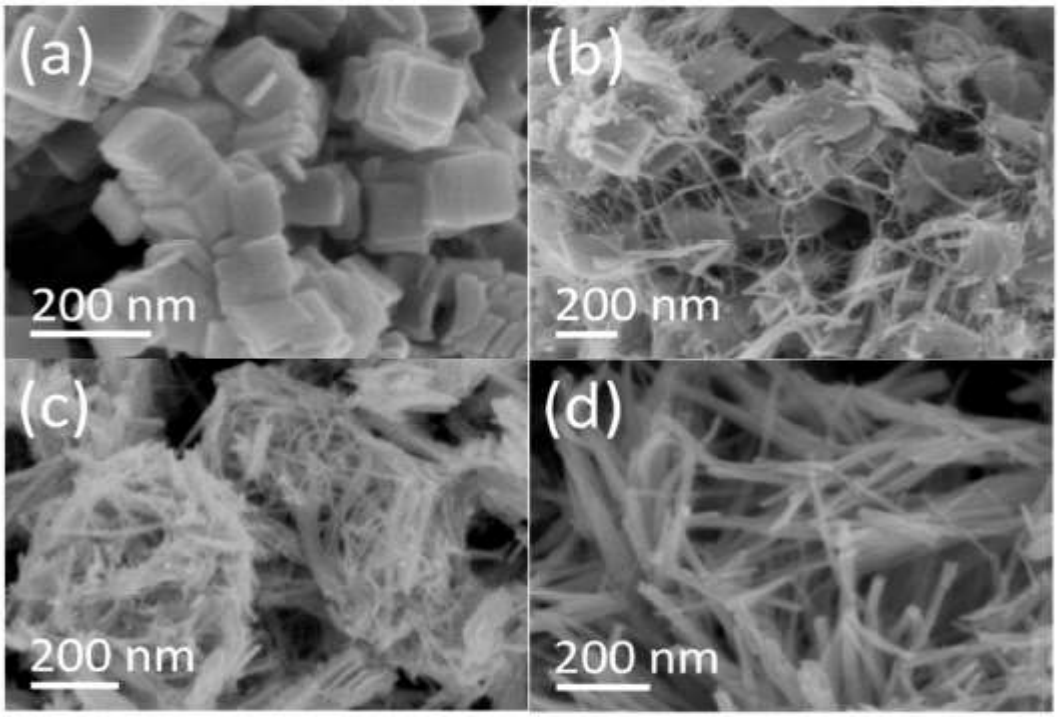


Figure 10: FESEM images of (a) $WO_3 \cdot H_2O$ synthesized at room temp. for 1h (b) 3h, (c) 6h, and (d) 12h solvothermal treatment of $WO_3 \cdot H_2O$ nanoplates at 200°C in ethanol **[33].**

2.2 hydrothermal temperature [34].

In a traditional synthesis, a clear, colorless peroxo-polytungstic acid solution was created by dissolving 2.5 g of tungstic acid (0.01mol) in 10 mL of 30% hydrogen peroxide (H2O2) and 30 mL of filtered water while stirring, after the combination was hydrothermally treated for 24 hours at 200 °C, a white colloidal suspension was produced, centrifugation was used to

gather the products. To create thin coatings, a glass slide was quickly dipped into the white colloidal mixture and withdrawn at a rate of 30 cm/min. altered the hydrothermal temperature from 100 to 200 °C while maintaining the same synthesis conditions in order to investigate the impact, increased the quantity of the peroxo-polytungstic acid precursor from 0.25 to 1.00 mol/L while keeping the hydrothermal temperature at 150 °C in order to examine the impact of this variation, altered the H_2O_2 quantity (0, 5, 10 mL; equivalent to a weight percentage of 0%, 3.75%, and 7.50%, respectively) while maintaining the hydrothermal temperature at 200 °C in order to investigate the impact of H_2O_2 amount.

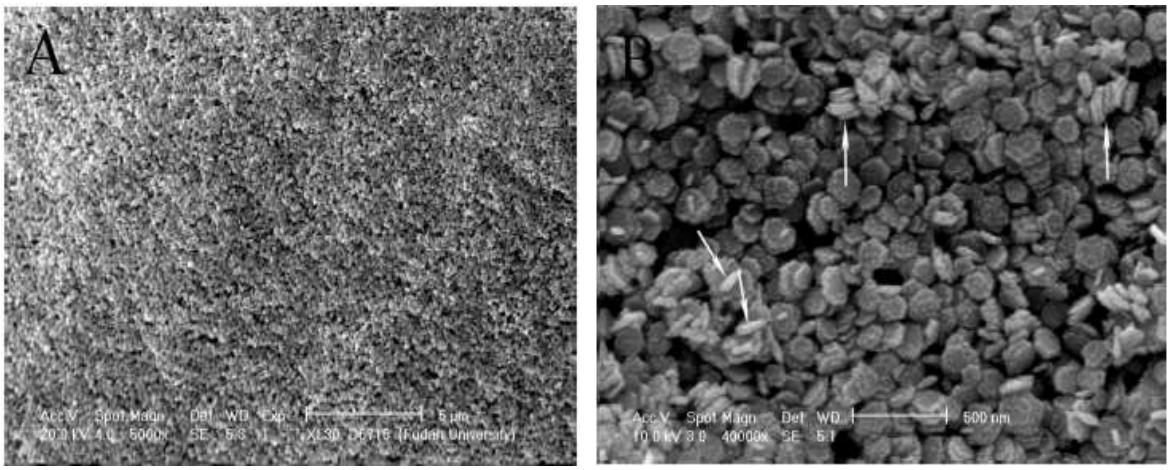


Figure 11: SEM images of $WO_3 \cdot 0.33H_2O$ Nanodiscs **[34].**

2.3 Teflon liner autoclave [35].

Hydrothermal preparation was used to create WO_3 nanoparticles. The following steps were taken during the fabrication process whereas the tungsten-film-covered alumina substrates were positioned on top of the sample holders, which were secured inside the Teflon liner. H_2O vapor was exposed during this process. The Teflon liner was filled with 20 milliliters of distilled water. 4 mm, the liner's H_2O level was lower than the sample holders'. The autoclave reactor received the Teflon liner. The metallic tungsten films were treated for twenty-five hours at 180 °C while being exposed to H_2O vapor. Samples underwent hydrothermal synthesis and were then dried using artificial airflow.

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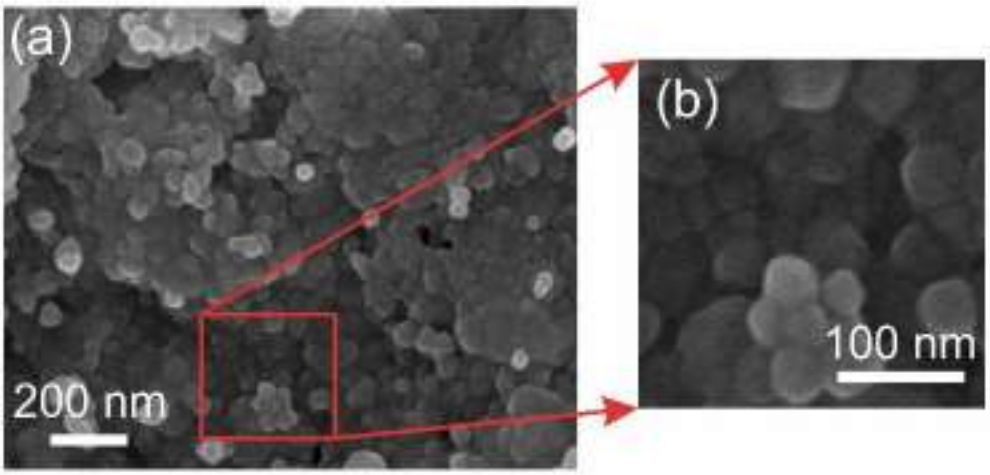
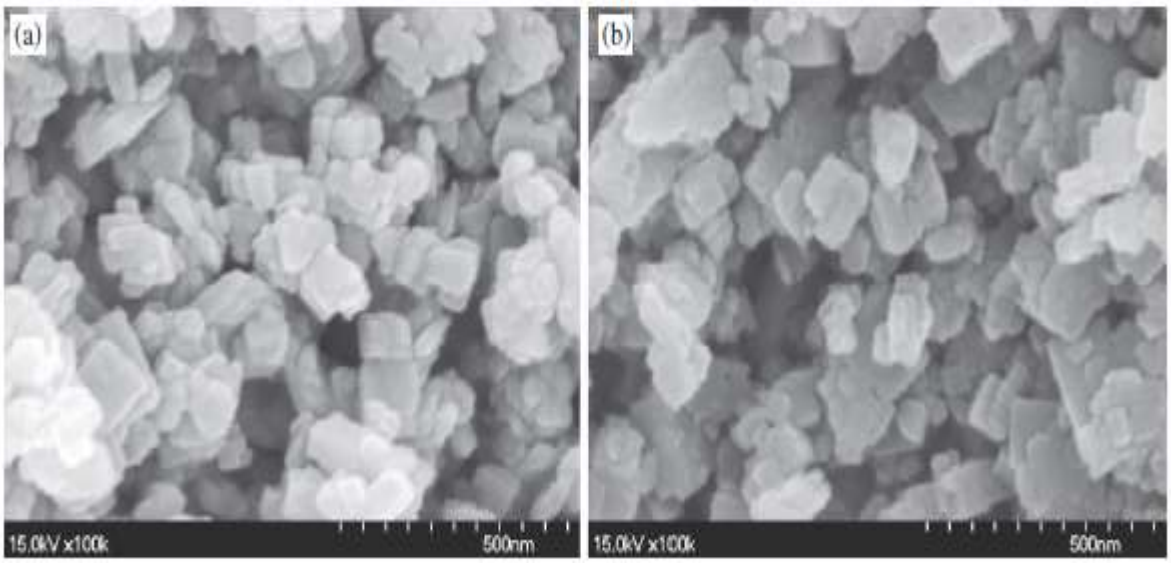


Figure 12: (a and b) FESEM images of the WO_3^{-1} **[35].**

2.4 Stainless steel autoclave with catalytic function of tungsten oxide [36].

Analytical grade reagent without additional purification, 0.5 g of H_2WO_4 powder was mixed with 45 ml of deionized water while being continuously swirled, the suspension that resulted was then put into a 50 cc stainless steel autoclave that was lined with Teflon. For 12 hours, hydrothermal treatments were conducted at 160 C. Following that, the autoclave was left to cool down on its own. The finished powder were gathered, repeatedly cleaned with ethanol, and dried at 80 degrees Celsius in the air.



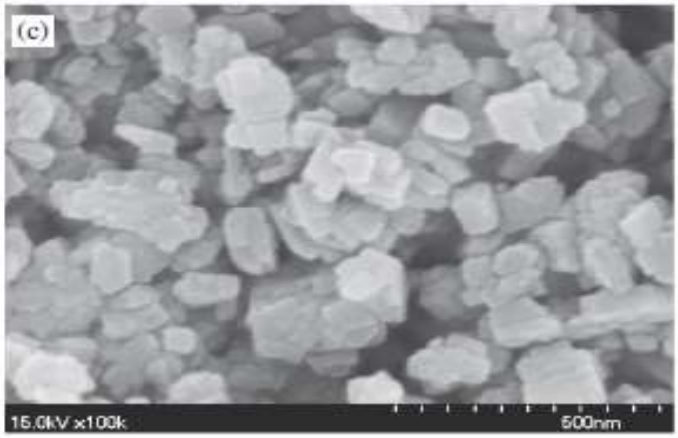


Figure 13: SEM images of the sample prepared at (a) 140, (b) 160 and (c) 180 C **[36].**

2.5 Microwave-assisted method [37].

The $WO_3.0.33H_2O$ snowflake-like particles were created by a hydrothermal process with peroxo-polytungstic acid acting as a precursor, helped by a microwave. In a standard synthesis, the temperature of reaction was kept > 40 °C by gradually adding 4 g of finely powdered tungsten to 20 mL of hydrogen peroxide (30 wt%) in water bath at 10 °C. A colorless and transparent peroxo-polytungstic acid solution was the result. The tungsten content was then brought down to 0.127 mol L^{-1} by diluting the solution with deionized water. A 100 mL Teflon-lined autoclave with a 60% filling ratio was then filled with the diluted solution. After that, an MDS-8 system operating in temperature-controlled mode heated the autoclave to 180 °C for 60 minutes.

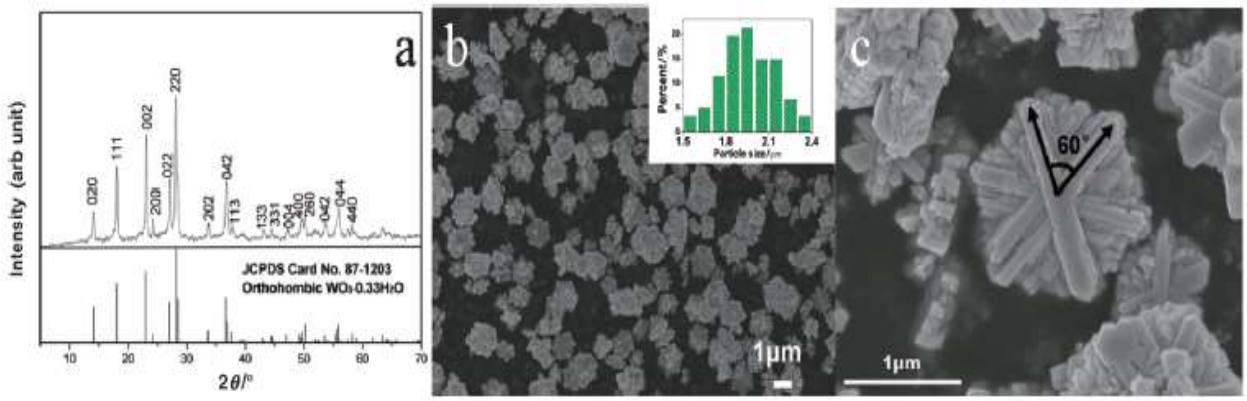


Figure 14: (a) XRD (b, c) FESEM images of the $WO_3.0.33H_2O$ nanoparticles produced by microwave [37].

2.6 Microwave-Assisted Solution Combustion [38].

50 milliliters of deionized water were used to dissolve sodium tungstate dihydrate (Na, $WO_4.2H_2O$) to create a clear solution (0.5M). Under magnetic stirring, the sodium tungstate solution was supplemented with the same molar ratio of HCl until light yellow precipitates were visible, the reaction vessel was placed in a 90°C oil bath. Following that, oxalic acid was added to the reaction system at a ratio of 1:1.6 between sodium tungstate solution and oxalic acid (W/C). Yellow precipitate formed after air drying at 60°C, H_2O nanoplates were cleaned with deionized water. First forming in an acidic environment, $WO_3.2H_2O$ later changed into WO_3. H_2O when oxalic acid is present.

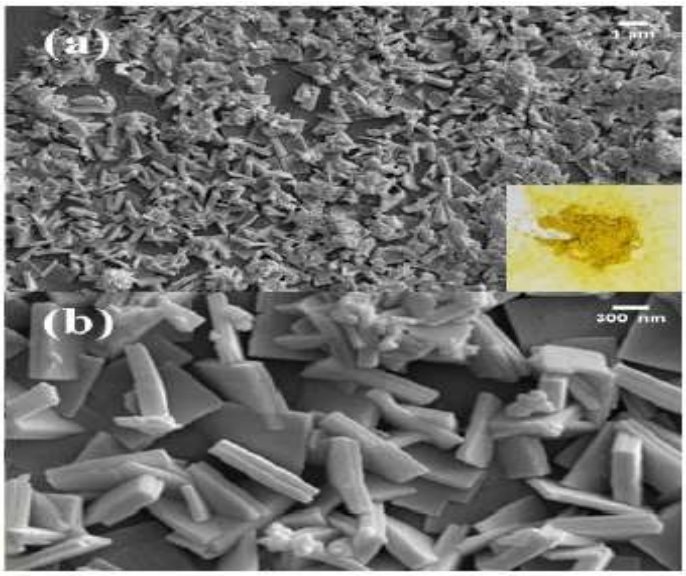


Figure 15: FESEM images of the $WO_3.0.33H_2O$ particles by microwave-assisted solution combustion **[38].**

3. Synthesis tungsten oxide nanoparticles by biosynthesis methods.

3.1 Taguchi Method [39].

The S. maltophilia growing culture was then exposed to physicochemical alteration and incubated with sodium tungstate to look into how different factors affect the generated nanoparticles' PDI and particle size, centrifugation was used to extract the cell detritus and sonication with a QSONICA sonicator to burst the cells, A snakeskin dialysis membrane (10 K MWCO) (Milli Q systems Merck) was used to dialyze the membrane against

deionized water for 24 hours after the supernatant had been filtered through a 0.22µm cellulose acetate membrane, following dialyzing, After being lyophilized in a Buchi lyovapor L-200, the samples were further examined for particle size using a Malvern Zeta sizer Nano DS.

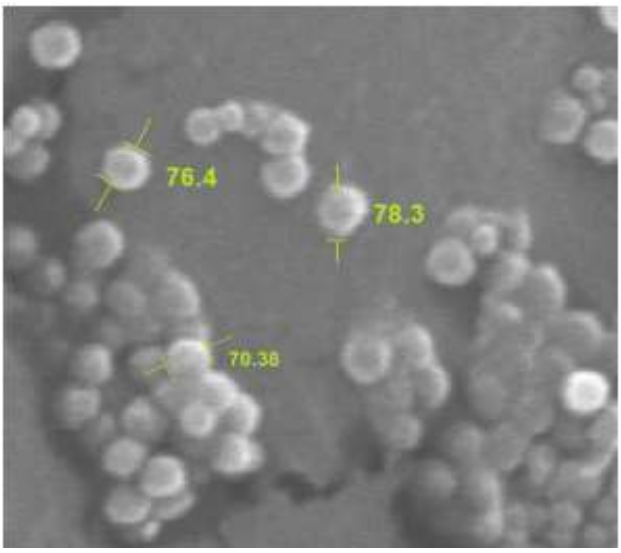


Figure 16: SEM images Of WO_3 NPs synthesized by taguchi **[39].**

4. Synthesis tungsten oxide nanoparticles by sonochemical methods.

4.1 Ultrasonic probe [40].

In the sonochemical synthesis, distilled water was used as a solvent of tungsten hexachloride (99%, WCl_6). Usually, a solution was made using 500 ml of distilled water and 10 g of Tungsten hexachloride WCl_6. The solution was placed on a hot plate at 200 °C. The solution stayed at a boil for the entire procedure. Ultrasonic probe (P100-20 sonic processor, 20 kHz, 1000W) immersed in the solution was used to achieve ultrasonic irradiation. The solvent completely evaporated after the reaction, leaving yellow powder for further examination.

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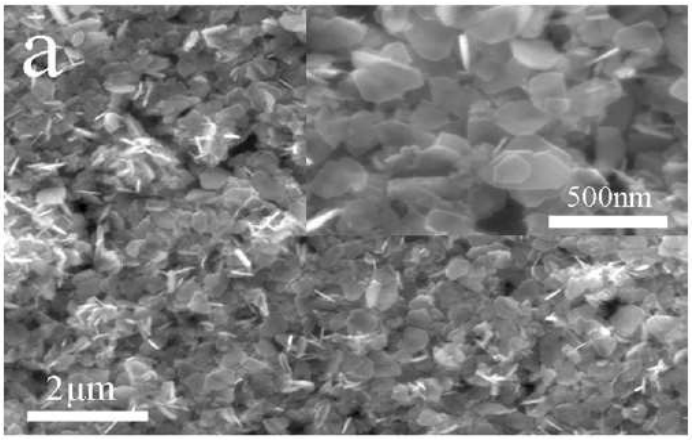

. **Figure 17:** (a) SEM image of tungsten trioxide monohydrate nanosheets by sonochemical **[40].**

Table 1: Show some properties of different synthetic methods

No.	Method	Temp.	Time	pH	Morphology	Average Crystallite Sizes	Ref.
1-	*biosynthesis*	120 °C	60 min	---------	spherical-shaped and packed closely together	25 - 60 nm	25
2-	*Melia azedarach leaves extract*	50°C	4 h	----------	Small spherical nanoparticles or short nanorods	44.84, 42.68, 40.70, 44.66nm for the blank , 2%, 5% and 10% sample respectively .	26
3-	*Rhamnus Prinoides Leaf*	100°C	2 h	----------	spherical shapes nanoparticles	60 nm	27
4-	*Moringa oleifera Lam*	4°C	15 to 20 min.	----------	spherical shape	10 to 20 nm.	28
5-	*Psidium Guajava* leaves	60°C	4 h	----------	rod shape	~ 41 nm.	29
6-	*Spondias mombin*	120 °C	1 h	1, 4, 7, 10, and 13	at pH1, pH4, pH7and pH10 well-defined agglomerated spherical shape , a purely hexagonal shape, a mixture of hexagonal–spherical, agglomerated purely cuboid morphology	at pH 1, 4, 7, and 10 were7.1, 14.7, 28.30, and38.90nm, respectively. at temp. 250°,350°, 450°, 550°, and 650 °C. were 13.1, 14.7, 25.2, 27.1, 29, and 31 nm respectively.	30
7-	*gelatin*	Room Temp.	16 h	1.5	hexagonal shape	40 nm	31
8-	*Vitamin C*	Room Temp.	5 h	2	agglomeration of particles	52 nm	32
9-	*solvothermal*	200 °C	1, 3, 6, 12 h	---------	Nanowires, nanoplates	< 20 nm	33

10-	*solvothermal*	200 °C	24 h	---------	hexagonal disk-like	~ 50 nm	34
11-	*solvothermal*	180 °C	25 h	----------	hierarchical structure composed of nanoparticles	40 nm	35
12-	*solvothermal*	160 °C	12 h	----------	agglomeration of particles	~ 70 nm	36
13-	*solvothermal*	180 °C	60 min	----------	hexagonal snowflake like	1.95 µm.	37
14-	*solvothermal*	90 °C	---------	---------	Orthorhombic 01-084-0886	13.9 nm	38
15-	*biosynthesis*	----------	24 h	----------	spherical-shaped	68 nm	39
16-	*sonochemical*	200 °C	--------	----------	An integral square-shaped nanosheet	100 nm	40

Conclusions

The results of the review indicate that the method of preparing tungsten oxide using the method of extracting the *Moringa oleifera Lam* was the best method used because it is considered one of the green methods that has no effect on the environment, there is no need to use chemicals, and the temperature is 4 degrees Celsius. The preparation method is also distinguished by the fact that the time required to produce nanoparticles is short, and these specifications apply to industrial processes that are economically feasible as well. The nanoparticles are 10-20 nm in size and have spherical shapes, which enhances their role in various applications.

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