

ENANTIOSELECTIVE APPLICATION OF TiO₂ AND ZnO ENCAPSULATED HOLLOW SILICA IN THE PHOTOCATALYTIC DEGRADATION OF DIMETHYLARSINIC ACID

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ABSTRACT

Titanium dioxide (TiO₂) and zinc oxide (ZnO) have attracted wide attention due to their photocatalytic properties despite other usages such as pigments in foodstuffs, paint, and cosmetics. They have a high demand due to their broad functionality, long-term stability, and non-toxicity. Although TiO₂ and ZnO are good photocatalysts for the degradation of various environmental pollutants, their small adsorption coefficient limits this ability. This work deals with encapsulating TiO₂ and ZnO with hollow silica (HS); TiO₂-HS and ZnO-HS to increase the surface area adsorption and to control the photocatalytic activity of both catalysts. Sol-gel method through a wet precipitation process following that of the Stober Method was applied to prepare the desired TiO₂-HS and ZnO-HS which gave distinct outcomes in the characterization by Brunauer–Emmett–Teller (BET) where TiO₂-HS has a reduced surface area compared to the naked TiO₂ while ZnO-HS has an increased surface area compared to the naked ZnO. This was proven by further characterizations using FTIR, XRD, and EDX. The physical appearance and morphology of TiO₂-HS and ZnO-HS were shown by the FESEM and TEM images. The catalytic activity of TiO₂-HS and ZnO-HS was tested in the degradation of dimethylarsinic acid. Results show a lower percentage of degradation by TiO₂-HS compared to TiO₂ while ZnO-HS gave a higher degradation percentage as compared to ZnO. Thus, the silica doping has been proven to reduce the photocatalytic properties of TiO₂ but enhance the photocatalytic activity of ZnO

Keywords: Titanium dioxide (TiO₂), zinc oxide (ZnO), hollow silica, photodegradation, dimethylarsinic acid (DMA)

RASĀYAN J. Chem., Vol. 16, No.3, 2023

INTRODUCTION

Nowadays, polluted water caused by hazardous materials has become a serious problem. Due to its widespread existence, dimethylarsinic acid (DMA) pollution recently received a lot of attention. DMA^{1,2} is known to have much higher toxicity than inorganic arsenic regarding cytotoxicity effects.

It was introduced into the environment through industrial and agricultural activities. Recent studies on *Ulmus laevis* and *Acer playamoides* found dead because they were exposed to DMA, Arsenic (II), and Arsenic(V).^{3,4} Therefore, the presence of DMA especially in groundwater is a threat to organisms as it is highly toxic and carcinogenic. Photocatalysis is the application of a catalyst in a reaction in which light is consumed to trigger a substance that alters the rate of chemical water treatment. It is well known that TiO₂

and ZnO are promising photocatalysts that are able to degrade various kinds of organic pollutants in the presence of light due to their similar band gap. TiO₂, particularly in the anatase form is a photocatalyst under ultraviolet light. It has potential uses in energy production as a photocatalyst that can carry out hydrolysis i.e., breakdown water into hydrogen and oxygen⁵. Although TiO₂ and ZnO are good photocatalysts⁶ for the degradation of various environmental pollutants, their small adsorption coefficient limits their capacity.^{7,8} Hence, it is important to enhance their photocatalytic activity as demonstrated by previous studies work deals with the encapsulating of TiO₂ and ZnO with hollow silica (TiO₂-HS/ZnO-HS) to increase the surface area adsorption and to control the photocatalytic activity of both catalysts.⁹ The method used to synthesize TiO₂-HS/ZnO-HS followed that of the Stober method.¹⁰ The composite¹¹ was prepared by the precipitation of TiO₂/ZnO with ammonia, tetraethyl orthosilicate (TEOS), and ethanol. Silica was chosen as a support because it has a uniform aperture, high surface area, and does not agglomerate on folding¹². However, several types of popular support materials such as alumina, zeolite, and activated carbon also can be used to support the catalysts. The purpose of this research focuses on the method of synthesizing and characterizing the TiO₂ and ZnO encapsulated hollow silica (TiO₂-HS/ZnO-HS) and applying them in the degradation of DMA in aqueous solution. The TiO₂ used in this work is in the anatase form which is commercially available and the same as ZnO. The synthesized TiO₂-HS/ZnO-HS were characterized by FTIR, SEM-EDX, BET, XRD, FESEM, and TEM. Results on the photocatalytic activity of the catalysts were examined using UV-Vis Spectrometry in order to calculate the percentage degradations of DMA catalyzed by TiO₂, TiO₂-HS, ZnO, and ZnO-HS as well as to analyze the degradation potential for all catalysts.

EXPERIMENTAL

Material and Methods

All chemicals and commercial TiO₂ and ZnO powders used were in analytical grade or the highest synthetic purity from Fluka, Merck, and Sigma-Aldrich.

Synthesis of TiO₂-HS and ZnO-HS

TiO₂-HS was prepared by encapsulating the naked TiO₂ with silica. Firstly, commercially ready TiO₂ anatase powder (0.5 g) was weighed in a Teflon bottle. Then, 20 mL of ethanol, 9 mL of distilled water, and 0.5 mL of ammonia solution (25%) were added to the bottle, respectively. The mixture was stirred slowly and then 0.5 mL of tetraethyl orthosilicate was added into it. The mixture was left for an hour to precipitation. The mixture was then filtered under gravity and washed with (v/v) 50% ethanol and (v/v) 50% deionized distilled water sufficiently. The filtered product was calcined at 500 °C for 4 hours and the precipitate was collected and weighed. The same method was applied for the preparation of ZnO-HS.

Characterization of TiO₂-HS and ZnO-HS

Characterization of TiO₂-HS and ZnO-HS are significant in determining the structural and chemical properties of the compound. It is essential to prove that the compound has been encapsulated. Five instruments were used which are Fourier Transform Infrared (FTIR), Scanning Electron Microscope-Energy Emissive X-ray (SEM-EDX), Brunauer-Emmett-Teller (BET), X-ray diffractometer (XRD), Field Emission Scanning Electron Microscope (FESEM) and Transmission Electron Microscope (TEM). The infrared spectra were recorded at room temperature with 4 cm⁻¹ resolutions between 4000-400 cm⁻¹ using a Perkin-Elmer Spectrum TM 400 FTIR Spectrometer. The BET surface area of TiO₂-HS was determined by nitrogen adsorption using the automated adsorption instrument Micromeritics ASAP 2010. Characterization using XRD was conducted with a CuK α radiation ($\lambda = 1.5418$) at 30 kV and 20 mA ranging from $2\theta = 15^\circ$ to 90° and a scanning speed of 0.05° per second in a Siemens D5000 X-Ray diffractometer. FESEM images and elemental analysis of titanium, zinc, and silica elements in the particles were obtained using Philips XL 40. TEM images were taken with a JEOL-JEM-2100 transmission electron microscopy operated at an accelerating voltage of 200 kV.

Photocatalytic Degradation of Organ Arsenic

A standard solution of DMA was prepared with a concentration of 1000 ppm. The standard solution was diluted into 100 ppm and used for photodegradation. The DMA solutions were then introduced into the TiO₂, TiO₂-HS, ZnO, and ZnO-HS. The photocatalytic properties of all catalysts were investigated from the

results of the percentage degradation of DMA which was calculated from the absorbance value obtained from UV-Vis spectrometry of the collected DMA solutions obtained during the period of reactions (3 h) under UV radiation by UV lamp (365 nm, 6 watts). Quantitative determination of the degraded samples was measured using a Perkin Elmer UV-Vis spectrometer.

RESULTS AND DISCUSSION

Characterization of TiO₂-HS and ZnO-HS

The simple sol-gel method yielded the expected titanium dioxide and zinc oxide encapsulated with the hollow silica. The TiO₂-HS and ZnO-HS were successfully synthesized by the proposed method and the characterization of the products synthesized was done using FTIR, SEM-EDX, BET, XRD, FESEM, and TEM. From these techniques, the structural composition and morphology of TiO₂-HS and ZnO-HS synthesized were subsequently attained. The FTIR spectrum of TiO₂-HS shows two peaks for the absorption of Ti-O at 3398.76-1633.80 cm⁻¹ and Si-O at 1090.49 cm⁻¹ shown in Fig.-1 (a, b). The Si-O peak indicated the attachment of silica in the TiO₂-HS complex since showed expected peaks indicating the presence of titanium and silica in the complex. For ZnO-HS, the FTIR spectrum revealed a broad peak indicating the peak of Zn-O and Si-O overlapped at 1044.37 cm⁻¹. This frequency is assigned to the vibration of the Si-O-Si asymmetric stretching peak. This frequency is assigned to the vibration of the Si-O-Si asymmetric stretching peak.

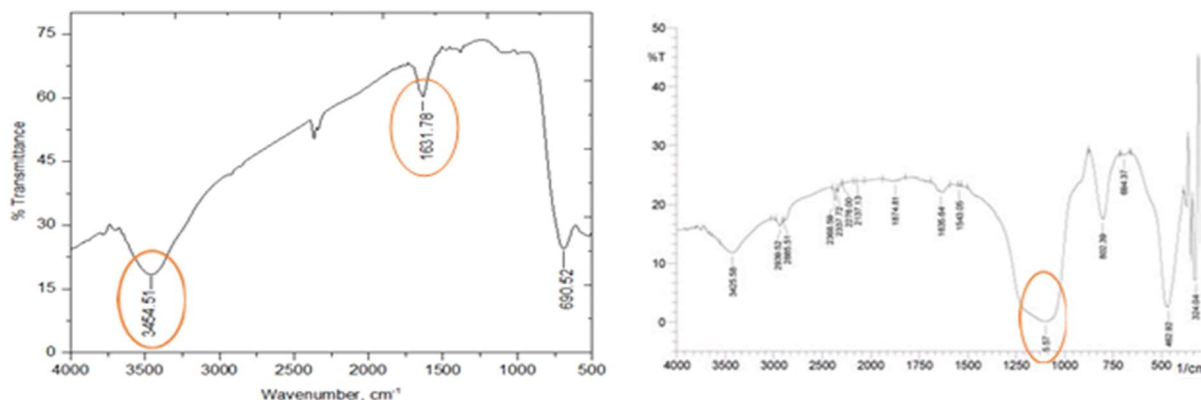


Fig -1 (a) FT-IR TiO₂-HS Shows Two Peaks for the Absorption of Ti-O at 3454.51 and (b) Si-O at 1090.49 cm⁻¹

Moreover, the SEM-EDX analysis results for Ti, Zn, and Si are shown in Table-1 and Table-2. The result from SEM-EDX analysis showed a decreasing atomic percentage of titanium and zinc while the atomic percentage of silica increased significantly in TiO₂-HS and ZnO-HS as compared to TiO₂ and ZnO, respectively.

Table-1: Weight and Atomic Percentage of Titanium and Silicon of TiO₂ and TiO₂-HS

Element	Weight percentage of titanium and silicon(%)		Atomic Percentage of titanium and silicon(%)	
	Ti	Si	Ti	Si
TiO ₂	60.36	0.42	40.39	0.48
TiO ₂ -HS	49.46	8.53	30.63	9.01

Table-2: Weight and Atomic Percentage of Zinc and Silicon of ZnO and ZnO-HS

Element	Weight percentage of zinc and silicon (%)		Atomic Percentage of zinc and silicon (%)	
	Zn	Si	Zn	Si
ZnO	92.98	0.00	76.44	0.00
ZnO-HS	88.33		66.00	1.25

Other than that, BET was used to determine the surface area.¹³ BET profile showed a decreasing value in surface area for the synthesized TiO₂-HS as compared to TiO₂ which decreased by 15.15 m²g⁻¹. This is due to the partial occupation of the pores by TiO₂, has been encapsulated with TiO₂. In contradiction, the surface area of ZnO-HS has a higher value as compared to ZnO by 10.78 m²g⁻¹. This shows that surface area of ZnO increased when coated with silica, hence will increase its efficiency. Result from XRD revealed that

in TiO_2 and ZnO , there are no other interfering peaks in the diffractogram indicating the absence of impurities in the TiO_2 and ZnO samples used in the characterization. For TiO_2 -HS, the peaks obtained are similar to the naked TiO_2 diffractogram at a certain degree but the shape of the peaks observed are slightly different. The peak for TiO_2 -HS shown is not as sharp as compared to peaks from the naked TiO_2 . The wide peaks shown in TiO_2 -HS could be originating from the silica which was encapsulated with TiO_2 . The crystallinity shown in the diffractogram of the sample is determined by the sharpness of peaks present. Since titanium in the form of anatase is a crystalline metal, it produced a sharp peak at an angle of 25° (2θ) with 100% intensity. The peaks seen resembling those from noise may be due to the existence of silica. It has low intensity due to its amorphous form. From the diffractogram, it can be observed that at an angle of 54° (2θ), the peak obtained in TiO_2 has good resolution while the peak from TiO_2 -HS is seemed to be overlapping. This indicated the existence of silica has strongly contributed to the overlapping of peaks with low intensity from the sample. The same explanation applied to ZnO -HS as the diffractogram shows peaks which are relatively hold a lower intensity as compared to ZnO .¹⁴ Further characterizations were carried out to investigate the morphology of both catalysts. The structural conformation of the synthesized TiO_2 -HS was evaluated by FESEM. The image shows the morphological appearance and surface structure of the synthesized TiO_2 -HS nanoparticles. The images were displayed in Fig.-2 (a, b).

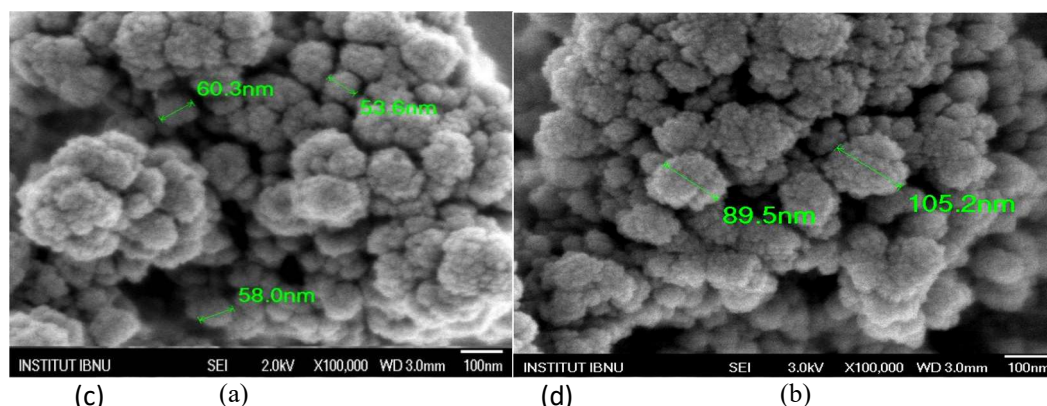


Fig.- 2: FESEM Morphology: (a) TiO_2 and (b) TiO_2 -HS Showing the Nano shapes of TiO_2 and TiO_2 -HS Particles

The morphology of the complex TiO_2 -HS is clearly shown in Fig.-2(a, b). The shapes for both bare TiO_2 and TiO_2 -HS resemble those of nanosphere particles. The average diameter for TiO_2 is 57.3 nm slightly shorter than the TiO_2 -HS with an average of 97.4 nm indicating the attachment of silica on the surface of TiO_2 with a weak bond which subsequently contributed to the larger size of the synthesized TiO_2 -HS. It may be due to the slight particle aggregation of the TiO_2 hollow sphere during the encapsulation procedure. Both TiO_2 and TiO_2 -HS appeared as amorphous materials. The sample intended for FESEM analysis must be kept carefully without being exposed to light or any interference. Disturbed or exposed samples can contribute to the production of unclear images. The images for ZnO and ZnO -HS can be observed from Fig.-3(a, b).

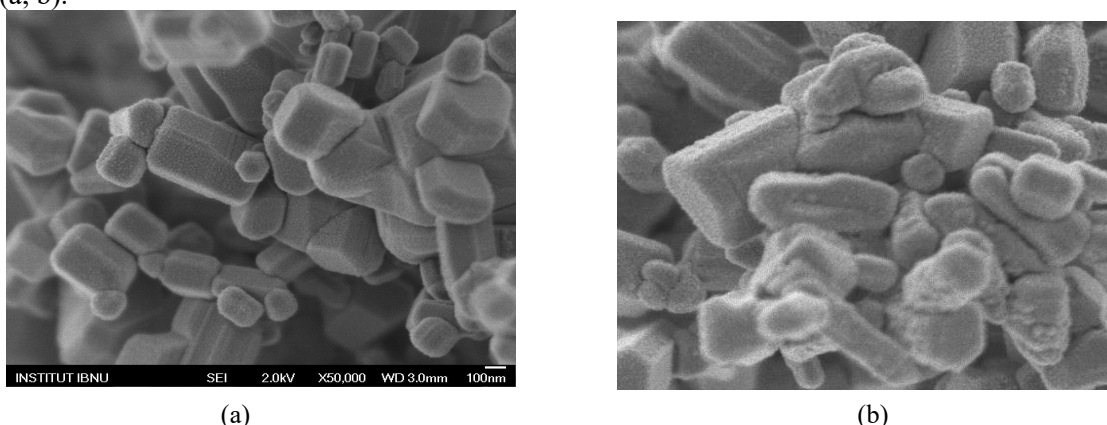


Fig.-3: FESEM Morphology of (a) ZnO and (b) ZnO -HS Showing the Hexagonal Shape of ZnO and ZnO -HS

FESEM photograph of ZnO and ZnO-HS respectively revealing the differences in surface morphology between the samples. Cubic and hexagonal structures are clearly visible in the ZnO sample as shown in Fig.-3(a). The addition of Tetraethyl orthosilicate (TEOS) into ZnO to form the silica coating on the surface of ZnO is clearly observed as nanoparticles attaching to the surface of ZnO as depicted in Fig.-3(b). Further evaluation of the structures of TiO_2 -HS was characterized by TEM. TEM morphologies were obtained on a JEOL-JEM-2100 microscope, using an accelerating voltage of 200 kV under various magnifications as shown in Fig.-4(a, b, c, and d). Figure-4 shows the TEM images of TiO_2 -HS under magnifications of 4(a) 100 nm, 4(b) 5 nm, 4(c) 50 nm and 4(d) 1 nm.

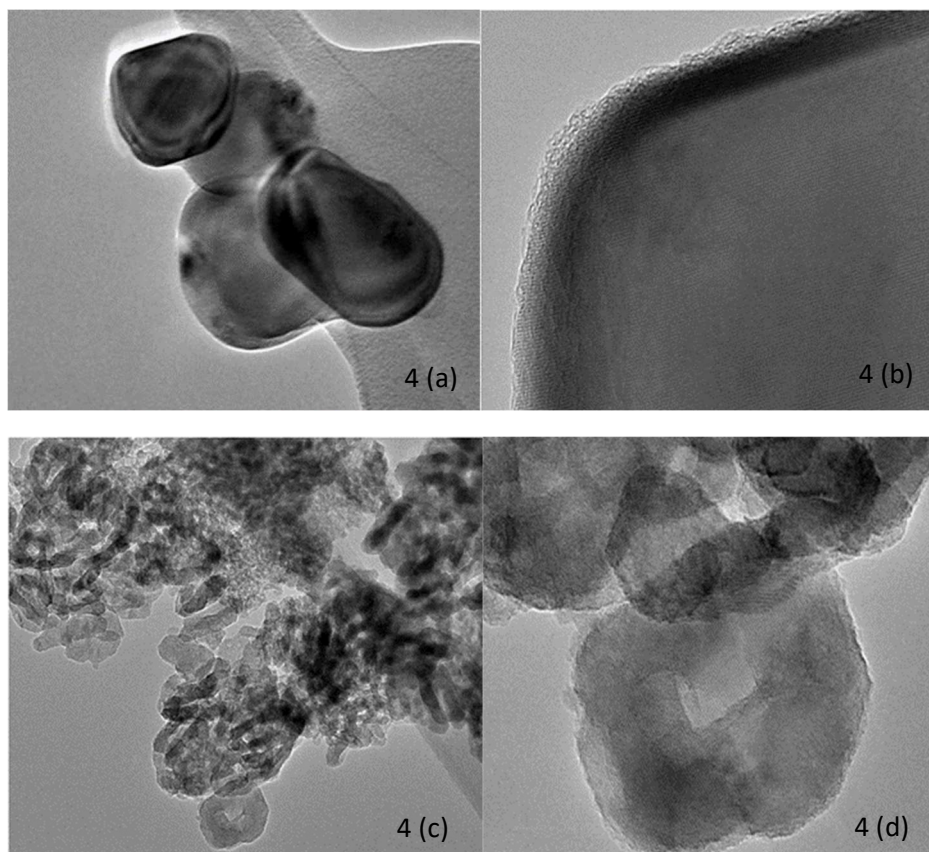


Fig.-4: TEM Images Showing Cross Section of: (a) TiO_2 , (b) TiO_2 , (c) TiO_2 -HS and (d) TiO_2 -HS

Figure-4(a) and (b) show dark crystalline rectangular shapes indicating the presence of titania particles. The strong contrast in 4(c) between the dark center and bright edges indicates the titanium dioxide particles have been surrounded by the silica particles. The hollow silica shape indicated by the bright centres and dark edges of the silica particles is shown in 4(d). It can be clearly seen from these images that the titania particles have been encapsulated in the amorphous hollow silica. The morphology of ZnO characterized by TEM is clearer than FESEM. The formation of a silica layer coated onto ZnO can be traced in the TEM image as in Fig.-4. Image 4(a) shows the ZnO particles which are the dark geometric-shaped particles and image 4(b) shows the encapsulation of ZnO particles into silica coating which can be clearly seen as the bright edges surrounding the dark particles inside.

Photocatalytic Degradation of Organoarsenic

After the encapsulation and characterizations of TiO_2 -HS and ZnO-HS, the complexes were then further used in the photocatalytic degradation of DMA in aqueous solutions. This work is related to the application of TiO_2 -HS and ZnO-HS as a catalyst to enhance the photodegradation of Organo arsenic. The study of degradation of DMA was controlled by a parameter namely the amount of catalyst being added into the DMA solutions. In contrast, the percentage degradation of DMA decreases as the amount of TiO_2 -HS added increases from 0.1 g to 0.5 g. The addition of TiO_2 -HS results in a lower percent of degradation as compared

to the naked TiO_2 . This is due to the limited surface area for absorption onto the surface of TiO_2 -HS as it was covered by the silica particles. Thus, only small molecules of DMA can be incorporated through the silica layer and undergo degradation.¹⁵ From Fig.-5, it can be calculated from absorbance obtained from UV-Vis spectrometry that the percentage degradation of pesticide decreased as the amount of TiO_2 added being reduced from 0.50g to 0.10g used this study of degradation of DMA was controlled by a parameter namely the amount of catalyst being added into the DMA solutions. In contrast, the percentage degradation of DMA decreases as the amount of TiO_2 -HS added increases from 0.1 g to 0.5 g. The addition of TiO_2 -HS results in a lower percent of degradation as compared to the naked TiO_2 . This is due to the limited surface area for absorption onto the surface of TiO_2 -HS as it was covered by the silica particles. Thus, only small molecules of DMA can be incorporated through the silica layer and undergo degradation.¹⁴

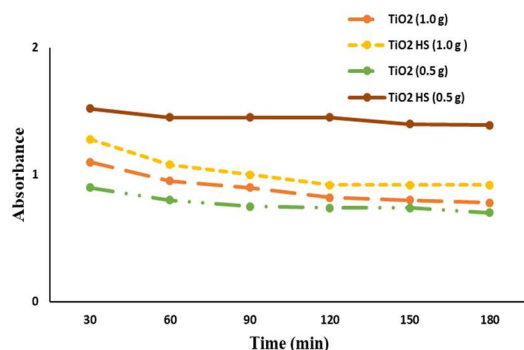


Fig.-5: Absorbance Versus Time for Degradation of DMA using TiO_2 and TiO_2 -HS in Different Weights

The percentage degradation of DMA using ZnO and ZnO-HS calculated is shown in Fig.-6 and Fig.-7.

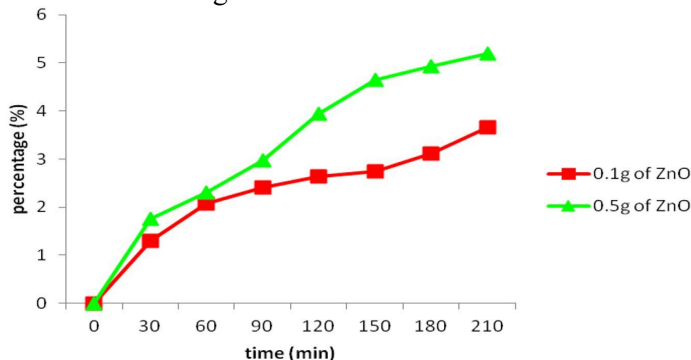


Fig.-6: Percentage degradation of DMA by using ZnO

It was observed in Fig.-6 that there is a slight difference in the percentage degradation of DMA using 0.1 g and 0.5 g of ZnO.

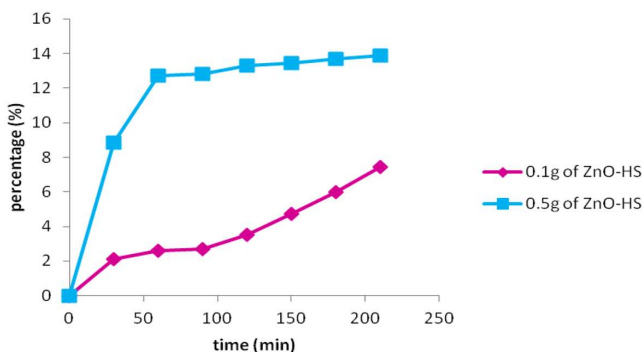


Fig.-7: Percentage Degradation of DMA by using ZnO-HS

On the other hand, Fig.-7 shows a big difference in the percentage degradation of DMA between the loadings. It is very clearly shown in Fig.-6 that a relatively higher percentage degradation of DMA was

affected by ZnO-HS was recorded with similar loadings as ZnO clearly showing the enhanced photodegradation properties. The encapsulation of hollow silica to both catalysts has given an enantioselective application in the photodegradation of DMA. The encapsulation of hollow silica in ZnO enhanced its photocatalytic activity but the existence of silica in TiO₂ has reduced its photocatalytic abilities to degrade DMA. This is proven by Fig.-8 which shows the overall percentage of DMA degradation for the four types of catalysts prepared.

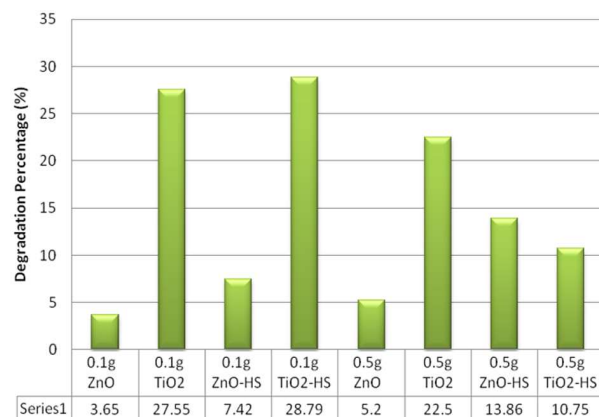


Fig.-8: Degradation Percentage of DMA using both Naked and Encapsulated Hollow Silica Catalysts with Different Loadings

It is shown that TiO₂ is a better catalyst as compared to ZnO because of the highest percentage of degradations held by TiO₂ (0.1 g) and TiO₂-HS (0.1 g). It can also be seen that there are no significant differences between percentage values for TiO₂ whether it is encapsulated in hollow silica or not. This is proven by the similar percentages which are 28.79 % and 27.55% from Fig.-8. So, it is known that the existence of silica is very minute in the 0.1 g catalyst loads. As for the 0.5 g TiO₂ loads, it gives a lower degradation percentage of DMA, and its percentage drastically decreased when the TiO₂-HS catalyst was introduced to DMA from 22.5 % to 10.75 %. These prove that silica has reduced the photocatalytic activity of TiO₂ by limiting the surface area for the adsorption of TiO₂. Surprisingly for ZnO, the encapsulation of hollow silica has increased the degradation percentage of DMA as can be seen from Fig.-8. The degradation percentage of DMA has increased from 3.65 % to 7.42 % for 0.1 g catalyst load and significantly increased from 5.2 % up to 13.86 % for the 0.5 g ZnO loads in Fig.-8.

CONCLUSION

From the analyses that were conducted and the results obtained, it can be concluded that the objectives of this work have been achieved. The TiO₂-HS and ZnO-HS were successfully prepared using the sol-gel method; Stober Method which was then proven by the characterization results. The photocatalytic activity of TiO₂-HS and ZnO-HS was examined and showed that the percentage for photodegradation of DMA catalyzed by TiO₂-HS is lower than the degradation catalyzed by the naked TiO₂. In contrast, the encapsulation of silica in ZnO has enhanced its catalytic activity as the percentage of degradation of DMA in ZnO-HS increased as compared to the naked ZnO. Hence, it was proven that silica doping gives enantioselective applications for both catalysts. Thus, silica doping has been proven to reduce the photocatalytic properties of TiO₂ but enhance the photocatalytic activity of ZnO.

ACKNOWLEDGMENTS

This research was supported by UMT (UMT 1+3 Matching grant/2021/53432). We also would like to thank UMT for the APC support.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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