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SYNTHESIS, CHARACTERIZATION AND CATALYTIC STUDIES OF TITANIA PHOTOCATALYST FOR DEGRADATION OF SELECTED DYES IN TEXTILE INDUSTRIAL WASTEWATER

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In this study, the optimum condition of Microwave Assisted Synthesis (MAS) method, in terms of its temperature and heating time setting were verified by evaluating the photocatalytic performance of the MAS prepared titania towards Rhodamine B and Reactive Golden Yellow R dyes. Three level of MAS temperatures (120^{o}C, 150^{o}C, 180^{o}C) and heating times (2 hours, 4 hours, 6 hours) have been selected and each prepared titania was calcined at 450^{o}C for an hour to activate the catalyst. The prepared titania was characterized by Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), nitrogen sorption (BET and BJH calculation), and Fourier Transform Infrared Spectroscopy (FTIR).

The photocatalytic performance of each prepared titania was evaluated by calculating the percentage of degradation of the selected dyes using the

UV/Visible spectroscopy data. It was found that the T-MAS-120-6h, T-MAS-150-4h, and T-MAS-180-4h condition have successfully synthesized titania with superior performance. At 120^{o}C, the superior performance of the MAS 6 hours prepared titania was due to its smaller anatase crystallite size (19.21 nm), larger surface area (117.51 m^2/g) and high pore volume (0.7726 cm^3/g) compared to titania prepared by shorter MAS heating time. Meanwhile, the higher photocatalytic activity of MAS 150^{o}C (4 hours) prepared titania was because of its small anatase crystallite size (13.62 nm), higher crystalline degree, higher pore volume (0.8254 cm^3/g), and high purity. For MAS 180^{o}C (4 hours), it was found that the large pore diameter on the ribbons surface, has contributed to the higher photocatalytic activity. Therefore, the charge–trapping site can be nearer to the pore so that the electron-hole can react with the dye molecule. Then, these MAS condition was selected to undergo the optimization by calcination.

The calcination treatment was tested at various temperatures (450^{o}C, 600^{o}C, 750^{o}C, 900^{o}C). Each calcined titania was characterized and analyzed using the SEM, XRD, nitrogen sorption, and FTIR. The result showed that the calcination process was important for attaining high photocatalytic activity. It was found that the 750^{o}C of calcination temperature has optimized the photocatalytic activity of the MAS 150^{o}C (4 hours) prepared titania to the highest level. This was due to the synergistic effect of anatase and rutile crystalline phase of the titania although the surface area (23.92 m^2/g) and pore volume (0.2718 cm^2/g) were smaller compared to titania calcined at lower temperature.

The performance of the optimized titania (titania prepared by MAS 150^{o}C (4 hours) – calcined at 750^{o}C) was tested to degrade Reactive Golden Yellow R dye in real textile wastewater (*Batik* industry) which was collected from a small Batik workshop located in Kuala Terengganu. The optimized titania displayed satisfactory photocatalytic performance towards degradation of Reactive Golden Yellow R dye in real *Batik* wastewater. However, the prepared titania was insufficient to remove all the Reactive Golden Yellow R dye in the wastewater within 4 hours. This was due to the effect of the properties and content of wastewater, including its pH value, chemicals and impurities, in which deteriorated the degradation process.

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SINTESIS, PENCIRIAN DAN KAJIAN PEMANGKINAN KE ATAS TITANIA FOTOPEMANGKIN UNTUK MENDEGRADASIKAN PEWARNA YANG TERPILIH DI DALAM AIR SISA INDUSTRI TEKSTIL

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Dalam kajian ini, suhu dan tempoh masa optima bagi kaedah Sintesis Bantuan Gelombang Mikro (MAS) telah ditentukan dengan menilai prestasi pemangkinan titania yang telah disintesis terhadap pewarna *Rhodamine B* dan *Reactive Golden Yellow R*. Tiga tahap suhu (120^{o}C, 150^{o}C, 180^{o}C) dan tiga tempoh masa (2 jam, 4 jam, 6 jam) untuk kaedah MAS telah dipilih bagi menyediakan titania, dan setiap titania yang disediakan telah dikalsinasikan pada suhu 450^{o}C selama sejam bagi tujuan pengaktifan pemangkin. Setiap titania yang disediakan telah dicirikan menggunakan teknik Mikroskopi Imbasan Elektron (SEM), Pembelauan Sinar-X (XRD), serapan nitrogen (pengiraan menggunakan kaedah BET and BJH), Spektroskopi Infra Merah (IR).

Prestasi pemangkinan untuk setiap titania yang disediakan telah dinilai dengan mencatat peratusan degradasi pewarna daripada data Spektroskopi Ultralembayung Sinar Nampak (UV-Vis). Kajian menunjukkan T-MAS-120-6h,

T-MAS-150-4h, dan T-MAS-180-4h telah berjaya menghasilkan titania dengan prestasi pemangkinan yang tinggi. Pada suhu 120^{o}C, titania yang disediakan selama 6 jam telah menunjukkan prestasi yang tinggi disebabkan oleh saiz hablur anatase yang lebih kecil (19.21 nm), luas permukaan yang lebih besar (117.51 m^2/g), dan isipadu liang yang lebih tinggi (0.7726 cm^3/g), berbanding dengan titania yang disediakan melalui MAS yang lebih singkat masanya. Manakala, titania yang disediakan melalui T-MAS-150-4h menunjukkan aktiviti pemangkinan yang tinggi kerana titania tersebut mempunyai saiz hablur anatase yang kecil (13.62 nm), darjah hablur serta isipadu liang (0.8254 cm^3/g) yang lebih tinggi, dan ketulenan yang tinggi. Bagi T-MAS-180-4h pula, kajian mendapati bahawa titania yang disediakan mempunyai aktiviti pemangkinan yang tinggi kerana titania tersebut mempunyai diameter liang yang besar, yang wujud di atas permukaan ribennya. Maka, tapak perangkap-cas adalah terletak berdekatan dengan liang, yang mana membolehkan rongga-elektron bertindak terhadap molekul pewarna.

Ketiga-tiga pasangan suhu dan tempoh masa bagi kaedah MAS ini telah dipilih untuk menjalani pengoptimuman melalui kalsinasi. Proses kalsinasi ini diuji pada suhu yang berbeza-beza (450^{o}C, 600^{o}C, 750^{o}C, 900^{o}C). Setiap titania yang dikalsinasi telah dicirikan dan dianalisa menggunakan teknik SEM, XRD, serapan nitrogen, dan IR. Keputusan menunjukkan bahawa proses kalsinasi adalah penting bagi meningkatkan prestasi pemangkin. Hasilnya, suhu kalsinasi 750^{o}C telah berjaya meningkatkan aktiviti pemangkinan bagi titania yang disediakan melalui T-MAS-150-4h pada tahap yang tertinggi. Hal ini adalah disebabkan oleh kesan sinergi yang dihasilkan oleh fasa hablur anatase dan rutile dalam titania tersebut walaupun luas permukaan (23.92 m^2/g) dan isipadu liangnya (0.2718 cm^2/g) adalah lebih kecil daripada titania yang dikalsinasi pada suhu yang lebih rendah.

Titania yang mempunyai prestasi yang tertinggi (titania yang disediakan melalui T-MAS-150-4h-C750) telah diuji untuk mendegradasikan pewarna *Reactive Golden Yellow R* di dalam air sisa tekstil sebenar (industri Batik) yang diambil daripada bengkel perusahaan Batik kecil-kecilan di Kuala Terengganu. Titania tersebut telah menunjukkan prestasi fotopemangkinan yang memuaskan terhadap degradasi pewarna *Reactive Golden Yellow R* di dalam air sisa Batik tersebut. Namun, titania tersebut tidak dapat membantu mendegradasikan sepenuhnya pewarna *Reactive Golden Yellow R* di dalam air sisa tersebut dalam masa 4 jam. Hal ini adalah disebabkan oleh kesan daripada sifat dan kandungan air sisa tersebut termasuk nilai pH, bahan-bahan kimia dan kehadiran bendasing, telah membantutkan proses degradasi.