Synthesis of MoO$_2$ nanoparticles via the electro-explosion of wire (EEW) method

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Abstract
In this study, MoO$_2$ nanoparticles have been synthesized via the electro-explosion of wire (EEW) method. Different experimental conditions such as different wire thicknesses, media temperatures, current densities, and surfactant concentrations have been applied to determine the effects of each one on the properties of the particles. The polyethylene glycol 20 000 has been used as the surfactant. The FT-IR and XRD results have confirmed the synthesis of MoO$_2$ nanoparticles. The SEM and TEM images were used to calculate the particle sizes. The average particle size of synthesized nanoparticles is between 18.2 nm to 76.7 nm. However, the TEM images shows the exact particle size of 4.3 nm and 2.6 nm in 2 samples. The antibacterial property of synthesized nanoparticles has been tested using a gram negative bacteria, and MoO$_2$ nanoparticles showed antibacterial behavior.

1. Introduction

Molybdenum oxide nanoparticles and their compounds have been used in many applications in medical applications [1, 2], batteries, and sensors [3–5] and in the electronic Industry [6, 7]. There are different ways to synthesis nanoparticles such as sonochemistry [8, 9], chemical vapor deposition [10, 11], sol–gel [12, 13], thermal decomposition [14, 15], chemical precipitation [16, 17] and electro-explosion of wire.

The electro-explosion of wire (EEW) method is one of the most interesting approaches for producing nanoparticles because it is a single-step process, the synthesis rate is high (up to 200 g h$^{-1}$) [18], and it is an environment-friendly method. In this technique, a delicate wire of a conductive metal (usually less than 0.5 mm in diameter) is exploded by an electric discharge that is caused by a high-power DC source. The massive temperature that is created by the electric arc vaporizes the thin wire, turning it into gas atoms [18–20]. Atoms that are chilled in surrounding media develop initial nuclei. Lastly, over a desublimation process, the nanoparticles are synthesized. Many metallic and ceramic nanoparticles have been produced through this technique such as Cu, Ag, Fe, Al, Ni, TiO$_2$, Cu$_2$O, ZnO, and Fe$_3$O$_4$ nanoparticles [21–26]. In this approach, some experimental conditions including wire thickness, current density, input power, and medium temperature change the properties of the product.

The antibacterial property found in some nanoparticles such as Cu [27], Ag [28], and Mn$_3$O$_4$ [29] has been studied to develop a new method to replace traditional techniques using organic agents. One of the reasons that the nanoparticles are good substitutes for conventional antibacterial agents is that they have strong antibacterial activity in small amounts [30]; some of the nanoparticles and nanostructures such as MgO [31] and ZnO [32] contain the required mineral elements for the human body.

The bacterial cells usually have sizes of micrometers, while outer membranes have a permeable pore size in the range of nanometer where nanoparticles can easily be absorbed by these pores [33]. Precise mechanisms have not been reported for metal nanoparticles, but according to the study by Hajipour et al [34] the effects of metal ions on existing proteins and enzymes in the bacteria lead to the antibacterial activity of metal nanoparticles. The
antibacterial activity of nanoparticles occurs through ions that are released from nanoparticles, and this activity is increased by diminishing the size of the particle size [30], and these ions communicate directly with the microbial membranes and disturb the intracellular biochemical activities.

In the work by Krishnan et al [35], when the medium temperature was reduced, the mean particle size will increased. The current density affect on nanoparticles size has been investigated [24], and by increasing the current densities, the mean particle size decreased. Some analysis focused on the outcome of adding surfactant on the sounding media [24], and they were reported that by adding surfactant, the particle size decreased.

The antibacterial potential increases by raising the volume of nanoparticles; hence, higher concentrations of nanoparticles are remarkable and significant antibacterial effects. Different mechanisms have been reported for the antibacterial effect of nanoparticles such as the decomposition of nanoparticles and formation of Reactive Oxygen Species (ROS), electrostatic interaction of nanoparticles with the bacterial cell wall, and optical photocatalytic activation of nanoparticles.

ROS formation is one the most important mechanism assumed by nanoparticles. This ROS molecule causes cell death by restraining or changing DNA replication, Respiratory cycle, synthesis cycle or food metabolism cycle Protein.

Some nanoparticles act as the efflux pump inhibitor. Efflux pump is existing in the membrane of bacteria cell and they are implicated in extracting toxic chemicals and waste material out of the cell [36].

In a work by Chen et al [37] they shown that there are a direct connection between the nanoparticle size and the cell interaction. They mentioned by decreasing the size of nanoparticle interaction between nanoparticles and the cell membrane increased and the they show more toxicity to the cell.

One of the possible reasons for the antibacterial effect is interaction between metal oxide nanoparticles and the outer membrane surface of the bacterial cell. Optical activation of nanoparticles is the main mechanism for metal oxide nanoparticles, and this activation induces oxidative stress by reactive oxygen species which include superoxide radicals, hydroxyl radicals, hydrogen peroxide, and discrete oxygen that can form on the surface of metal oxide nanoparticles and can damage existing proteins and enzymes in the bacteria and eventually can cause the death of bacterial cells [38]. In addition, a similar study shows that MoO3 nanoparticles have antibacterial properties.

In this study, MoO2 nanoparticles prepared with the EEW method have been synthesized in different experimental circumstances such as various applied current densities, temperatures, and surfactant concentrations. A probable parameter that can influence the average particle size in the EEW method is wire thickness. Two different wire diameters have been used in this study to evaluate this supposition, and the synthesized nanopowders have been analyzed using transmission electron microscopy (TEM), scanning electron microscopy (SEM), Fourier transform infrared (FT-IR), and x-ray Diffraction (XRD) techniques to characterize the chemical and physical features of synthesized nanoparticles. To examine the antibacterial property of MoO2 nanoparticles, two samples have been tested with different concentrations in the presence of a gram-negative bacteria via the optical density measurement method.

### 2. Experimental

#### 2.1. Sample preparation

The experimental conditions for the syntheses of samples H1 to H10 are shown in table 1. All samples were prepared via the EEW method at different temperatures between 1 °C and 60 °C and current densities between

| Sample | Wire thickness (mm) | Temperature (°C) | Voltage (V) | Current density (KA/mm²) | Surfactant concentration (ppm) | Average particle size (nm) |
|--------|--------------------|-----------------|-------------|--------------------------|------------------------------|---------------------------|
| H1     | 0.25               | 35              | 82.2        | 2.0                      | —                            | 36.4                      |
| H2     | 0.25               | 35              | 82.2        | 5.2                      | —                            | 29.8                      |
| H3     | 0.25               | 35              | 82.2        | 10.2                     | —                            | 27.6                      |
| H4     | 0.25               | 1               | 82.2        | 10.2                     | —                            | 76.7                      |
| H5     | 0.25               | 15              | 82.2        | 10.2                     | —                            | 33.0                      |
| H6     | 0.25               | 50              | 82.2        | 10.2                     | —                            | 23.8                      |
| H7     | 0.25               | 60              | 82.2        | 10.2                     | —                            | 18.2                      |
| H8     | 0.35               | 35              | 82.2        | 5.2                      | —                            | 62.1                      |
| H9     | 0.35               | 35              | 82.2        | 5.2                      | 6000                         | 42.9                      |
| H10    | 0.35               | 35              | 82.2        | 5.2                      | 10000                        | 29.8                      |
2.0 KA/mm² and 10.2 KA/mm². Samples H1 to H7 were synthesized via 0.25 mm Molybdenum wire, and samples H8 to H10 were synthesized via 0.35 mm Molybdenum wire. Samples H1 to H8 were synthesized in DI water without surfactant, and samples H9 and H10 were synthesized in the presence of different amounts of Polyethylene glycol 20 000 (PEG) as the surfactant. The synthesized samples were prepared as solid specimens for XRD and FT-IR, and a suspension was prepared for SEM and TEM characterizations. The gram negative bacteria which was used for the antibacterial test was Escherichia coli (ATCC:25922). The solid medium containing agar was used to prepare bacterial culture media. These media were inoculated with Escherichia coli bacteria and placed into an incubator shaker set at 37 °C to reach a standard bacterial concentration 10⁶ of cfu/ml at the wavelength of 600 nm. The samples with given concentrations were prepared in the presence of nanoparticles and were placed on the shaker with the specific round and temperature of 37 °C to provide the necessary conditions for the growth of the bacteria.

2.2. Characterization methods
The samples were stable in liquid (after 10 days without any motion, the samples were not settled), and to separate the nanoparticle from the liquid, samples were placed in a centrifuge machine and rotated with 13 000 rpm for 45 min. After the sedimentation of the particles, samples were dried in an oven at 45 °C for 72 h. A PANalytical x-ray diffractometer was used for XRD characterization employing high-intensity Cu-Kα radiation (λ = 1.54060 Å). The IR spectra were recorded in the range of 400–4000 cm⁻¹ using a Bruker Tensor27 spectrometer. A JEOLSEM JSM-7401F (using accelerating voltage of 2 KV), and JEOLTEM JEM-2010F (using accelerating voltage of 200 KV) were used for detection of particle size and morphology. The SEM samples were set up via the following process: a drop of the suspension which was contained particles was dripped on a 1 × 1 cm² glass sheet. After the deposit dried, the glass sheet was used as an SEM sample. The optical density measurement method at a wavelength of 6000 nm (OD-600 nm) using Spectro UV-VIS UVD-3200 machine was used within 2 h to determine the bacterial growth rate in each sample.

Figure 1. SEM image of samples (a) H1, (b) H2, and (c) H3.
3. Results and discussions

SEM images of samples H1 to H3 are shown in figures 1(a)–(c). The size distribution diagrams shows that the average particle size reduced from 36.4 to 27.6 nm by increasing the current density from 2.0 KA mm$^{-2}$ to 10.2 KA mm$^{-2}$. This phenomenon is explainable because in high concentrated electric energy, a few atoms form an embryo, so the ultimate size of particle reduced. When the electrical energy increases, the temperature increases because the explosion rates works too, so the temperature of the MoO$_2$ molecules decreases at a faster rate. As a result, each MoO$_2$ particle had a low concentration of MoO$_2$ molecules. In contrast, when the electrical energy diminishes (by reducing current density), the explosion temperature declines, so the cooling rate will be reduced, and the chance of gathering a larger number of atoms expands. This result agrees with the results obtained by other reports [19, 24, 28].

SEM images of samples H3 to H7 are presented in figures 2(a)–(e). According to the size-distribution diagrams, the average particle size increased from 18.2 nm to 76.7 nm by reducing the temperature from...
60 °C to 1 °C. When the explosion occurs, regarding the same current density, each evaporated atom receives almost an equal amount of heat. The atoms cool down and form an embryo, and because the medium temperature is lower, it takes more time to reach that temperature, so the cooling process is increased. This longer cooling process gives the atoms more opportunity to merge and create an embryo. As a result, the average particle size increases as the medium temperature decreases. Some other researchers have reported the same result [35].

SEM images of samples H2 and H8 are presented in figures 3(a) and (b). The size-distribution diagrams show that by increasing the diameter from 0.25 mm in sample H2 to 0.35 mm in sample H8, the average particle size increases from 29.8 nm to 62.1 nm. This result is because both samples have the same current density and receive equal electrical energy. When the explosion occurs, a specific quantity of energy is shared between evaporated atoms. In sample H8, each explosion creates more evaporated atoms than H2 (because of the larger exploded area in H8 than in H2 regarding their thicknesses). Each particle in sample H8 receives less energy when it evaporates than particles in sample H2 because that specific quantity of energy is shared between more particles in sample H8 than in H2. As a result, it takes more time for H2 particles than H8 particles to gather and form an embryo regarding their higher energy. In addition, the higher energy influences the growth rate, and the growth rate is slower in H2 than in H8. All the mentioned reasons have caused the average particle size in H8 to almost double that in sample H2.

The TEM image of samples H4 and H8 are presented in figures 4(a) and (b). The size-distribution diagram shows the exact particle size of 4.3 nm and 2.6 nm respectively. This difference between SEM and TEM based calculation of particle size can refer to the fact that in SEM image, it’s possible that the particles congregate and create an Agglomeration, but in the TEM image the exact particle size is shown.
The SEM image of samples H8 to H10 are shown in figures 5 (a)–(c). The samples H9 and H10 were produced in the presence of PEG as the surfactant. The results show that the average particle is decreased from 62.1 nm to 42.9 nm with the added surfactant because of the role of PEG molecules. This fact is reliable due to the role of PEG molecules. When some surfactant molecules are presented in the synthesis medium, they surround the embryos, so the growth of embryos is disturbed, and the average particle size is diminished. In contrast, by raising the surfactant concentration, this phenomenon is intensified, so the average particle size is diminished. The results show increasing the surfactant concentration from 6 000 ppm to 10 000 ppm caused the particle size decreases from 42.9 nm to 29.8 nm. This fact is in good agreement with another report [24, 29].

The XRD patterns of samples H3, H8, H9, and H10 are presented in figures 6 (a)–(d). Based on the Rietveld method, the percentage of oxide phase in samples H3 and H8 are 96% and 86%, respectively. This relationship is because the current that was used for producing these samples was 500 A, so the H3 particles had more energy than the H8 particles, In addition, based on the differences in diameters, they reacted remarkably with sounding media. In contrast, the percentage of surface atoms of the 0.25 mm wire is more than that of the 0.35 mm wire, so the Molybdenum atoms of sample H3 encounter more free oxygen atoms, and the percentage of the oxide phase increases more than in sample H8. The free oxygen atoms form from evaporation and decomposition of the water and surfactant molecules. As a result, the percentage of the oxide phase in H3 increased. In samples H8, H9 and H10, the percentage of oxide phase were 86%, 92%, and 100%, respectively, because of the role of the surfactant in the synthesis process. When the explosion occurs, the high temperature that is caused by the explosion causes some of the molecule bonds to break. In this case, some of the H–O bonds are broken, creating O atoms in the area. By adding surfactant molecules in sounding media, the creation of O atoms is accelerated, so the chance of forming MoO2 particles increases, which this result agrees with the result of other research [24].

Regarding figure 6, the XRD peaks of sample H9 that contains 6000 ppm PEG are sharper than H10 with 10 000 ppm PEG, and the sample H8 does not have any surfactant, as indicated by the Scherrer equation. As table 1 shows, sample H10 has finer particles than samples H8 and H9. Regarding the Scherrer equation, the peak width increases as the particles size decreases.
The FT-IR spectra of samples H8 and H10 are shown in figures 7(a) and (b). The characteristic bands of sample H8 appear at 3429 cm$^{-1}$, 895 cm$^{-1}$, and 560 cm$^{-1}$. The 3429 cm$^{-1}$ broad peak is associated with the H–O band [24, 30], which belongs to the absorbed water. The peak located at 895 cm$^{-1}$ is related to Mo=O band.

Figure 6. XRD pattern of samples (a) H3, (b) H8, (c) H9, and d) H10.
In addition, the $560 \text{ cm}^{-1}$ peak is associated with the Mo–O band [31, 33]. The chemical structure of MoO$_2$ or Molybdenum (IV) Oxide is $\text{O}=\text{Mo}=\text{O}$. The probable reason for existing Mo–O band is because of the high temperature, which is produced during the synthesis process, some of the Mo=O bands has broken and became Mo–O bands. The H10 peaks appeared at 3414 cm$^{-1}$, 3209 cm$^{-1}$, 2914 cm$^{-1}$, 1401 cm$^{-1}$, 1283 cm$^{-1}$, 1095 cm$^{-1}$, 945 cm$^{-1}$, 897 cm$^{-1}$, and 553 cm$^{-1}$. The peaks located at 3414 cm$^{-1}$ and 3209 cm$^{-1}$ are related to the H–O band that belongs to absorbed water [24, 30] and the H–O band in the surfactant’s structure. The 2914 cm$^{-1}$ peak is associated with the C–H band [39]. The peak at 1401 cm$^{-1}$ is attributed to the –H–C–OH band [35, 38]. The peak at 1283 cm$^{-1}$ is related to the –CH$_2$ band [40, 41]. The 1095 cm$^{-1}$ peak is related to the C–O band [42]. The peak at 945 cm$^{-1}$ is attributed to the C–C band [43]. The 897 cm$^{-1}$ and 560 cm$^{-1}$ peaks are associated with Mo=O and Mo–O [31–33]. The chemical formula of Polyethylene glycol 20 000 is H($\text{OCH}_2\text{CH}_2$)$_n$OH. The results show that the H10 sample’s FT-IR spectra contain all the chemical bonds that exist in PEG. Both samples H8 and H10 contained Mo=O and Mo–O which confirm the XRD results and proves the existence of the MoO$_2$ phase in samples H8 and H10.

The antibacterial property of nanoparticles against Escherichia coli was evaluated with a growth curve method using optical density measurement. In this method, more optical density indicates a higher number of live bacteria [34].

The mechanism accepted by gram negative and gram positive bacteria for attachment of the nanoparticle to the cell surface, transporting in to the cell is different and it is depended on the different membrane structure.

Gram negative bacteria have a triple layer which made of peptidoglycan in their cell membrane and the porins are exist in the outer layer. Porins serve as an ion channels present in external peptidoglycan layer that facilitates passive diffusion of nanoparticles inside the cell. There are other functions for porin such as cell–cell interaction, nutrient uptake by the cell, and recognition.
MoO$_2$ nanoparticles can dissolve in the medium and create Mo$^{4+}$ ion. The Electrostatic attraction of ions to negative charged membrane known as a common way for nanoparticle to attach to the surface of the bacteria cell.

The figures 8(a) and (b) show optical density diagram at a wavelength of 600 nm in time for 250 ppm, 500 ppm, and 750 ppm concentrations of synthesized nanoparticles in the presence of bacteria. Given the resulting curves, in the same conditions for different concentrations, by increasing the concentration amount of nanoparticles fewer bacteria are likely to survive because of the more contact with nanoparticles; as a result, the antibacterial property of molybdenum oxide nanoparticles augments. Also, for a certain concentration of nanoparticles, the nanoparticles can easily absorb into the existing cavities on the surface of bacteria by decreasing the size of particles and accelerate the bacterial destruction process and accordingly the antibacterial property increases. This result is in good agreement with pervious studies $^{37, 44}$

4. Conclusion

In this research, the MoO$_2$ nanoparticles were synthesized with the electro-explosion of wire method. The average particle size was between 18.2 nm to 76.7 nm. The TEM image shown the exact particle size of 4.3 nm and 2.6 nm for two samples. Some parameters including current density, media temperature, wire thickness, and surfactant concentration change based on the synthesis variables to investigate the effect of each synthesis variable on average particle size. Increasing current density from 2.0 KA mm$^{-2}$ to 10.2 KA mm$^{-2}$ caused the average particle size to decrease from 36.4 nm to 27.6 nm. The average particle size decreased from 76.7 nm to 18.2 nm by increasing the media temperature from 1 °C to 60 °C. Increasing the wire diameter from 0.25 mm to 0.35 mm caused the average particle size to increase from 29.8 nm to 62.1 nm. By increasing the diameter, the percentage of the oxide phase rose from 86% to 96%. Adding polyethylene glycol as the surfactant caused the average particle size to reduce from 62.1 nm to 42.9 nm. Also, by raising the surfactant concentration from...
6000 ppm to 10 000 ppm, the average particle size decreased from 42.9 nm to 29.8 nm. Adding the surfactant also affected the percentage of oxide phase too. According to the Rietveld method, the percentage of oxide phase changed from 86% to 100% by increasing the surfactant concentration from 0 ppm to 10 000 ppm. FT-IR and XRD results prove the forming of MoO₂ in the presence of PEG and the absence of it. Antibacterial behavior of samples H3 and H8 against Escherichia coli was also investigated. Generally, antibacterial behavior increased with further reduction in average particle size from 62.1 nm in sample H8 to 27.6 nm in sample H3 and increased with increasing the concentration of molybdenum oxide nanoparticles.

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