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Study to Evaluate Photocatalytic Decomposition of Several Organic Compounds and Self-Cleaning Ability of Fabrics Containing ZnO

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Abstract

ZnO's ability to decompose organic substances is receiving much attention in multiple applications. ZnO has a broad spectrum of light absorption natural or synthetic, for a wide range of wavelengths, and is an important material with excellent properties, environmentally friendly, nontoxic, inexpensive, and high redox potential features with simple manufacturing methods. In this study, zinc oxide nanoparticles were synthesized by precipitation method with the participation of precursors $(CH_3COO)_2Zn.2H_2O$ and KOH. The surface morphology, chemical properties, and chemical composition of the ZnO catalyst were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy, and Energy-Dispersive X-ray Spectroscopy (SEM-EDS). The photocatalytic activity of ZnO was evaluated with the color change of methylene blue (MB), methylene orange (MO), and diluted coffee solutions. The results show that ZnO has a high photocatalytic activity with a decomposition ability of over 90% for MO and MB pigments, and over 40% for a coffee solution when irradiated under ultraviolet light. This study's cotton fabric impregnated with ZnO showed good self-cleaning effects against organic pigments, which are MB, MO, and coffee. The results were demonstrated by the color changes over a 19-h period of time.

Keywords: ZnO, precipitation method, photocatalysis, compound decomposition.

1. Introduction

ZnO is a n-type semiconductor with various fascinating characteristics and has been studied for a long time [1]. Recently, ZnO nanoparticles as nano semiconductors have gained much interest in academia because of photocatalytic degradation and antibacterial properties. ZnO is an essential material with significant photocatalytic activities and interesting chemical features, which are very effective in varied practical applications, such as optical equipment, battery cells, catalysis, antibiotics, medicine, and so on [2,3]. ZnO has a proper band gap of 3.37 eV and binding energies of large excitons (~60 meV), which can absorb a large fraction of the UV spectrum and exhibit a high photocatalytic performance [4]. For this reason, nano ZnO allows exciton absorption and recombination even at room temperature.

ZnO particles have been used in self-cleaning applications on various substrates such as paint, textile, plastics, and steel surfaces. ZnO is usually prepared in form of powder with white color. The precursors for the synthesis are the mineral zincite, and zinc salt synthetically or present in nature. ZnO after being immobilized onto the textile substrate shows special features such as thermal stability, absorption of a wide range of light radiation, effective photocatalyst for self-cleaning, electrical conductivity, hydrophobicity, hydrophilicity, antimicrobial, moisture management, thermal insulation, and UV-protective agents. Interestingly, the flame retardancy and thermal stability of the fabrics can be improved by adding ZnO particles.

Among various applications of ZnO, the ability to apply photocatalytically in the decomposition of organic substances that cause environmental pollution is receiving much attention. ZnO has a wide spectrum of light absorption natural or synthetic, for a wide range of wavelengths and is an important material with excellent properties, environmentally friendly, nontoxicity, inexpensive, and high redox potential features with simple manufacturing methods [5]. ZnO nanoparticles exhibit high electron mobility than other semiconductors such as TiO_2, which accelerates electron transfer and contributes to great quantum efficiency. Therefore, the photocatalytic activity of ZnO in pollutant decomposition is higher than TiO_2. However, the recombination of photogenerated electrons and holes lowers the photocatalytic performance [6].

The color degradation efficiency of ZnO based on the principle of photocatalysis was investigated and evaluated through the efficiency of color decomposition in water for MB, MO - typical azo dyes, and the application of coffee stains on coffee grounds after immobilization on fabric with 100% cotton. The

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ZnO nanoparticle surface is reportedly hydrophilic with a water contact angle of less than 90°. Usually, hydrophobic surfaces can be used for antifouling, self-cleaning, anti-icing, and liquid transport, thus, various published articles have tried different methods to turn ZnO surfaces from hydrophilicity to hydrophobicity. Doping or co-doping with other elements, UV illumination, and heating seems to be an effective method to convert ZnO surface wettability. The self-cleaning characteristics of ZnO can be derived from the photocatalytic properties. With the presence of ZnO on the surface of materials, the photodegrading reactions can happen against various organic compounds, colored stains, or colorless dirt when encountering them. The ZnO self-cleaning effect can be controlled via the geometrical structure of ZnO such as nanowires, nanorods, and nanoarrays. The approaches to immobilize nano ZnO on the surfaces of materials can be sputtering, spraying, reactive evaporation, electrodeposition, and so on. There is a high need for hydrophobic nano ZnO for self-cleaning applications in the industry [7,8].

100% cotton fabric is a natural, soft, user-friendly fabric with high biodegradability and water absorption so it is widely used in the field of textile and garment to make clothes such as shirts, sweaters, jackets, and trousers [9]. However, due to its highly absorbent and moisture-retaining properties, up to 65% by weight, 100% cotton fabric makes it difficult for users to deal with stains on the fabric due to the generation of micro-organisms such as bacteria, molds, and fungi, which are harmful to human health. The process of putting nano ZnO on the surface of cotton fabric helps to add self-cleaning ability to the fabric. After the immobilization process, the newly developed material has the ability to decompose pigments based on the principle of photocatalysis [10]. The self-cleaning effects of ZnO particles have been mostly studied on cotton and polyester surface. Coffee, tea, MB, MO, and other dyes play the role of stains. The light irradiated can be solar or UV light for various intervals and under varied humidity conditions. The cleaning efficiency could be evaluated based on color-changing observance or color-difference measurement techniques. One very important factor is the durability of the photocatalysts which is evaluated by consecutively applying the same dyes or stains to the exact position. In addition, zinc oxide nanoparticles exhibit antibacterial activity against a wide range of pathogens through different mechanisms [11].

To fabricate ZnO-functionalized textile substrates, various procedures can be utilized such as dip-coating, sol-gel, padding, and electro-decomposition. ZnO at micro or nano size can be deposited on chosen textile materials. Additionally, ZnO can be synthesized in situ with precipitation, ultrasonic, mechanochemical, sol-gel, micro-wave supporting sol-gel, hydrothermal, and solvothermal. In this paper, ZnO's ability to degrade MB, MO, and coffee in water and the cotton fabric was investigated. The results show that the decomposition efficiency of some organic compounds is over 90% for azo dye samples, which are MB, MO, and over 40% for colorants present in coffee.

2. Experiment

2.1. Materials

Zinc acetate ($(CH_3COO)_2Zn$) and potassium hydroxide (KOH) were purchased from Shanghai Yuanye Biotechnology Technology Co., Ltd. All the chemicals used in the experiments were of analytical grade, and deionized water was used.

2.2. Methods

The ZnO oxide sample was obtained by the solution precipitation method. The reaction takes place according to the equation:

$$(CH_3COO)_2Zn + 2KOH \rightarrow ZnO + H_2O + 2CH_3COOK$$

Specifically, the first step is to dissolve 10.95 g $(CH_3COO)_2Zn.2H_2O$ in 250 ml of distilled water to obtain a solution with a molar concentration of 0.2M. Similarly, dissolving 5.6 g of KOH in 250 ml of distilled water to obtain a KOH solution with a concentration of 0.4 M. Afterwards, 0.4 M KOH solution was slowly added to $(CH_3COO)_2Zn$ solution under strong magnetic stirring. After adding all the KOH to $(CH_3COO)_2Zn$, the mixed solution was stirred for 1 h and allowed to settle for 2 h. The centrifugation was carried out to separate the water to collect the solids. The ZnO nanoparticles were obtained after being dried at 100 °C for 24 hours and were further crushed and calcined at 500 °C for 3 hours, the heating rate was 1 °C/min.

The photocatalytic activity of ZnO was evaluated through the efficiency of photocatalytic degradation for azo MB dye (MB concentration in distilled water is 10 ppm), azo MO dye (MO concentration in water is 10 ppm), and coffee solution (0.2% by mass). Before conducting UV irradiation, the dye solutions containing ZnO were magnetically stirred in a dark chamber to adsorb MB (for 75 min), MO (for 75 min), and coffee (for 120 min). The adsorption process was carried out in three independent 100 ml glass flask systems containing 20 ml of MB solution, 20 ml of MO solution and 20 ml of coffee with the corresponding amount of ZnO 0.005 g each.

The effectiveness of stain removal or self-cleaning effect on fabrics was evaluated by the ability to discolor stains on 100% cotton fabrics (Vietnam) coated with ZnO after being irradiated with ultraviolet light for a defined period of time. The cotton fabric used in the study is a treated fabric, single weave, with the main specifications as follows: weight 201.6 g/m^2, breathability 274 ($l/m^2/s$).

For each colorant sample, it is necessary to prepare a powder ZnO catalyst that is ultrasonically vibrated in ethanol (with a concentration of 0.005 grams in 10 ml of ethanol) for 20 minutes. The process is to separate ZnO particles, which are clustered together. Apply the ZnO solution to a cotton cloth measuring 3 x 3 cm (weight 0.223 ± 0.001 grams) by impregnation and let it dry completely. The amount of colorant used for each sample includes 2 ml of azo MB dye solution, 2 ml of azo MO dye solution, and 2 ml of coffee.

2.3. Analytical Methods

ZnO particles incorporated on cotton fabric sample were analyzed by SEM-EDS on Hitachi TM4000 Plus instrument at Hanoi University of Science and Technology at an accelerating voltage of 15 kV. The surface of the nanofibers was coated with platinum (Pt) in a sputtering chamber for 120 s at 30 mA.

The UV radiation source used in the experiment was emitted from a 60 W UV lamp (Sterilizer lamp, wavelength 254 nm, China). In the process of ultraviolet irradiation, after certain periods of time, 5 ml of the experimental solution would be filtered using a 0.45 nm filter to analyze the amount of MB, MO, and colorants in the coffee remaining in the coffee using UV-Vis spectrophotometer (Agilent 8453, USA) at RoHan Research Laboratory, School of Chemical Engineering, Hanoi University of Science and Technology.

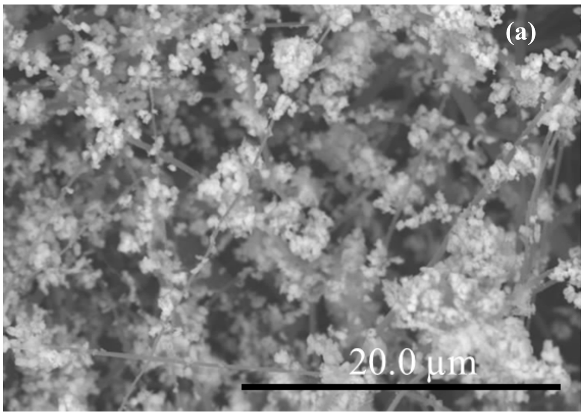
The amorphous, crystalline, and semi-crystalline phases of ZnO sample were investigated by a Panalytical XPERT PRO diffractometer (Almelo, The Netherlands) with CuKα radiation, a scanning step of 0.05° and a collecting time of 100 sec per step, operating at 30 kV and 500 mA, at the 2θ ranging from 20° to 80°.

3. Results and Disscution

3.1. Research Results on the Characteristics of Catalysts

The SEM images of the sample are depicted in Fig. 1(a) and 1(b). The results show that the ZnO particles are uniform. The SEM images of the sample are shown in Fig. 1 (a) and (b). The results demonstrate that the ZnO catalyst has a circular shape with the average diameter is less than 100 nm.

XRD analysis is used to provide insight into the crystal structure and crystal size of the particles. The powder preparation was essential to obtain ZnO with high purity. The powder after the collection was washed with ethanol, centrifuged, and dried for one day to remove all the by-products and impurities. The XRD pattern of ZnO is shown in Fig. 1(c). From the analysis results, it can be seen that XRD peaked at 2θ = 31.745°, 34.385°, 36.185°, 47.465°, 56.555°, 62.825°, 66.485°, 67.925°, and 69.065° attributed to (100), (002)), (101), (102), (110), (103), (200), (112) and (201) crystal planes and hexagonal crystal geometries, respectively, according to JCPSD No. 01-007-2551. These peaks are characteristic peaks of ZnO polycrystalline hexagonal Wurtzite structure. The XRD result also demonstrates the complete conversion of zinc acetate to zinc oxide.



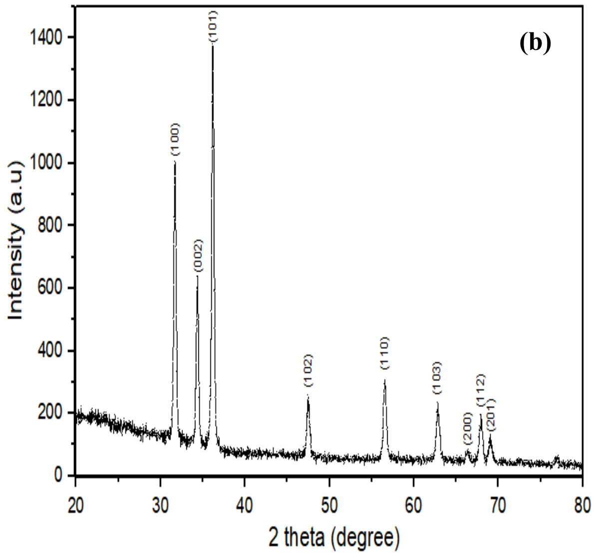


Fig. 1. (a) SEM image of ZnO on cotton fabric with 5000 magnification, (b) Result of XRD diffraction measurement of ZnO

The average particle size of the nanoparticles was determined using the Scherrer equation:

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

where D is the crystal size of the particle, K represents the Scherrer constant equal to 0.9, λ is the wavelength of light used for diffraction (λ = 1.54 Å), β is the FWHM (whole total width at half maximum) of the diffraction peak and θ is the diffraction angle at the diffraction surface position [12]. The average size of ZnO nanoparticles calculated from the XRD sample according to Scherrer formula is 45.68 nm.

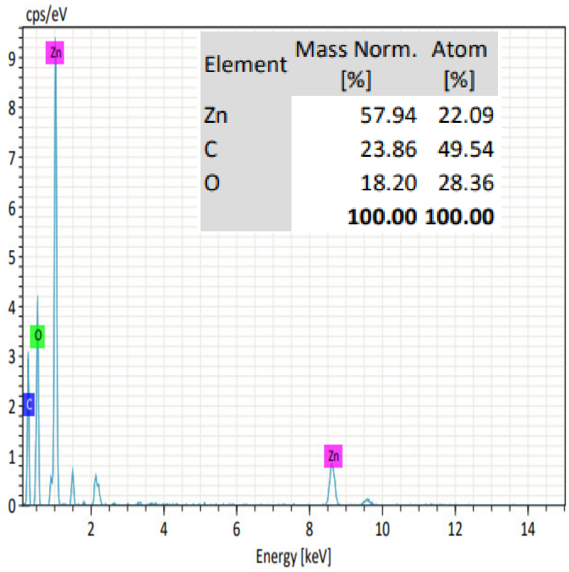


Fig. 2. Results of EDX analysis of cotton fabric coated with ZnO

The ZnO-coated fabric was subjected to EDX analysis, the results are shown in Fig. 2. From the results, ZnO was demonstrated to be successfully applied onto the fabric. The characteristic results verified that ZnO containing cotton fabric is composed majorly of carbon, oxygen, and zinc. The Zn element in some places on the surface of the composite fabric material accounts for 57.94 mass percent over all present elements. For atom percentage, Zn and O are relatively equal, and only about 6% difference might come from the oxygen atoms existing in cotton fabric. Carbon element comes from the fabric with the main component being cellulose.

3.2. The Adsorption Capacity of MB, MO and Colorants in Coffee of ZnO

In this experiment, the amount of ZnO used was 0.005 g for every 20 ml of 10 ppm MB, 10 ppm MO, and 0.2% coffee (by mass) in three different reaction bottles. The results of adsorption efficiency are presented in Fig. 3 and Fig. 4.

According to the results from Fig. 3, ZnO effectively adsorbed the color in the first 15 min (approximately 39.1%) and slowly over the next 60 min intervals for MB, whereas, for MO pigment, the slope of discoloration graph line continues to rise up to about 17% before reaching the plateau. The adsorption toward MO was much lower than MB and reached the highest level at 18.5%. At the end of the adsorption process, which lasted about 75 min, the MB conversion over time was about 47.5%.

The MB is positively charged while MO is negatively charged moiety thus the interaction of ZnO toward the positively charged ions is better. The chemical structure of ZnO particles can create a negatively charged surface according to the lone pair electron of Zn-O bonding, which induces the columbic interactions with MB. Moreover, the hydroxyl groups on ZnO can compete with sulphonic groups of MO during the adsorption process. Despite the repulsive force between MO ions and ZnO particles, the MO adsorption can be elucidated by the mesoporous structure of the particles.

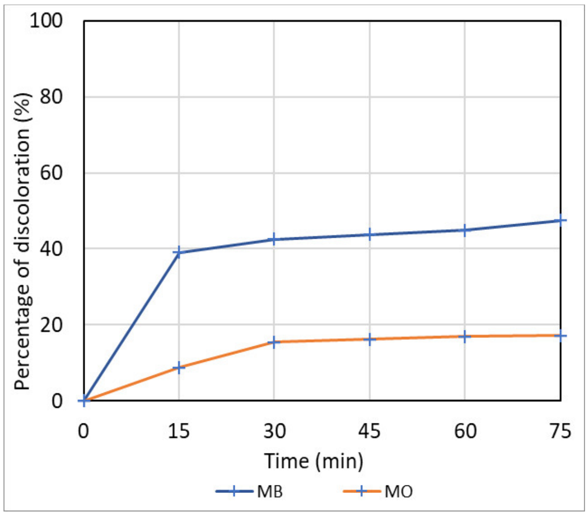


Fig. 3. ZnO adsorption capacity of MB and MO in the dark chamber over the period of 75 min

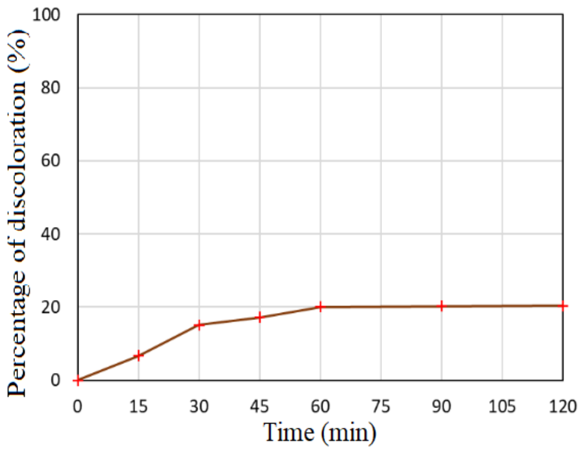


Fig. 4. ZnO adsorption capacity of colorants in coffee in the dark over time

Synthetic dyestuffs released by textile industries pose a critical threat to the aquatic and land environment. The dye contamination is quite visible due to distinguished color changes in water. When digesting into human bodies, dye molecules can cause acute or chronic consequences on organs. The removal of dyestuffs either by adsorption or degradation can be the treatment for polluted water.

The adsorption process can be explained as the involvement of multiple steps: (1) dissolving coffee or dye molecules into the solution, (2) the external diffusion of the molecular, (3) internal diffusion of the molecules, and (4) desorption and adsorption reach equilibrium. The adsorption process is necessary for an effective photocatalytic reaction.

The adsorption capacity of ZnO for coffee colorants is shown in Fig. 4. In the first 30 min of the adsorption process, the amount of coffee adsorption reached about 15.5% and ended after 75 min with discoloration in the range of 204%.

ZnO adsorption toward coffee reached equilibrium after a period of 60 min with a discoloration efficiency of 20.4%. The results suggest that since a coffee effluent consists of a large number of unidentified macromolecules, the efficiency achieved was not as high as MB and MO dye. The coffee effluent was decomposed via multiple steps and created complicated intermediate products. Moreover, coffee effluent contains many suspended organic particles that cannot be decomposed by hydroxyl radicals.

3.3. The Ability to Decompose MB, MO and Colorants in Coffee under UV Light

The MB, MO and coffee solutions, after undergoing adsorption in the dark chamber, continued to evaluate the color concentration conversion with ZnO under the effect of UV lamp under continuous stirring conditions. The results are shown in Fig. 5, Fig. 6, and Fig. 7, respectively.

In Fig. 5 and Fig. 6, the MB and MO dye solutions lost color rapidly in the first 30 min (MB reached 37.8%, MO reached 50,6%). Both dye degradation rates slowly reduced in the following period of time. After 17 hours, the color conversion of MB ended with 97.4% results, and that of MO was 91.7%.

The mechanisms of photocatalytic actions are as follows: when photons reach the surface of semiconductors, zinc oxide's electrons are excited in the valence band and move to the conduction band. The excited electrons will react with oxygen molecules on the surface of the semiconductor to generate radical anions of super oxide. These super oxide radicals via several mediate redox agents present strong organic pollutants oxidization and thus can decompose varied organic compounds under light irradiation.

The kinetics curves for the discoloration of MO using photocatalyst ZnO assure that the photodegradation nature against MO is better than MB at the beginning with the concentration reduction of MO being 67.8% at the first 60 min whereas it is 47.7% for MB. It illustrates the optical decomposition nature of MO using ZnO. However, after 14 h, the concentration reduction of MB is slightly higher, which can be explained by that the charged interaction between ZnO and MB dye molecules is better than MO dye molecules at low concentrations. However, the natural decomposition of MO happens faster at higher concentrations.

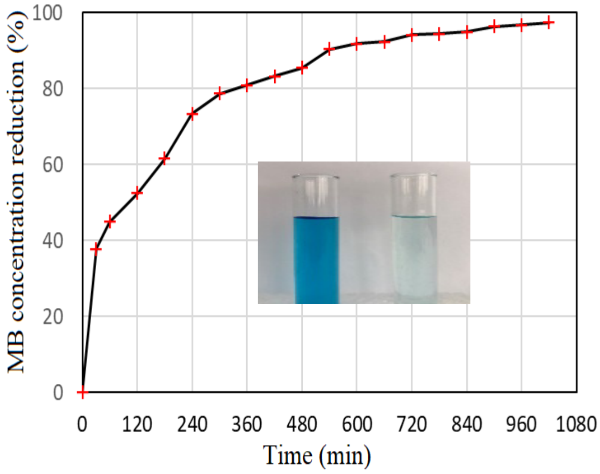


Fig. 5. MB degradability of ZnO with UV lamp

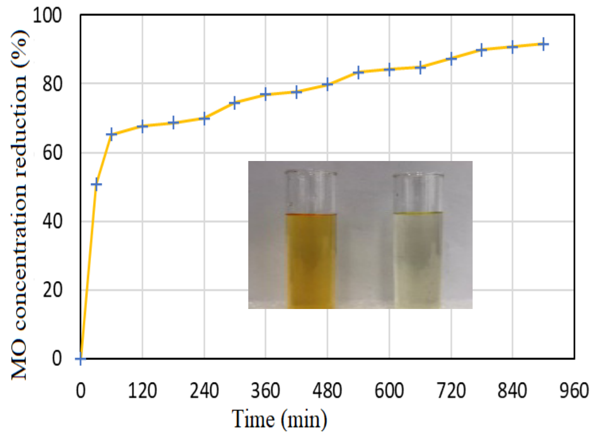


Fig. 6. ZnO MO decomposition with UV lamp

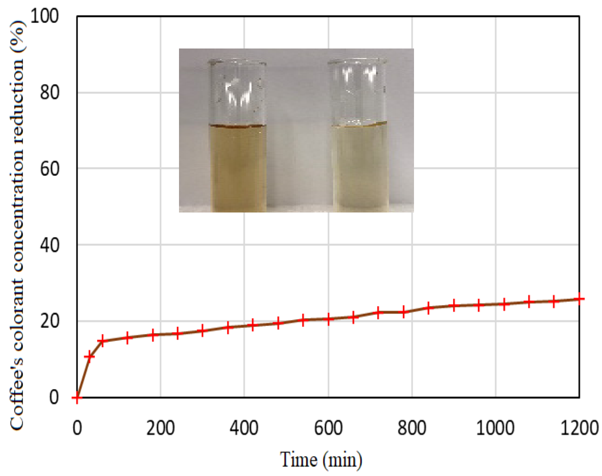


Fig. 7. ZnO's ability to degrade coffee colorants under UV radiation

ZnO nanoparticles showed a good adsorption capacity and rapid absorption rate for MB mainly due to the ionic interaction between ZnO and sulfonic groups. The ionic bond is a strong bonding and does not show directivity. The ZnO particles showed a good adsorption capacity for both MB and MO maybe due to its high surface area as well as the high concentration of effective adsorption sites. The removal of MB and MO by ZnO particle was mainly due to the decomposition efficiency of the ZnO under UV irradiation rather than the adsorption process.

The conducting procedure to degrade colorants in coffee with ZnO is similar to that of MB and MO. During the first 30 minutes, the process is fast at about 10,6% and also slows down later. At 60 min, the degradation reached 17.2% and after that time, the increasing rate of concentration reduction happened much slower. After about 20 hours, the decoloration process stopped at 25.8%. Coffee release a wide variety of many different macromolecules including polyphenols, and melanoidins making the dark brown color. Other than that, there are many non-biodegradable compounds leading to slow degradation.

The coffee degradation process can be presented in a number of reaction steps. First, the electron-hole pairs are formed due to the UV light excitation on the photocatalyst. Then the migration of these elements to the ZnO surface and the redox reactions occur, afterward. The generated superoxide radical anions and hydroxyl radicals are the main oxidizing species that directly participate in consecutive chemical reactions. However, the photodegradation cannot happen without the preceded adsorption step. Thus, the dark brown coffee effluent first is adsorbed onto the ZnO surface and, subsequently, the mineralization and decolorization of coffee components happen.

At the end of the process of evaluating the adsorption and degrading ability of ZnO against various colorants in different solutions, the converted colorant concentrations recorded for MB, MO, and coffee were 98.5%, 93.0%, and 40.8%, respectively. The decomposition of MB was highest due to the good adsorption capacity of ZnO. The decoloration of coffee solution was lowest according to the complicated coffee chemical composition.

Zinc oxide particles can be improved in photocatalytic efficacy by the doping method. Pure ZnO has the drawback of fast recombination of photogenerated holes and electrons. The remedy is to inhibit the e-h recombination by creating a trap by making new energy-favorable levels via doping methods.

3.4. The Self-Cleaning Ability of ZnO to Degrade Dyes and Coffee on Fabrics under UV Light Conditions

The photocatalytic activities of semiconductors such as ZnO against organic pollutants in contact with the surface can prevent the organic compounds and dirt from building up. Zinc oxide semiconductor photocatalyst has a photocatalytic mechanism similar to that of TiO2. ZnO particles also possess many advantages, such as porosity, cheap price with high reactivity, great absorption efficiency of varied light sources, and human safety. The photoactivity of ZnO coated cotton fabric was investigated by exposing the fabric samples containing MB, MO, and coffee to UV light radiation. For this purpose, the same amounts of liquid were applied to each sample.

The photocatalytic decomposition of ZnO on fabric is shown in Fig. 8 and Fig. 9. MB decomposition on ZnO-coated fabric is studied. After 19 h of UV irradiation, the color of the untreated cotton sample remained unchanged during the test, while the color of the ZnO-coated fabric faded and changed to the original color of the fabric, Fig. 8(a). For MO, Fig. 8(b), after 17 h of UV irradiation, the color of the fabric coated with ZnO on the surface gradually faded to the natural fabric color.

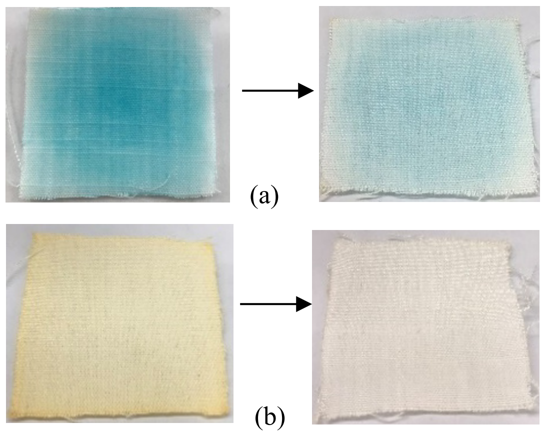


Fig. 8. Decomposition results of (a) MB and (b) MO on ZnO coated fabric

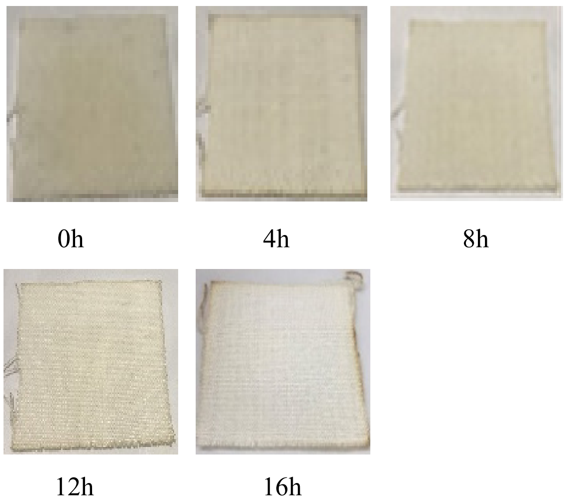


Fig. 9. Decomposition of colorants in coffee on ZnO coated fabric after different time frames

Fig. 9 records the decoloration process of coffee on ZnO-coated fabric after every 4 hours of UV irradiation. The photocatalytic degradation of colorants in coffee results in observable although the stains are not completely removed, the reason is that in coffee there are many complex organic compounds, the decomposition of these substances goes on for a long and difficult time. These research results show that the fabric after coating with ZnO nanoparticles has the ability to decompose stains based on photocatalytic effect and has self-cleaning ability.

3.5. Surface Wettability

The hydrophilicity of the cotton fabric was assessed by the water contact angle, using a contact angle meter, with reproduction on ten specimens of each sample, the results were presented on average values with standard deviations. Cotton fabric samples were found to be highly hydrophilic with an average water contact angle of 149°, Fig. 10. After coating with ZnO nanoparticles, the surface of cotton fabric reduced hydrophilic performance remarkably. In published reports, ZnO was considered a hydrophobic agent; thus, its impregnation to the cotton fabric impairs the hydrophilic feature. Additionally, with the capability to be used as a surface functional agent, ZnO coating provides a controllable hydrophobic property for the material. The observed hydrophilicity of ZnO integrated cotton fabric was much lower than the untreated one. It can be concluded that cotton fabric has changed from hydrophilic to hydrophobic with the presence of ZnO nanoparticles.

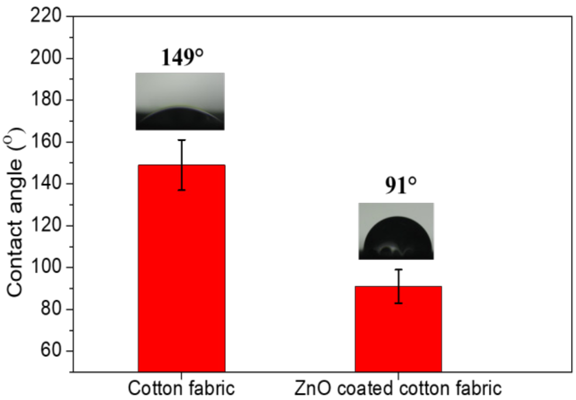


Fig. 10. Contact angle measurement of cotton fabric and ZnO coated cotton fabric

4. Conclusion

In the paper, a simple, low-cost and highly efficient precipitation method was used to successfully synthesize ZnO with photocatalytic activity. The successful synthesis of ZnO was confirmed by SEM-EDS and XRD. The photocatalytic activity was evaluated by UV-Vis spectrophotometer and showed the effectiveness for self-cleaning application for stains on fabrics, through the results of the evaluation of the photodegradability of the pigments. ZnO, the converted pigment concentrations recorded for MB, MO and 0.2% coffee were 98.5%, 93.0% and 40.8%, respectively. At the same time, the successful application of decontamination on cotton fabric from this research offers the potential to create high-economic-value textile products.

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