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Research Article

Fabrication and study on the structural and photocatalytic characterization of Ag doped ZnO nanostructure materials

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ABSTRACT

ZnO and 10% Ag doped ZnO (10%Ag-ZnO) nanopowders have been researched and fabricated with using extracts from red peony leaves. These materials have been prepared by route of a simple wet chemical method and calcined at a low temperature of about 400^{o}C for 2 hours. Ag^+ ions was doped into the ZnO material compared to Zn^{2+} ions at a molar ratio of 1: 10. Methods to study the characteristics of nanopowders have been used such as XRD, SEM/EDX and reflection spectroscopy. The Kubelka–Munk method based on the reflectance spectrum of the sample was exploited to determine the optical gap energy (Eg) of ZnO and 10% Ag doped ZnO nanopowder samples. The results show that the Ag doped ZnO nanopowder has a smaller average crystal size than undoped ZnO (these value are 22.6 and 32.27 nm, respectively). With doping into transition metal oxide semiconductor material by ions with a radius much larger than the radius of the host ions of the transition metal oxide crystal lattice (the radius of the Ag^+ ion (1.22 Å) is higher than the radius of the Zn^{2+} ion (0.74 Å)), it is likely that the Ag element will be doped into the ZnO crystal lattice in an interstitial form. The Eg value of 10%Ag-ZnO nanopowder is also reduced compared to undoped ZnO, reaching a value of Eg≈ 3.16 eV (the ZnO sample reaches a value of Eg ≈ 3.28 eV). The Ag doped ZnO sample has improved the material's optical application range, achieving high photocatalytic efficiency (97.8%) for decomposing methylene blue (MB) dye solution under 40 minutes of visible light irradiation. The 10%Ag-ZnO nanopowder is prepared from using red peony leaf extract -this is one of the "green" manufacturing methods. This prepared material has achieved outstanding advantages compared to undoped ZnO material such as reducing the average crystal size, reducing the optical band gap energy, and achieving photocatalytic decomposition reaction for MB dye solution under visible light with high efficiency.

Key words: Wurtzite ZnO, Ag doped ZnO, Kubelka–Munk, optical band gap, visible radiation

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INTRODUCTION

ZnO is a semiconductor material with a large band gap energy at room temperature (~3.37 eV). Wide bandgap semiconductors have the advantage of having high breakdown voltage, being able to withstand large electric fields, operating in high temperature environments, providing large capacity, and large exiton binding energy (about 60 meV)[1–6]. ZnO material has the ability to be applied in many fields of optoelectronics such as manufacturing light-emitting diodes, thin-film fluorescent electrical components, transparent thin-film transistors, etc. ZnO has a combination of many valuable properties include electrical properties, optical properties, sustainability with hydrogen environment, compatible with applications in vacuum environment, good thermal conductivity and thermal stability, quite cheap price compared to materials with similar properties[7–14]. Silver (Ag) is a precious metal playing a very important role in many fields. With good antibacterial properties, Ag is often used to make jewelry, to protect health or to make food containers to prevent rancidity. Also due to its antibacterial ability, Ag and Ag compounds are widely used in medical products, and other superior properties of Ag such as optical properties, high thermal conductivity, and good electrical conductivity in most of all metals. Therefore, Ag has many important applications in the fields of electrical, electronic, and optical engineering[15–17]. Nano-meter-sized Ag materials have all the properties of bulk silver such as electrical conductivity, thermal conductivity, catalytic ability, electro-optical properties, and bactericidal ability. In addition, Ag nano has many different properties compared to metallic silver thanks to nano properties such as large surface area, good dispersion ability in solvents, typically good bactericidal and catalytic ability. Much better than Ag metallic or Ag ionic with much less Ag needed to use, contributing to saving production costs[18–20]. ZnO nanomaterials doped with Ag+ ions (Ag/ZnO) have many practical applications. These nanoparticles, in addition to general

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properties such as: electrical, optical, magnetic, catalytic... also have a number of other important properties such as bactericidal ability, application in the electronics industry and photodegradation ability for sustainable organic substances, dyes, lignin,... Using Ag/ZnO composite as a photocatalyst could significantly reduce the amount of catalyst needed and be more efficient. Some research experiments showed that the photodegradation ability using nano ZnO reached the highest organic carbon reduction of 7%, while using Ag/ZnO the organic carbon reduction ability is 2 times greater than ZnO (15%) thus reducing the toxicity of organic molecules [21–24]. There are many fabricated methods for the Ag/ZnO material such as chemical vapor deposition method, photochemical reduction method, chemical ultrasound method, etc. These methods face many difficulties due to high energy consumption and reaction conditions harsh response. Therefore, recent research has prioritized the synthesis of nanomaterials based on biological methods (green methods). This method is simple, easy to implement, and produces materials with smaller, uniform, safe, and controllable particle sizes. A significant number of green approaches use natural sources such as microorganisms and plant extracts to create nanomaterials [21–24].

In this report, we fabricated 10% Ag doped ZnO and ZnO materials using red peony leaf extract solution by route of a simple wet chemical method and calcined at a low temperature. Study on the structural and MB dye photocatalytic decomposition under visible light radiation.

MATERIALS AND METHODS

Chemicals

Zinc acetate dihydrate ($Zn(CH_3COO)_2.2H_2O$ 99%, AR-China); Silver nitrate ($AgNO_3$ 99.98%, AR-China) Ethanol (alcohol, C_2H_5OH 99.7%, AR-China); double distilled water.

Experimental process

Preparation of red peony leaf extract solution: Accurately weigh 20 grams of chopped red peony leaves, boil in 100 ml of distilled water at 70^oC, and stir this mixture slowly for 6 h. After that, the solution of boiling peony leaves was filtered and vacuumed to obtain the extract solution.

Fabrication of Ag doped ZnO nanopowder samples (10%Ag-ZnO): A mixed solution consisting of 50 ml of extraction solution and 50 ml of 0.2M $Zn(CH_3COO)_2.2H_2O$ salt solution. Next, add a calculated amount of $AgNO_3$ salt in molar ratio of Ag^+ : Zn^{2+} = 1 : 10. This mixed solution was continuously stirred with a stirring rate of 350 rpm at a temperature of 70^oC until it was concentrated and transformed into a paste powder. This paste mixture was dried at 110^oC for 24 h. Then calcined at 400^oC for 2 h, a fine powder sample was obtained and was named as 10%Ag-ZnO. The ZnO nanopowder sample was prepared similarly according to the above process but without adding $AgNO_3$ salt, used for comparison and sample was named as ZnO.

+ Methods for determining the materials properties. X-ray diffraction method (XRD, X'pert Pro), Cu-Kα radiation (λ= 1.54065 Å), speed, scanning angle 0.03^o/2s, $2\theta \approx 25\text{-}75^o$); scanning electron microscopy/ energy dispersive spectroscopy (SEM/EDX, Hitachi TM4000 Plus); solid UV-Vis absorption/reflectance spectroscopy (Jasco V-750).

+ *Investigation of photocatalytic properties.* Prepare the MB solution (10 ppm). The photocatalytic reaction mixture included MB solution and a 10%Ag-ZnO fine powder photocatalysts with a concentration of 0.025 g/mL. The reaction mixture solution was stirred in complete darkness for 1 h to reach an equilibrium of adsorption and desorption. Then, the visible light source was radiated on the reaction mixture solution (the visible light source was taken from the Osram 220V-250W lamp source, Philip brand). At each period, 2 mL of solution was extracted and measured on a UV-Vis photometric system (Agilent 8453). The decomposition efficiency of MB dye was determined according to the following formula (in which, C_o and C_t were the initial concentration of MB solution and at time t, respectively).

$$H\ (\%) = ((C_o\text{-}C_t)/C_o)x100\ (\%)\ (1)$$

RESULTS AND DISCUSSION

X-ray diffraction spectra analysis

Figure 1 was the X-ray diffraction spectra (XRD) of ZnO and 10%Ag-ZnO samples. On the XRD spectra indicated that diffraction peaks appeared at positions 2q ≈ 31.7°, 34.3°, 36.2°, 47.45°, 56.6°, 62.8°, 67.25° and 69.2^o which assigned as (100), (002), (101), (102), (110), (103), (112)and (201) lattice planes of ZnO-hexagonal wurtzite structure (JCPDS card No. 89-7102). In addition, on the XRD spectra, no spectral peaks of Ag element or Ag compounds appeared. Thus, it could be said that ZnO, 10%Ag-ZnO samples were hexagonal wurtzite ZnO single-phase [10,11,20–24]. To calculate the average crystallite size of samples, applying Debye-scherrer equation [10,11,23,24]: D= Kl/cosq (2). (In which, D is average crystallite size (nm); K is the shape factor value; l is X-ray wavelength (l=

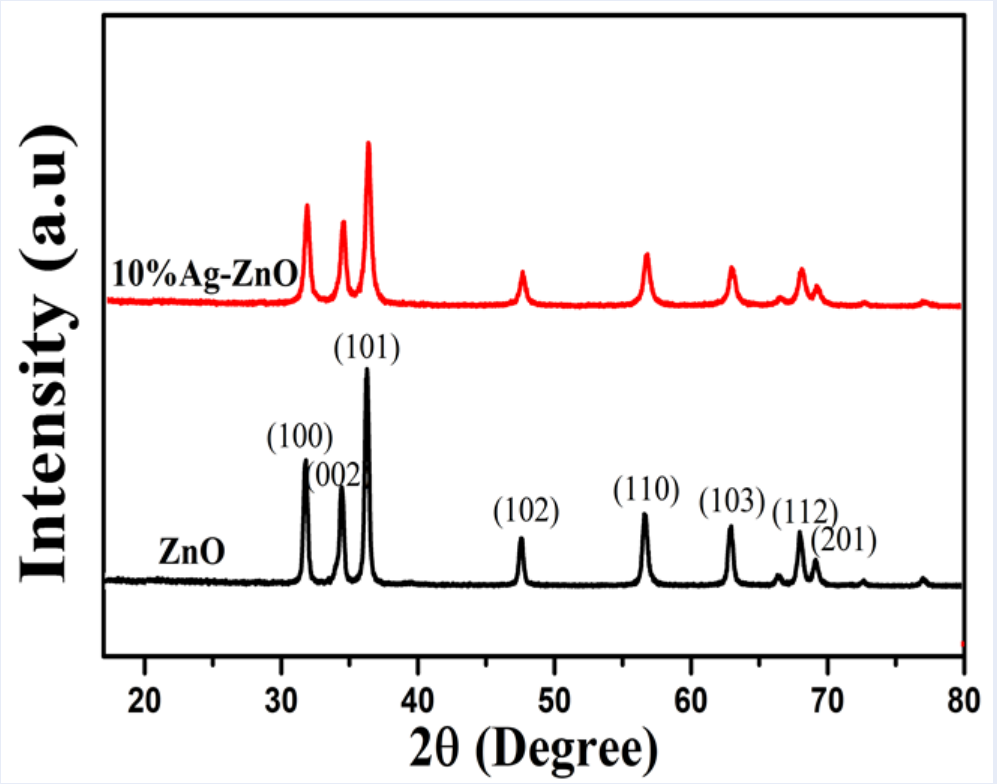

Figure 1: The X-ray diffraction spectra of ZnO and 10%Ag-ZnO samples

1.54056 Å); is the full width at half maximum (radian), q is Bragg angle (radian)).Calculated results determined that the average crystallite size of ZnO and 10%Ag-ZnO samples was 32.27 and 22.6 nm, respectively. It was found that the Ag doped ZnO sample reduced the average crystal size more than undoped ZnO sample. Thus, doping Ag into ZnO prevented the ZnO crystal growth process, therefore, decreased the material's average crystal size. It is noticed that the radius of Ag^{+} ion is higher (1.22 Å) than the radius of Zn^{2+} ion (0.74 Å). So, if the Ag element was doped into the ZnO crystal lattice, it would be doped in an interstitial form [7,10,11,21–24].

Scanning electron microscopy analysis

The surface morphology of the fabricated samples were studied by scanning electron microscopy (SEM) method. Figure 2 showed the SEM image results of ZnO and 10%Ag-ZnO samples. In the SEM image results, it was observed that the ZnO sample had a spherical shape and is evenly distributed on the surface of the material. The 10%Ag-ZnO sample showed that the shape of the crystal particles created was smooth and porous, evenly distributed on the sample's surface. Thus, the doping of 10% Ag^{+} ions to the ZnO material had changed the surface morphology of the material, making the material surface smoother and more porous. The energy dispersive X-ray (EDX) spectra results of ZnO and 10%Ag-ZnO samples were shown in Figure 3. The EDX spectra indicated that the ZnO sample only had spectral peaks belong to the two Zn and O elements that made up the ZnO material. Meanwhile, the 10%Ag-ZnO sample had spectral peaks of the Zn, O and Ag elements. Thus, it could be further confirmed by analyzing the XRD results above that the 10%Ag-ZnO sample had Ag elements doped into the ZnO lattice. In addition, on the EDX spectra of the samples, no other strange element peaks were observed. Thus, it could be seen that the ZnO and 10%Ag-ZnO samples were prepared in pure form of hexagonal wurtzite ZnO single- phase.

Reflection spectra analysis

Figure 4 showed the reflectance spectra of ZnO and 10%Ag-ZnO samples. The reflection spectra showed that the reflectivity in the wavelength region 250-370 nm was low, from the wavelength region 370-400 nm the reflectance increased rapidly, then the reflectivity continued to increase steadily. This reflection spec-

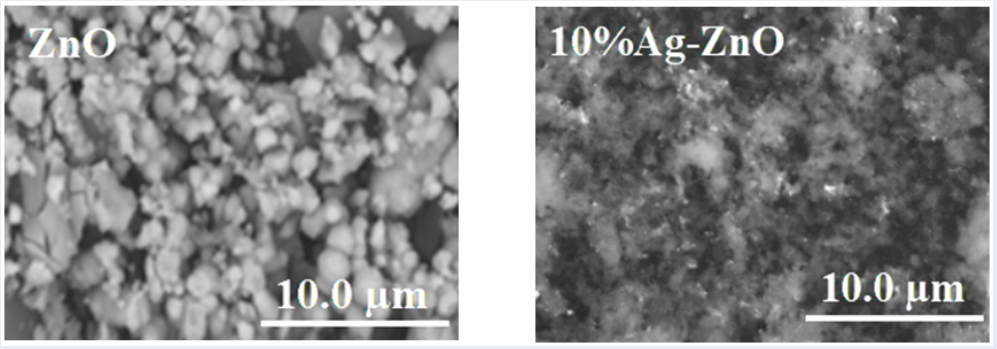

Figure 2: Scanning electron microscopy (SEM) of ZnO and 10%Ag-ZnO samples

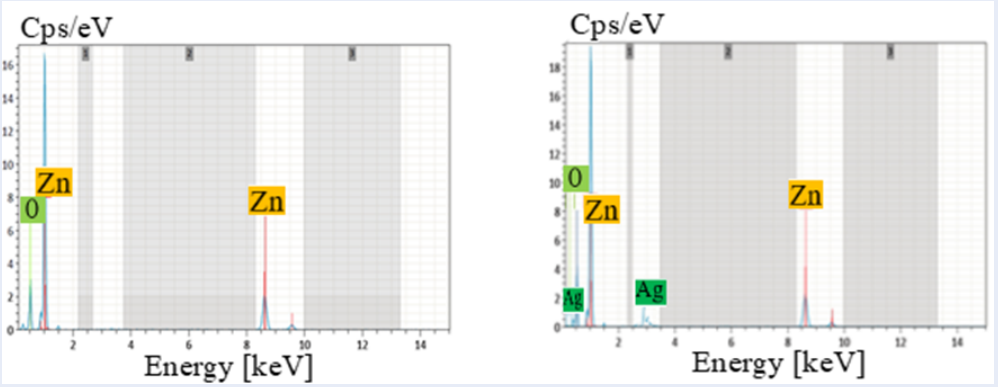

Figure 3: The EDX spectra of ZnO and 10%Ag-ZnO samples

tra results showed that it was consistent with the theory of optical properties of ZnO and Ag/ZnO samples. Based on these reflection spectra, the Kubelka–Munk (K-M) method[4,12] was applied to determine the optical gap energy (Eg) of the materials. Figure 5 was a plot of the K-M function d(lnF(R)´h)/dE with energy h of ZnO and 10%Ag-ZnO samples. The energy Eg of samples was determined by extrapolation from to the K-M differential equation (in which, F(R) is a function of the diffuse reflectance coefficient; hν is the photon energy variable; Eg is the band gap energy).

$$d[\ln(F(R)\times h\nu)]/d(h\nu) = n/(h\nu\text{-}Eg) \quad (3)$$

The extrapolation results showed that the Eg values of ZnO and 10%Ag-ZnO samples were 3.28 eV and 3.16 eV, respectively. Thus, it could be seen that the Ag doped ZnO sample had reduced the Eg value more than that of the undoped ZnO sample. This gave the possibility to speculate that the 10%Ag-ZnO nanopowder sample would be able to respond to stimulation of the light in visible region.

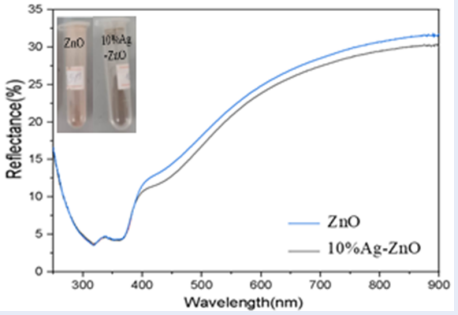

Figure 4: Reflection spectra of ZnO and 10%Ag-ZnO samples

Evaluation of photocatalytic properties

Figure 6 was a plot investigating the decomposition efficiency of methylene blue (MB) dye solution on ZnO and 10%Ag-ZnO nanopowder photocatalysts. Figure 6 indicated that the 10%Ag-ZnO nano powder sample achieved a high MB dye decomposition efficiency of 97.8% after 40 min under visible light irradi-

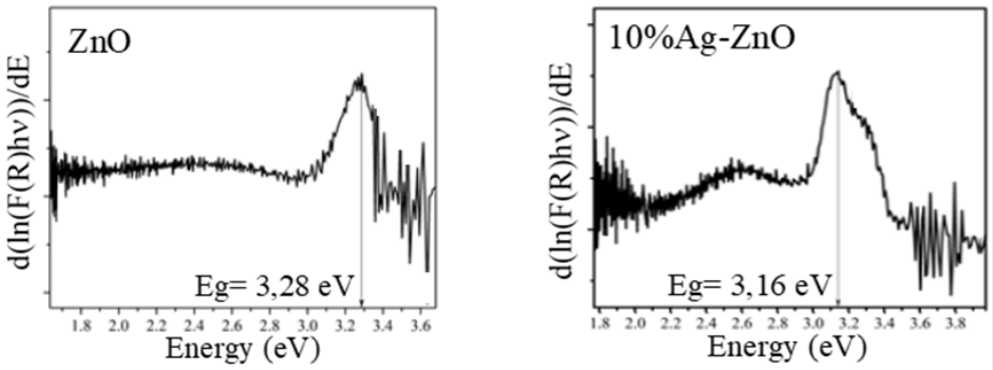

Figure 5: The function plot d(lnF(R)×h)/dE with energy hv of ZnO and 10%Ag-ZnO samples

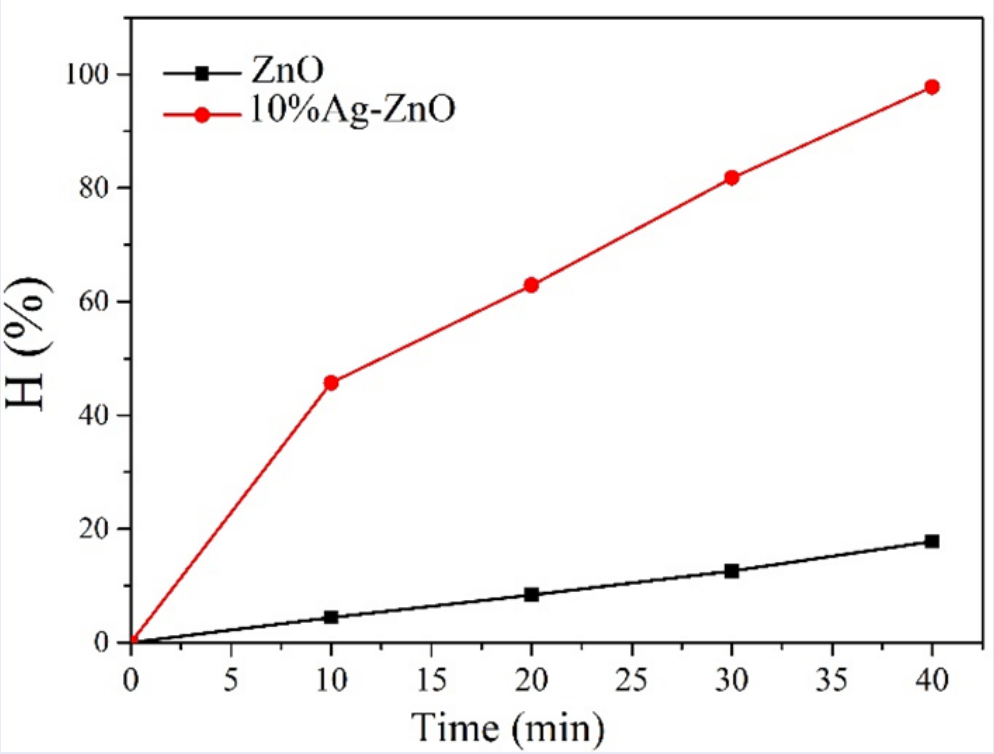

Figure 6: The efficiency for decomposition of MB dye with visible light radiation time of ZnO and 10%Ag-ZnO samples

ation. Meanwhile, the ZnO sample could hardly decompose MB, the MB decomposition efficiency was very low at 17.8% after 40 min of visible light exposure. Thus, the ZnO sample doped with Ag+ ions had improved the application efficiency of the material, achieving the ability to photocatalyze decomposition of MB dye in the visible light region, with high effective for decomposition of MB dye solution. This is desirable for research aimed at improving the properties of ZnO materials by modification [7,10–12,21–24].

CONCLUSION

Ag-doped ZnO (10%Ag-ZnO) and ZnO nanopowder materials were successfully fabricated using red peony leaf extract solution by the simple hydrothermal method. The samples were fabricated in the form of pure single-phase hexagonal wurtzite nanostructures. The average crystal size of the 10%Ag-ZnO

sample (22.6 nm) was smaller than that of the undoped ZnO sample (32.27 nm). The ZnO sample had a spherical particle shape. The 10%Ag-ZnO sample had a smoother and more porous surface than ZnO sample. The Ag doped ZnO sample had improved some properties compared to ZnO such as: reducing the average crystal grain size, increasing the porosity of the material surface; reduces the optical gap energy ($Eg_{(10\%Ag-ZnO)} \approx$ 3.16 eV, $Eg_{(ZnO)} \approx$3.28 eV); achieved effective for photocatalytic decomposition of MB solution in the visible light excitation region (97.8% after 40 minute under visible light irradiation), while the undoped ZnO sample had almost no effect for decompose MB dye in the visible light irradiation region (decomposition efficiency was very low reaching 17.8% after 40 minute of visible light exposure).

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CONFLICT OF INTEREST

The authors would like to confirm that there is no conflict of interest in publishing the article.

AUTHORS' CONTRIBUTION

Luu Thi Lan Anh: Investigation, original draft; Nguyen Thi Tuyet Mai: Investigation, original draft, Formal analysis, Supervision; Ta Ngoc Dung: Methodology, Formal analysis, Supervision; Huynh Dang Chinh: Methodology, Formal analysis, Supervision.

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Tạp chí Phát triển Khoa học và Công nghệ – Engineering and Technology 2024, 6(SI3):80-87

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Bài nghiên cứu

Chế tạo và khảo sát đặc tính cấu trúc và tính chất quang xúc tác của vật liệu cấu trúc nano ZnO pha tạp Ag

Lưu Thị Lan Anh[1], Nguyễn Thị Tuyết Mai[2,*], Tạ Ngọc Dũng[2], Huỳnh Đăng Chính[2]

TÓM TẮT

Bột nano ZnO và ZnO pha tạp 10% Ag đã được nghiên cứu chế tạo bằng sử dụng dung dịch chiết lá cây mẫu đơn đỏ. Các vật liệu này được điều chế bằng phương pháp hóa ướt đơn giản và nung ở nhiệt độ thấp khoảng 400^{o}C trong 2 giờ. Ion Ag^+ được pha tạp vào vật liệu ZnO so với ion Zn^{2+} với tỷ lệ mol là 1:10. Các phương pháp nghiên cứu đặc tính của bột nano đã được sử dụng như XRD, SEM/EDX và phổ phản xạ. Phương pháp Kubelka–Munk dựa trên phổ phản xạ của mẫu đã được khai thác để xác định năng lượng khe trống quang của các mẫu bột nano ZnO và ZnO pha tạp 10% Ag chế tạo. Kết qủa khảo sát các đặc tính của vật liệu cho thấy, vật liệu bột nano ZnO pha tạp Ag có kích thước tinh thể trung bình nhỏ hơn so với ZnO không pha tạp (các giá trị này lần lượt là 22,6 và 32,27 nm). Với việc pha tạp vào vật liệu bán dẫn oxit kim loại chuyển tiếp bằng các ion có bán kính lớn hơn nhiều bán kính của ion chủ trong mạng tinh thể oxit kim loại chuyển tiếp (bán kính của ion Ag^+ (1,22 Å) lớn hơn bán kính của ion Zn^{2+} (0,74 Å)), thì rất có thể nguyên tố Ag sẽ được pha tạp vào mạng tinh thể ZnO ở dạng xen kẽ. Năng lượng khe trống quang của bột nano ZnO pha tạp Ag cũng được giảm hơn so với ZnO, đạt giá trị Eg≈ 3,16 eV (mẫu ZnO đạt giá trị Eg≈ 3,28 eV). Mẫu ZnO pha tạp Ag đã làm nâng cao được phạm vi ứng dụng quang của vật liệu, đó là đã đạt được hiệu quả quang xúc tác cao (97,8%) làm phân hủy chất màu metylen xanh (MB) sau 40 phút chiếu ánh sáng nhìn thấy. Bột nano 10%Ag-ZnO được điều chế từ dịch chiết xuất của lá mẫu đơn đỏ -đây là một trong những phương pháp chế tạo ``xanh''. Vật liệu chế tạo này đã đạt được những ưu điểm vượt trội so với vật liệu ZnO không pha tạp như giảm kích thước tinh thể trung bình, giảm năng lượng vùng cấm quang và đạt được phản ứng phân hủy xúc tác quang dung dịch chất màu MB dưới ánh sáng nhìn thấy với hiệu suất cao.

Từ khoá: Wurtzite ZnO, ZnO pha tạp Ag, Kubelka–Munk, khe trống quang học, ánh sáng nhìn thấy

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