

Pengembangan fotokatalis bermagnet ($\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$) untuk eliminasi zat organik metilen biru dan paraquat dalam air =
Development of magnetic photocatalysts ($\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$) for elimination of organic compounds methylene blue and paraquat in water

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Abstrak

[ABSTRAK

Fotokatalis bermagnet $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ telah dibuat dengan cara heteroaglomerasi.

Fotokatalis bermagnet ini diterapkan dalam reaktor sistem slurry untuk eliminasi zat organik (metilen biru dan paraquat) dalam air. Dan juga, fotokatalis bermagnet ini memberi kemudahan untuk dikumpulkan kembali dengan bantuan medan magnet luar sehingga fotokatalis bekas pakai ini dapat digunakan kembali secara berulang-ulang.

Sintesis fotokatalis bermagnet $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ diawali dengan membuat nanopartikel Fe_3O_4 dengan cara presipitasi menggunakan campuran Fe(III)/Fe(II) (rasio mol 2:1) dalam larutan amonia dan kemudian dilapisi dengan SiO_2 dengan cara hidrolisis ion silikat. $\text{Fe}_3\text{O}_4/\text{SiO}_2$ yang terbentuk dicampurkan dengan TiO_2 dengan cara hetero-aglomerasi, untuk memperoleh fotokatalis bermagnet $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$. Dalam penelitian ini, ada dua jenis TiO_2 komersil yang digunakan, yaitu nanopartikel TiO_2 Aldrich dan nanopartikel TiO_2 P25 Evonik. Fotokatalis bermagnet yang telah dibuat dikarakterisasi dengan berbagai teknik, di antaranya, difraksi sinar-x (XRD) untuk menentukan fasa kristal, zeta potensial meter untuk menentukan muatan permukaan partikel, VSM untuk menentukan sifat kemagnetan, spektrometer FTIR dan Raman untuk identifikasi gugus fungsi dan ikatan logam-oksida, EDS untuk menentukan komposisi unsur permukaan, TEM untuk mengetahui morfologi dan adsorpsi- N_2 untuk menentukan luas permukaan. Hasil karakterisasi XRD menunjukkan bahwa TiO_2 -Aldrich mengandung fasa anatase 55% dan rutil 45% sedangkan TiO_2 -P25 Evonik mengandung fasa anatase 86% dan rutil 14%. Fasa magnetit (Fe_3O_4), TiO_2 anatase dan rutil dipertahankan dalam fotokatalis bermagnet yang terbentuk. Dalam tahapan pembentukan bahan, ke dua jenis TiO_2 telah berhasil menempel secara permanen pada $\text{Fe}_3\text{O}_4/\text{SiO}_2$ melalui interaksi elektrostatik gugus hidroksil permukaan masing-masing oksida. Spektrometri FTIR mengamati ikatan Si-O-Ti yang terbentuk, hasil interaksi elektrostatik TiO_2 dengan $\text{Fe}_3\text{O}_4/\text{SiO}_2$, selanjutnya dinotasikan sebagai $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ -Ald dan $\text{Fe}_3\text{O}_4/\text{SiO}_2/\text{TiO}_2$ -P25. Kedua jenis bahan mempunyai sedikit perbedaan sifat fisika-kimia dan morfologi. Semakin banyak fraksi TiO_2 dalam bahan maka luas permukaannya semakin turun. Fotokatalis bermagnet yang diperoleh mempunyai sifat magnet cukup baik dan nilai remanent magnetization dan coercivity yang rendah, artinya bahan ini dapat

dengan mudah dikumpulkan kembali dari cairan dengan bantuan medan magnet luar dan tanpa medan magnet dapat terdispersi kembali dengan baik dalam air. Fe₃O₄/TiO₂-Ald dan Fe₃O₄/TiO₂-P25 yang telah dibuat mampu mengeliminasi metilen biru dan paraquat dalam air melalui proses fotokatalitik, namun demikian aktivitasnya dianggap masih rendah. Diduga terjadi pelemahan aktivitas TiO₂ yang ada dalam fotokatalis bermagnet akibat dari efek fotodisolusi. Adanya barrier SiO₂ di antara Fe₃O₄ dan TiO₂, metilen biru yang terdegradasi meningkat dari 44,2% menjadi 52,3% oleh Fe₃O₄/SiO₂/TiO₂-Ald dan paraquat yang terdegradasi meningkat dari 16,3% menjadi 45,8% oleh Fe₃O₄/SiO₂/TiO₂-P25. Aktivitas kedua fotokatalis bermagnet yang punya barrier SiO₂ ini setara dengan aktivitas rasio fraksi aktif TiO₂ terhadap TiO₂ murninya. Tidak ada kehilangan aktivitas TiO₂ dalam fotokatalis bermagnet setelah diberi barrier SiO₂. Adanya SiO₂ dalam fotokatalis bermagnet ini juga mampu mengeliminasi senyawa metilen biru dan paraquat melalui proses adsorpsi. Maka dari itu, lebih banyak lagi metilen biru dan paraquat tereliminasi melalui kedua proses. Secara total, Fe₃O₄/SiO₂/TiO₂-Ald mampu mengeliminasi metilen biru 87,3% dan paraquat 71,5%. Sedangkan Fe₃O₄/SiO₂/TiO₂-P25 mampu mengeliminasi paraquat 82,6%. Kapasitas adsorpsi Fe₃O₄/SiO₂/TiO₂-Ald lebih rendah dari kapasitas adsorpsi Fe₃O₄/SiO₂/TiO₂-P25, tetapi aktivitas fotokatalitik Fe₃O₄/SiO₂/TiO₂-Ald lebih tinggi dari aktivitas fotokatalitik Fe₃O₄/SiO₂/TiO₂-P25. Fotokatalis bermagnet yang dikembangkan menunjukkan kestabilan fotokatalitik paling tidak sampai empat kali pemakaian berulang dan juga masih dapat dikumpulkan kembali dengan mudah dengan bantuan medan magnet luar

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ABSTRAK

Magnetic photocatalysts of Fe₃O₄/SiO₂/TiO₂ have been prepared using heteroagglomeration method. The magnetic photocatalysts were applied in slurry reactor system for elimination of organic compounds (methylene blue and paraquat) in water. In addition, the magnetic photocatalysts are able to be recollected easily with the assistance of an external magnetic field so that the spent composite can be used repeatedly.

Synthesis of magnetic photocatalysts of Fe₃O₄/SiO₂/TiO₂ were preceded by preparing Fe₃O₄ nanoparticles through precipitation method using mixture of Fe(III)/Fe(II) (2:1 mole ratio) in ammonia solution and further coating with SiO₂ through hydrolysis of silicate ion. The formed Fe₃O₄/SiO₂ were mixed with TiO₂ in hetero-agglomeration manner, to obtain Fe₃O₄/SiO₂/TiO₂ magnetic photocatalysts. In this study, two type of commercial TiO₂ nanoparticles were used, namely; TiO₂ Aldrich and TiO₂-P25 Evonik.

The prepared magnetic photocatalysts were characterized with various techniques, i.e, x-ray diffraction (XRD) to determine the crystal phase, zeta potensial meter to determine the surface charge of particles, VSM to determine the magnetic properties, FTIR and Raman spectrometer to identify the functional groups and

metal-oxide bond, EDS to determine the surface chemical composition, TEM for morphological examination and N₂-adsorption to determine surface area. The results of XRD characterization showed that TiO₂-Aldrich contains 55% of anatase and 45% of rutile, while TiO₂-P25 Evonik contains 86% of anatase and 14% of rutile. Magnetite (Fe₃O₄), anatase and rutile phase of TiO₂ were retained in the formed composites. In the stage of composite formation, both TiO₂ types have been successfully attached to Fe₃O₄/SiO₂ via electrostatic interaction of surface hydroxyl group of oxides. FTIR spectrometry analysis revealed the formed Si-O-Ti bond, resulting of electrostatic interaction both TiO₂ and Fe₃O₄/SiO₂. Hence, they were denoted as Fe₃O₄/SiO₂/TiO₂-Ald dan Fe₃O₄/SiO₂/TiO₂-P25. The both magnetic photocatalysts have a slight different physico-chemical properties and morphology. The more the fraction of TiO₂ in magnetic photocatalysts, the lower its surface area. The obtained magnetic photocatalysts have high saturation magnetization and low coercivity and remanent magnetization value. It means that the magnetic photocatalysts can be still recollected from water with assistance of external magnetic field. In a non-magnetic field, magnetic photocatalyst can be well dispersed in water again.

The formed Fe₃O₄/TiO₂-Ald and Fe₃O₄/TiO₂-P25 was able to eliminate methylene blue and paraquat in water by photocatalytic process, nevertheless its activities was low. Allegedly it occurs weakening of TiO₂ activity in magnetic photocatalyst caused by photodissolution effects. The presence of SiO₂ barrier between Fe₃O₄ and TiO₂, the degraded methylene blue was increased from 44.2% to 52.3% by Fe₃O₄/SiO₂/TiO₂-Ald and the degraded paraquat was increased from 16.3% to 45.8% by Fe₃O₄/SiO₂/TiO₂-P25. The photocatalytic activity of the both magnetic photocatalysts which having SiO₂ barrier was equivalent to the activity of fraction ratio of TiO₂ to its pure TiO₂. There was no loss of photocatalytic activity of TiO₂ in magnetic photocatalysts after SiO₂ barrier being introduced. The presence of SiO₂ on magnetic photocatalysts was also able to eliminate methylene blue and paraquat compounds through adsorption process. Therefore, more methylene blue and paraquat eliminated via both process. Totally, the Fe₃O₄/SiO₂/TiO₂-Ald was able to eliminate 87.3% of methylene blue and 71.5% of paraquat. Meanwhile Fe₃O₄/SiO₂/TiO₂-P25 was able to eliminate 82.6% of paraquat. Adsorption capacity of Fe₃O₄/SiO₂/TiO₂-Ald was lower than that of Fe₃O₄/SiO₂/TiO₂-P25, but photocatalytic activity of Fe₃O₄/SiO₂/TiO₂-Ald was higher than that of Fe₃O₄/SiO₂/TiO₂-P25. The developed magnetic photocatalysts show its activity photocatalytic stability and still can be well magnetically separated after being repeatedly used for four times.;Magnetic photocatalysts of Fe₃O₄/SiO₂/TiO₂ have been prepared using heteroagglomeration method. The magnetic photocatalysts were applied in slurry reactor system for elimination of organic compounds (methylene blue and paraquat) in water. In addition, the magnetic photocatalysts are able to be recollected easily with the assistance of an external magnetic field so that the spent composite can

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