Arabian Journal for Science and Engineering
https://doi.org/10.1007/s13369-019-04029-9

RESEARCH ARTICLE - CHEMICAL ENGINEERING

Characterization of Bauxite as a Potential Natural Photocatalyst for Photodegradation of Textile Dye

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Received: 7 January 2019 / Accepted: 3 July 2019

Abstract

Previous water treatments used are nondestructive and lead to the formation of new type of pollution. Photocatalytic reaction which induced by illumination of semiconductors in suspension was one of the most promising processes for wastewater treatment due to the ability of providing an interface with the aqueous medium and induce an advanced oxidation process. In this study, Malaysian bauxite has been selected as a new potential candidate for photocatalytic degradation. Bauxite powder was undergone heat treatment ranging from 800 to 1000 °C and then was characterized by field emission scanning electron microscope, X-ray diffraction (XRD), Brunauer–Emmett–Teller, ultraviolet–visible–near infrared spectroscopy and suspension absorbance test to determine the existing components and their composition, structure of the powder, band gap energy as well as the photocatalytic efficiency of bauxite in suspension of various concentrations of Reactive Black 5 (RB5) ranging from 5 to 20 ppm. The finding revealed that bauxite possessed slight changes in terms of XRF composition as well as particle size due to thermal process. This is supported by XRD analysis which showed that phase conversion occurred at 800 °C. Heat-treated bauxite possesses low band gap energy (2.04 eV). The photocatalytic activity of raw bauxite suspension was measured after 360 min illumination, and it proved that there was degradation of 67% and 63% from the initial 5 ppm concentration of RB5 using visible and UV lights, respectively. This finding clearly shows that bauxite exhibits good photocatalytic degradation and hence has a promising potential in removing dyes for industrial wastewater.

Keywords Water and wastewater treatment · Bauxite · Photocatalytic degradation · Organic contaminants · Thermal treatment

1 Introduction

Water pollution is the contamination of water bodies including lakes, rivers, aquifers, oceans as well as groundwater, and it had become major global problem which necessitate for continuous monitoring and evaluation. Previous study conducted suggested that it is the global leading basis of deaths and diseases [1]. There are two main types of water pollution which are point source and nonpoint source [2]. Point source refers to the fairly easy to trace and identified the source of contaminants, whereas it is vice versa where there is no specific source for nonpoint source pollution. Water pollution has been seen itself to bring many implications to the daily lives. Generally, there are thousands of pollutants contaminating our environment including organic, inorganic and biological species [3] which has been reported to be in the environment at trace level [4]. When toxic substances (pollutants) entered to the water bodies, they get dissolved or lie suspended in water or get deposited on the bed. This results in the pollution of water whereby the quality of the water deteriorates, affecting aquatic ecosystems. Pollutants can also sleep down and affect the groundwater deposits.

Bauxite was the most important ore of aluminum which belongs to the family of lateritic rocks. It was characterized by the enrichment of aluminum hydroxide minerals, such

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as gibbsite [$Al(OH)_3$], boehmite [γ-AlO(OH)] and diaspore [α-AlO(OH)], together with the iron ore minerals hematite and goethite, the kaolinite and small amounts of anatase (TiO_2). The chemical, mineralogical and physico-mechanical properties of these bauxites widely vary depending upon the parent rock composition, mode of generation, geomorphologic position and duration and age of bauxite formation. Bauxite may be white, gray, yellow or red with white streak and dull to earthy luster. The existence of bauxite in Malaysia that has been discovered was reddish-brown color which indicated that it was naturally composed of heterogeneous material and comprises of more than one aluminum hydroxide minerals [5]. Bauxite contains of several trace elements such as lead, nickel, manganese and also naturally occurring radioactive materials (uranium and thorium) [6].

Bauxite was initially formed from laterite formation [7]. This formation can be yielded due to intense weathering in humid, warm, intertropical regions of the world and was typically rich in kaolinitic clay as in Fig. 1. Large amount (up to 90%) of bauxite resources can be found in tropical area [8]. Central and South America particularly Brazil and Australia had been recorded as large reserve of bauxite worldwide [9]. In Malaysia, bauxite resources were mainly located in Bukit Batu, Bukit Gebong, Lundu-Sematan and Tanjung Seberang in Sarawak, Bukit Mengkabau and Labuk Valley in Sabah, Sungai Rengit and Teluk Ramunia in Johor and Bukit Goh in Kuantan, Pahang [10].

Industrial evolution as well as rapid industrialization and unskilled treatment of wastewater has led to the destruction of water quality [1]. Water pollution due to the existing dyes particles gave negative influences to the human beings such as disturbance in genetic system, hormonal and enzymatic activities [11]. Wastewater from the industrial effluents has received more and more attention due to the unpleasant view and their toxic effects including nausea, vomiting, diarrhea, muscle weakness, salivation and cancer [12] which latter lead to serious contamination in many countries across the globe as some of the dyes contain aromatic amines. Dyes were easily to be recognized in the wastewater as they were highly visible, and their presence in the water was commonly undesirable. Reactive Black 5 (RB5) dye was the most frequently used synthetic reactive dyes in the textile industry. This dye was highly soluble in water and was able to form covalent bonds between dye pigment and fiber due to the existence of chromophoric reactive group. RB5 was chosen as model pollutant in this study due to incomplete fixation reaction during dyeing process. This occurrence happened as there was competition between the reaction of reactive dye group (vinylsulphone) and fibers [13].

The toxicity and carcinogenic effect of the reactive dyes have created serious hazard to the environment [14]. These harmful effects were not only disturbs the living organisms, but the entire aquatic ecosystem was also affected. In order to combat this issue, the toxicity assessment of dyes in the

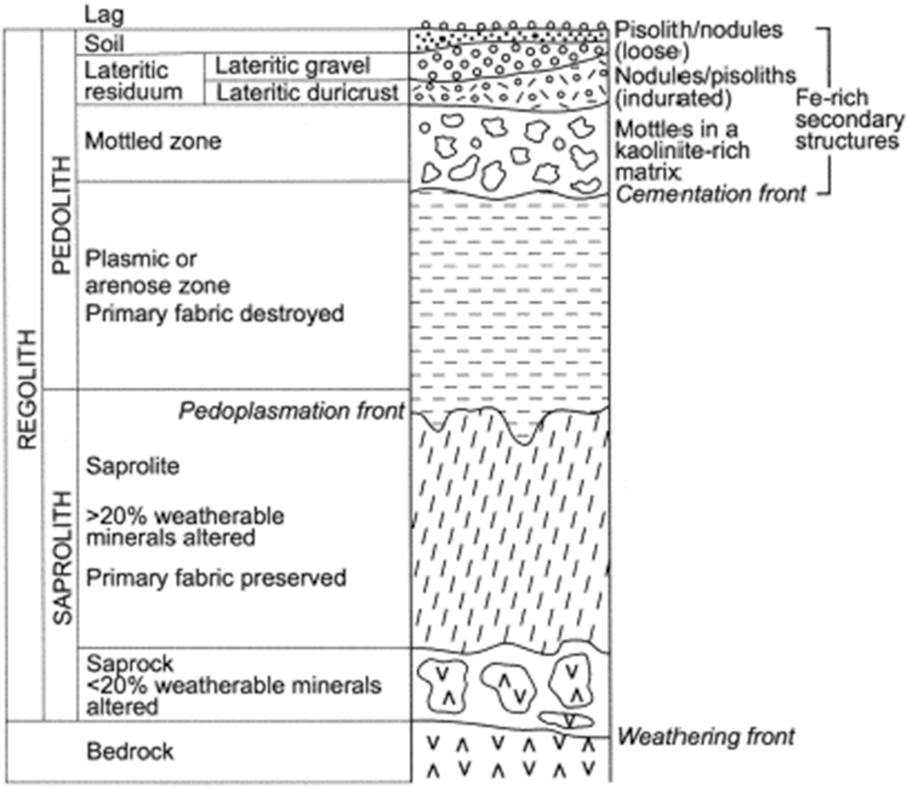


Fig. 1 Generalized lateritic regolith profile [27]

environment has been extensively studied [15]. Stringent environmental regulations create the elimination of reactive dyes from textile effluents become compulsory since most of them were recalcitrant due to their complexity of the structure [16].

Previous various water treatment technologies for instance ion exchange, reverse osmosis, electrochemical and absorption have been used for dye removal. Among these aforementioned methods, adsorption seemed to be the best technique as it was cost-effective, eco-friendly, can be reproducible and ease to be operated [17]. It also has universal in nature as can be used to remove almost all pollutants from the wastewater. However, this method was not capable to work under natural water conditions [11] and accumulation of pollutant load can be transferred from aqueous to the adsorbent phase.

Advanced oxidation process (AOP) was believed to decompose this contaminant [18] and the promising result that has been obtained previously in which they were able to degrade the pollutants [19, 20] and completely mineralize the contaminants to water (H_2O) [16], carbon dioxide (CO_2), and mineral acids had make it receive high interest to be studied. Besides, low cost and moderate pressure and temperature [21] required become additional winning factor to this technology. AOPs engage the degradation of pollutants by irradiating metal oxide semiconductor particles named as catalyst in the suspension under light exposure (visible or UV light). A semiconductor material has an electrical conductivity value falling between conductor and insulator [22]. Among several number of semiconductor materials, titanium dioxide (TiO_2) was widely employed as photocatalyst in wastewater treatment for industrial effluent. The reason why TiO_2 was often selected as a photocatalyst was due to its superhydrophilicity, strong oxidizing abilities for degradation of organic compounds and long-term chemical stability, inexpensive and nontoxic properties [22]. In addition, apart of TiO_2, bauxite also contains other potential photocatalysts such as zinc oxide (ZnO), ferrum oxide (Fe_2O_3) and cadmium sulfide (CdS) which made this study interesting as there were synergized photocatalytic properties [23]. The existence of several semiconductors in a single bauxite compound was believed that this material has self-cleaning ability, and further investigation on this particular study will be conducted in future.

Recent literature survey on RB5 degradation [19, 24–26] found that this contaminant has been studied frequently for water treatment; however, the usage of natural existing mineral as a catalyst has not been fully covered. Previous researches on similar topic focused on RB5 degradation using synthetic or commercial available catalyst such as nanotitanium dioxide [19], n-doped TiO_2 [24], MnO_2 with graphene oxide (GO) [25] and MnO_2 impregnated with activated carbon [26]. It was thus appropriate to evaluate the removal efficiency of this bauxite on RB5 contamination. Attempts have been made to evaluate the degradation of dye contaminants by varying the concentration of RB5 and catalyst loading.

This paper aims to investigate the existence of photocatalyst in bauxite powder from Kuantan, Pahang, Malaysia, and to study the effect of calcining temperature (ranging from 800 to 1000 °C) on the composition of the bauxite as well as their changes in terms of physicochemical characteristics. The main rational of conducting thermal treatments to the bauxite sample was to remove existed moisture, enhance particle density as well as reducing porosity. Degradation of textile dye, namely RB5 in the synthetic colored wastewater via photocatalysis process, was also being investigated in this study by varying the concentration of the contaminant (5–20 ppm). Thus, the novelty of this study was the usage of bauxite as new potential photocatalyst as it consists of heterogeneous semiconductors in a mineral and possesses good properties of semiconductors which are able to react under both UV and visible light irradiation. This application has a significant impact on the integrated utilization of bauxite resource, resource conservation and environmental protection.

2 Methodology

2.1 Materials

The starting materials, bauxite used, were obtained from Kuantan, Pahang. Obtained bauxite was then sieved up to 50 micron prior to the characterization. All the chemicals used in this study were analytical reagent grade and were used as received without further dilution. Bauxite powder was heat-treated to various temperatures ranging from 800 to 1000 °C to allow the purification of this mineral as well as remove unwanted trace elements. The exposure time was fixed to 60 min with the rate of reaction of 5 °C/min based on previous study [27].

2.2 Characterization of Bauxite Powder

The morphological nature of bauxite powder was explored qualitatively by field emission scanning electron microscopy (FESEM; Model: SU2080, Hitachi) at an accelerated voltage of 2.0 kV. The presence of photocatalyst was then detected using the line scan of spectrum of energy dispersion of X-ray (EDX; Model: X-MaxN 51-XMX1011, Oxford Instrument). The sample was positioned on a metal holder and then was coated with gold under vacuum sputtering approximately for 3 min.

The crystallinity and the phase formation of bauxite were analyzed using an X-ray diffractometer (XRD;

Model: D5000, Siemens). The measurements were carried out at 40 kV and 40 mA, which employed a CuKα radiation at a wavelength of 0.15418 nm at an angular incidence of $2\theta = 20°–80°$ with a scan step speed of 1°/min. To analyze the particle size of the bauxite, samples were dispersed in $NaP_2O_7 \cdot 10H_2O$ solution to ensure the suspension was stabilized. Then, the samples were treated in an ultrasonic bath and kept at 29 ± 1 °C for 10 min prior to the analysis. The surface areas were calculated by using Brunauer–Emmett–Teller (BET) surface area measurement by nitrogen adsorption–desorption analysis. Bauxite powder was degassed at 250 °C prior to BET measurement using a Micromeritics (3Flex, Ver: 3.01, USA).

2.3 Band Gap Determination

The UV–VIS–NIR spectra were used to examine the optical property of the bauxite powder on the light absorption capability under UV and visible light irradiation. Samples were clamped to the sample holder and scanned between 200 and 1000 nm using ultraviolet–visible–near infrared (UV–VIS–NIR) spectrophotometer (Model: UV-3101PC, Shidmadzu).

2.4 Photocatalytic Activity Measurement

In order to evaluate the photocatalytic activity of bauxite, Reactive Black 5 (RB5) was used as dye contaminant in the synthetic wastewater under UV light and visible light irradiation. Bauxite powder (0.1 g) was added in both aqueous solutions containing 500 ml of RB5 with several concentrations ranging from 5 to 20 ppm. The suspension was stirred in dark place for 30 min to reach adsorption–desorption equilibrium. The suspension (10 ml) was taken through 0.45-µm polyamide syringe filter to eliminate excess photocatalyst prior to analysis and treated as initial concentration (C_0). The differential absorbance of irradiated RB5 with time throughout the experiment was measured by PerkinElmer UV–visible spectrophotometer at 597 nm and was compared with the blank (photolysis of RB5 without photocatalyst). The photocatalytic activity was expressed in the percentage of RB5 degradation according to the following equation:

$$\text{Degradation of organic contaminants} = \frac{C_0 - C_t}{C_t} \times 100 \tag{1}$$

where C_0 is the initial concentration at time $t = 0$ and C_t is the concentration at time t (30, 60, 90, 120, 150, 180, 210, 240, 270, 300, 330 and 360 min).

3 Results and Discussion

3.1 Physicochemical Characterization of Bauxite

The physical characteristics of raw bauxite were analyzed using FESEM microscopy analysis as shown in Fig. 2. It was revealed that there was an aggregation of pisolitic (small, larger than 2 mm) and oolitic (very small) spherical masses within a matrix. Bauxite was a form of rock consists mainly with a mixture of two iron oxides, namely goethite and hematite, which gives the reddish color to this mineral [28].

The presence of the elemental composition of bauxite was further confirmed with EDX mapping analysis. EDX analysis confirmed the presence of oxygen (O), alumina (Al), titanium (Ti), carbon (C), ferrum (Fe) and silica (S) elements was detected in the bauxite sample as shown in Fig. 3a. The EDX spectrum in Fig. 3b shows that bauxite consists of aforementioned elements with the corresponding percentages given. The obtained result was comparable to the other bauxite from other countries; however, the constituents of the compounds were distinguished one another. This phenomenon was explained by weathering factors and geographical structure of the region [29] as the bauxitization generally formed due to weathering effect.

The XRF results listed in Table 1 revealed that bauxite was mainly composed by four compounds which were titanium dioxide (TiO_2), silicon dioxide (SiO_2), iron oxide (Fe2O$_3$) and aluminum oxide (Al_2O_3). Thermal treatment on bauxite did not give major significant impact to its composition. Most of the compounds in the bauxite were less toxic and only cause acute toxicity to the organisms except for manganese oxide (Mn_2O_3) which display high oxidative capability [30]; however, small quantity of 0.05% of this compound in bauxite demonstrated that the toxicity effect might be low and almost not toxic. As this study was focused

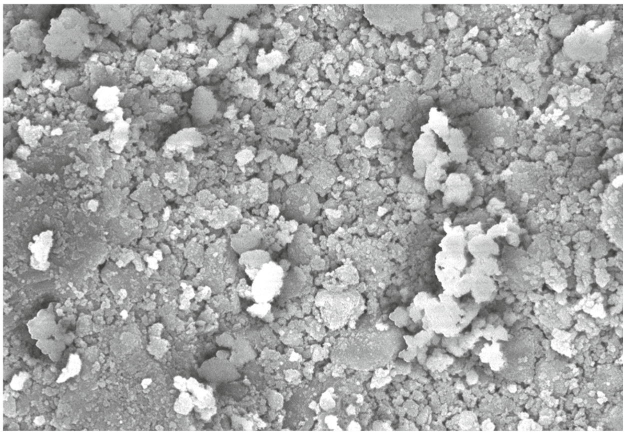
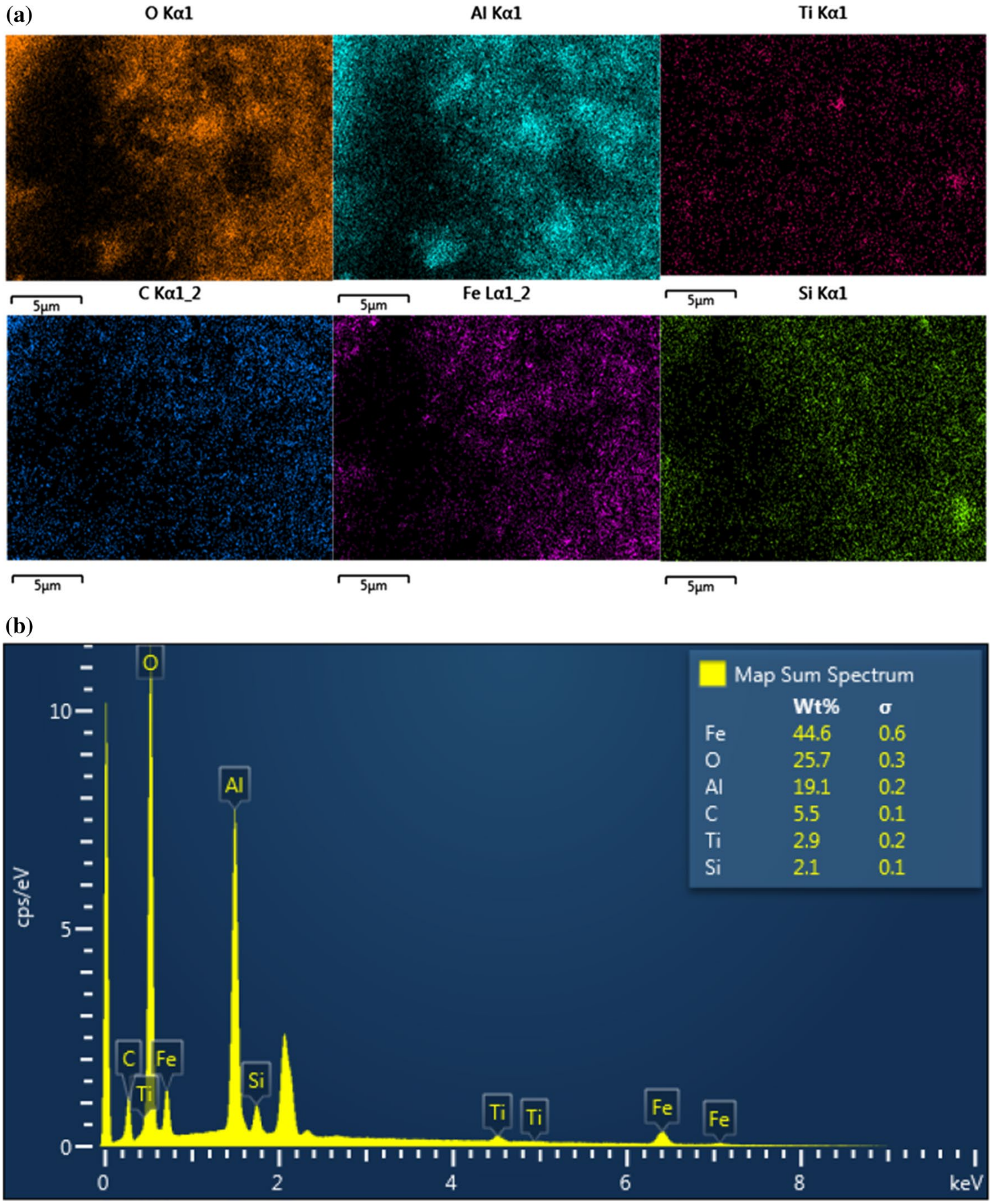


Fig. 2 FESEM of the bauxite



	Wt%	σ
Fe	44.6	0.6
O	25.7	0.3
Al	19.1	0.2
C	5.5	0.1
Ti	2.9	0.2
Si	2.1	0.1

Fig. 3 **a** EDX mapping image of bauxite and **b** spectrum of bauxite

on the photocatalytic process in which the main concern was on availability of potential photocatalysts, chemical compounds such as TiO_2, Fe_2O_3 and ZnO were considered as highly desired components. Previous study conducted by a group of researchers [31] showed that SiO_2 can also contribute to the improvement in the photocatalytic activity by increasing surface hydroxyl groups and contaminant adsorption. From the finding in Table 1, SiO_2 and aforementioned potential catalysts were naturally exist in the bauxite sample used in this study.

XRD diffraction patterns of untreated and heat-treated bauxite shown in Fig. 4 demonstrated that the samples consist of high crystallinity solid. The diffraction peak of anatase (101) was observed at $2\theta = 36.4°$ (DB Card Number 02-002-0387) and $2\theta = 37.8°$ for hematite (Fe_2O_3) (DB Card Number 01-072-6226) in the untreated bauxite, whereas,

Table 1 Chemical composition based on XRF analysis of Malaysian bauxite

Element	Bauxite samples (%)					
	Raw	600 °C	700 °C	800 °C	900 °C	1000 °C
MgO	0.43	0.00	0.00	0.41	0.47	0.49
Al_2O_3	50.39	54.20	54.60	55.41	56.87	60.14
SiO_2	18.92	5.11	5.27	15.59	13.34	11.56
P_2O_5	0.40	0.00	0.00	0.31	0.20	0.21
SO_3	0.48	0.31	0.27	0.51	0.26	0.20
TiO_2	4.28	6.37	5.96	4.05	4.56	4.12
Cr_2O_3	0.11	0.22	0.15	0.12	0.12	0.12
Fe_2O_3	24.82	32.5	32.0	23.45	24.07	23.06
Mn_2O_3	0.04	0.00	0.00	0.05	0.05	0.05

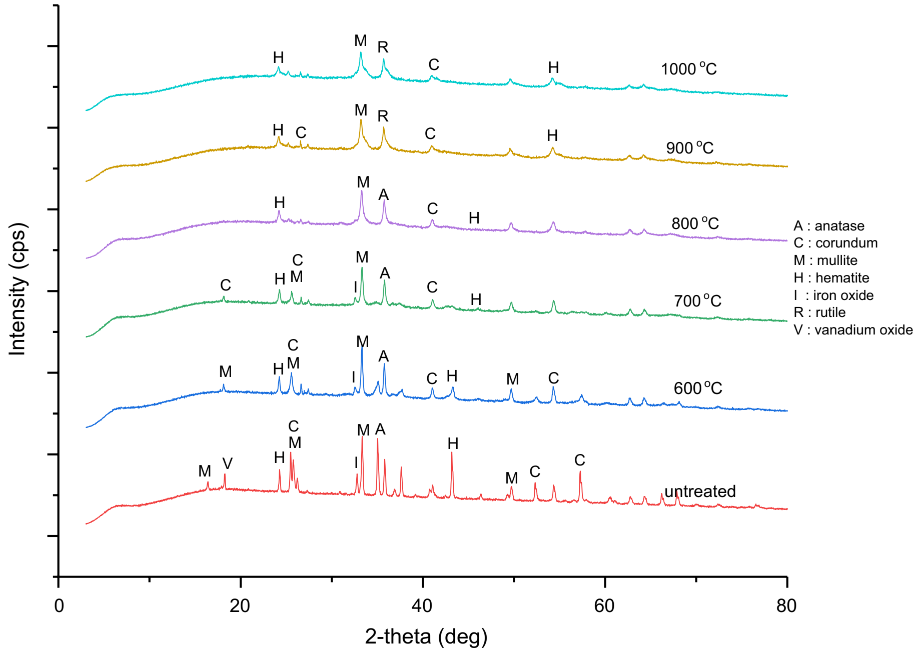


Fig. 4 XRD pattern of Malaysia's bauxite under thermal exposure

for treated bauxite, the peaks of same compounds (anatase and hematite) were detected at $2\theta = 36.4°$ and $2\theta = 24.7°$ for 600–800 °C. However, for 900 °C and 1000 °C, it can be seen that anatase has been converted into rutile at $2\theta = 23.6°$. Small changes on the crystalline structure of bauxite were detected when thermal treatment applied as can be seen in previous figure (Fig. 4). Pre-treatment at 900 °C and 1000 °C had removed vanadium (V) from the samples, and this was caused as deformation took place in the reaction [32]. These phase changes were latter affected the photocatalytic performances of the suspension as most of this reaction was responsible by anatase and hematite. As the anatase was replaced by rutile, the catalytic reaction was decreased due to larger band gaps compared to rutile TiO_2. It reduced the light that can be absorbed and move up the valence band maximum to higher energy levels relative to redox potentials of adsorbed molecules. This increases the oxidation power of electrons and facilitates electron transfer from the TiO_2 to adsorbed molecules

Figure 5 demonstrates the particle size distribution (PSD) of untreated and heat-treated bauxite sample used in this study. Bauxite sample which calcined at 1000 °C has the highest average particle size of 929.0 nm, followed by 900 °C (923.1 nm), 800 °C (662.1 nm), 700 °C (1010.5 nm), 600 °C (1345.7 nm) and untreated bauxite (600.7 nm). However, different patterns of particle size were observed for bauxite treated at 600 °C and 700 °C. These results revealed that heat treatment gives significant effect to the particle size of bauxite as there was particle expansion took place. Thermal exposure has caused grain growth on the sample which corresponds to a large value of variance due to drastic rearrangement of particles under this condition [33].

The specific surface area of untreated bauxite was determined by N_2 adsorption–desorption isotherms shows

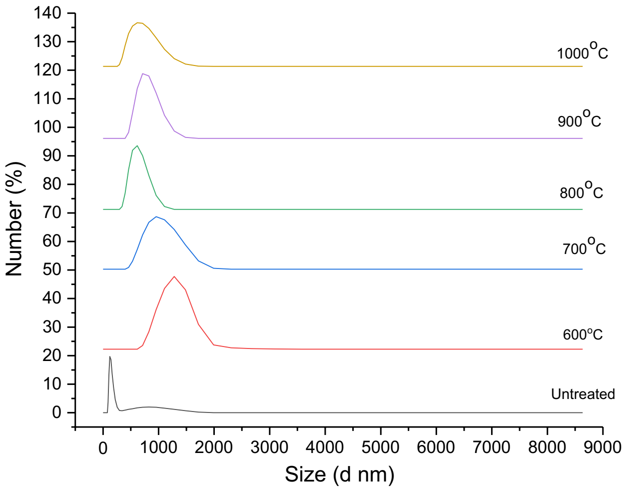


Fig. 5 Particle size analysis of bauxite

that the BET specific surface was 18.28 m^2/g, which was slightly lower compared to commercial TiO_2 P25 (52 m^2/g) [34]. It was indicated that bauxite had slightly bigger crystallite size, which promote comparable photocatalytic active surface sites per unit catalyst mass as compared to commercial TiO_2.

The obtained band gap energy of the bauxite for this study was 2.04 eV. This small band gap was obtained due to the existence of heterogeneous photocatalyst [35] of TiO_2, ZnO and Fe_2O_3 which latter contribute to the length gap between conduction and valence band [36]. As a semiconductor with relatively narrow band gap (possesses band gap energy less than 3.0 eV), it indicates that bauxite was able to conduct photocatalytic action under both UV and visible light irradiation in which this property was needed to be a good photocatalyst [37]. Narrow band gap energy also leads to a fast recombination of photoexcited electron–hole pairs which later on enhance the ability of catalyst for degradation [38].

3.2 Photocatalytic Degradation of Reactive Textile Dye (RB5)

The photocatalytic degradation of RB5 solution in synthetic wastewater has been conducted to evaluate the photocatalytic efficiency of bauxite under UV and visible light irradiation. Prior to photocatalytic measurement, the adsorption capacity of the bauxite was evaluated via adsorption test of RB5 in aqueous solution.

This study was conducted to demonstrate the photocatalytic degradation curves of various concentrations of RB5 under UV and visible light irradiation in the presence of bauxite as photocatalyst. Generally, as the exposure period prolonged, concentration of RB5 decreased which showed that photocatalytic reaction took place.

Under visible light condition as shown in Fig. 6, approximately 67%, 27% and 14% of 5 ppm, 10 ppm and 20 ppm of RB5 were degraded, respectively, within 360 min of exposure. This rate of degradation proved that bauxite was able to lower down or decolorize RB5 from its original concentration and thus revealed that photocatalytic reaction had took place. The existence of heterostructure photocatalyst in bauxite allows fast electron transport and decreases the electron–hole recombination rate [39].

The pattern of degradation of RB5 under UV light was similar to the degradation under visible light (percentage). Within 360 min of exposure to UV light, 5 ppm, 10 ppm and 20 ppm of RB5 were having reduction in concentration up to 63%, 24% and 13%, respectively, as shown in Fig. 7. As exposed to visible and UV light irradiation, it seems that the rate of degradation of the dye was comparable and less significant difference was detected for both conditions. This occurrence happened because the capacity of the semiconductor was analogous as bauxite possesses uniqueness in which there was combination of several catalysts in a single compound. Low concentration of dye showed high degradation rate as it was easily degraded due to less amount of complex structure. The findings of this photocatalytic degradation of RB5 by bauxite were in agreement

Fig. 6 Degradation rate of RB5 under visible light irradiation

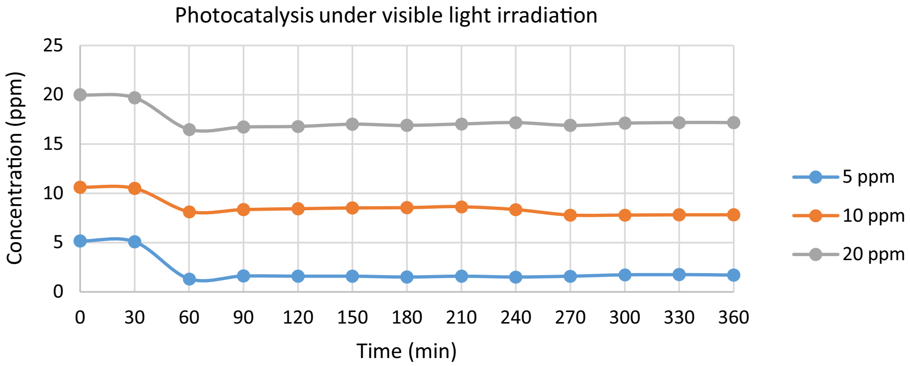
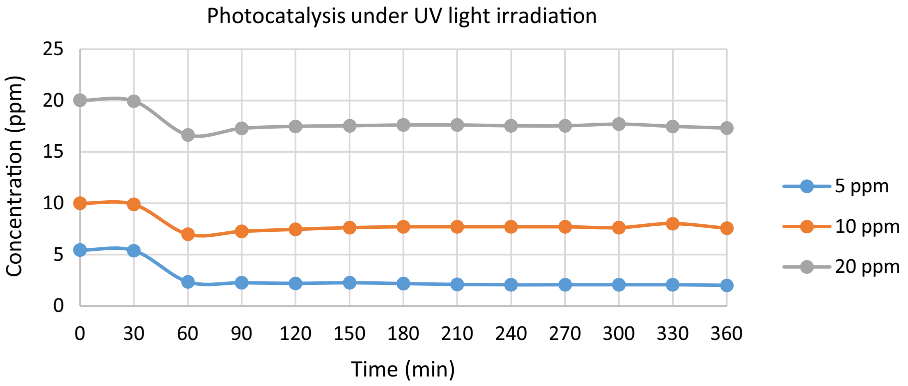


Fig. 7 Degradation rate of RB5 under UV light irradiation



with the degradation trend of methylene blue and phenol by previously reported studies [19].

In comparison with the other photocatalysts reported in the literature, Kamaludin et al. [40] reported that RB5 can be degraded up to 88.4% under UV light using n-doped TiO_2 with incorporation of visible-response substance and only 60.16% RB5 degradation was achieved using nanotitanium dioxide before addition of cavitation [19]. On the other hand, the combination of several substances and the presence of additional chemical were required to enhance photocatalytic reaction, for instance, GO-MnO_2 nanocomposite presented a high catalytic activity for the degradation/oxidation of RB5 [25] and the presence of H_2O_2 could increase the RB5 degradation as it could react with Mn ions to yield hydroxyl radicals which are able to attack and decompose the dye molecules [26]. Contrarily, for bauxite used as catalyst in this study, it can degrade up to 67% under visible light condition without any modification and this proved that this catalyst was worthy to be used as it is.

The effect of heat treatment on bauxite samples corresponding to the efficiency of degradation percentage is illustrated in Fig. 8a, b for visible and UV light condition. When exposed to visible light, the rate of degradation recorded was 69% at 600 °C, 70% at 700 °C, 75% at 800 °C, 39% at 900 °C, 35% at 1000 °C and 64% for untreated sample, respectively. On the other hand, RB5 was degraded up to 72% at 600 °C, 70% at 700 °C, 76% for 800 °C compared to 75% for untreated bauxite, 42% at 900 °C and 37% at 1000 °C under UV light irradiation.

Bauxite treated at 800 °C gives the highest photodegradation activity of RB5 compared to the other temperatures due to its smaller particle size after heat treatment as shown in Fig. 5. As the particle size becomes smaller, it promotes maximum formation of hydroxyl radical active sites between the catalysts [41]. Besides, the higher reaction between the organic molecules of RB5 and the hydroxyl radicals in bauxites and increasing collision frequency of molecules contributed to highly reactive electron under light excitation [42]. This finding was correlated to the changes of composition of the bauxite due to thermal condition as well as phase conversion of the bauxite. The formation of metakaolinite in the bauxite took place within 700–800 °C led to a wide peak of boehmite [43], and this attribute to the crystallinity of the bauxite and enhanced efficiency of the catalyst during photoreaction activity [44]. Other than that, the presence of anatase also gave positive response to this reaction due to its surface properties and charge transport [45].

The performance of photocatalysis was inversely proportional to the thermal exposure as the composition of the bauxite facing significant changes due to temperature-involved reaction. The changes might be due to the grain growth of the particle under high temperature and major

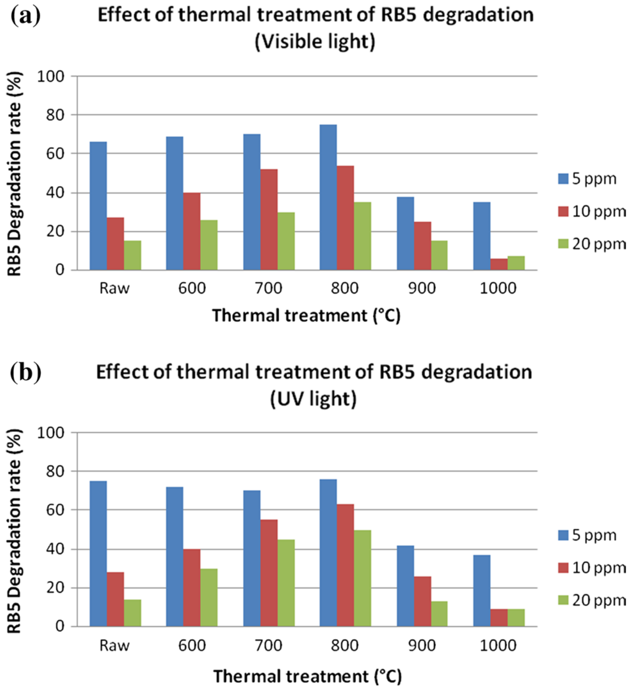


Fig. 8 Degradation rate of different concentrations of RB5 (5, 10 and 20 ppm) on the bauxite with thermal treatment (600–1000 °C) under **a** visible light and **b** UV light exposure

loss mass attributed to dehydration. Low degradation rate of RB5 when high heat-treated samples used might be due to the presence of inhibition of photo-corrosion on the surface of catalyst during photocatalytic reaction [45].

4 Conclusion

Natural bauxite from Malaysia contains heterogeneous photocatalyst and can be nominated as new and good potential catalyst for photocatalytic degradation of organic contaminants as it was able to work effectively under both visible and UV light conditions due to low band gap energy. Thermal treatment on the bauxite has affected the phase composition, and these changes attribute to the performance efficiency of the photocatalyst. 800 °C was an optimum heating temperature since up to 75% of RB5 was degraded using this sample after 360 min of exposure under both visible and UV light irradiation.

Acknowledgements The authors gratefully acknowledge financial support from the Ministry of Education, Malaysia, under the Higher Institution Centre of Excellence Scheme (Project Number: R.J090301.7846.4J193) and Universiti Teknologi Malaysia under Research University Grant Tier 1 (R.J130000.7746.4J230). The authors would also like to thank Research Management Centre, Universiti Teknologi Malaysia for the technical support.

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