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DESIGN OF THERMAL EQUIPMENT MILLING FOR FABRICATING THE TIO2 PHOTOCATALYSTS COATED GRAIN POLYMERS

RANCANG BANGUN PERALATAN THERMAL MILLING UNTUK PABRIKASI BULIR POLIMER BERLAPIS FOTOKATALIS TIO2

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ABSTRAK

Penjernihan air limbah organik dapat dilakukan dengan teknik fotokatalisis. Agar aktivitas fotokatalitik yang melibatkan faktor intensitas cahaya dan jumlah katalis dapat berlangsung secara optimal, pelapisan material katalis pada permukaan material penyangga berupa bulir polimer termoplastik dapat menjadi salah satu alternatif. Pengaturan temperatur dan lamanya pemanasan secara otomatis sangat efisien dalam mengendalikan karakteristik fisis material fotokatalis Dalam penelitian ini dilakukan modifikasi terhadap dua jenis peralatan thermal millng, yaitu peralatan milling cylinder yang dilengkapi dengan komponen pemanas dan peralatan miling berbasis oven listrik. Proses pengujian milling dilakukan dengan temperatur terkontrol pada kisaran 110 °C dan pengaturan timer 60 menit. Dalam pengujiankarakteristik termal kedua alat tersebut, dibutuhkan waktu selama 220 menit untuk setiap proses imobilisasi menggunakan milling cylinder dan 65 menit bila menggunakan peralatan berbasis oven listrik. Pengaturan temperatur dan waktu pada alat berbasis oven listrik dapat dilakukan secara otomatis, yang tidak dapat dilakukan menggunak milling cylinder. Peralatan milling berbasis oven listrik ini juga telah digunakan dalam pemilihan polimer penyangga material fotokatalis $TiO2$ serta pabrikasi fotokatalis $TiO2$ yang diujikan dalam fotodegradasi senyawa organik metilen biru (MB).

ABSTRACT

Organic waste water treatment can be conducted with technique of photocatalytic. Photocatalytic activity involves factors light intensity and amount of catalyst. In order this process can take place optimally, the catalyst material coating on the surface of the material buffer such as polymer shaped grains can be an alternative method. Setting the temperature and the duration of heating automatically is very efficient in controlling the physical characteristics of the photocatalyst materials. In this experiment, modification done on of the two types of thermal equipment milling namely cylindrical equipment milling equipped with heater and equipment milling based electric oven. The testing process of the thermal equipment milling performed with controlled temperature in the range of 110 ° C and setting the timer to 60 minutes. In testing the thermal characteristics of milling equipment, it takes as long as 220 minutes for each immobilization process using cylindrical milling and 65 minutes when using an electric oven. Setting the temperature and time in the electric oven milling can be performed automatically, which can not be performed using cylindrical milling. Milling equipment based electric oven has also been used in the selection of buffer polymer materials and fabricate $TiO2$ photocatalysts which tested on photodegradation of organic compound of methylene blue (MB).

Keywords: polymer shaped grains, TiO2 photocatalysts, thermal equpiment milling .

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INTRODUCTION

Pollution on water bodies by organic substances which derived from the textile industry became the main reason for the development of a wide range of water purification techniques (Harush, et al., 2011; Borchate, et al., 2012; Lotfy, et al., 2012; Sala & Bouz´an, 2012; Qin, et al., 2009; Liang, et al., 2008). One of them is a photocatalytic process that use TiO_2 semiconductor materials as a catalyst materials. This material is subjected to sunlight as a source of photons. This technique has several advantages namely can be applied directly to water bodies without requiring any special installation, has an strong oxidation properties, not form new toxic compounds, has a stable chemical bond to light and insoluble in water (Siddique, et al., 2011; Zahraa, et al., 2006; Mahne, et al., 2012).

Photocatalytic process takes place when a semiconductor catalyst materials induced by light as a source of photons. Light with photon energies greater than or equal to the band gap energy of the catalyst materials will result in the excitation of electrons to the conduction band, leaving holes in the valence band. Electrons and holes then reacts with radicals in water forming free radicals that play an important role in the degradation of organic compounds in water (Fujishima, et al., 2000; Moghaddam & Yangjeh, 2011).

Generally, using TiO_2 catalyst in the process of water treatment carried by inserting catalyst powder into the contaminated water and exposed by sunlight. This method is not efficient because TiO_2 powders have a large density and small size.

The process of photodegradation at the organic pollutants highly dependent on the light intensity of the photon source and the amount of catalyst. (Rashed and El Amin, 2007; Rajeswari and Kanmani, 2009). To optimize the photocatalytic activity that involves both of the above factors, it is very important to optimize the TiO_2 coating on the surface of the transparent polymer. Two parameters that become the focus of our attention are the masses attached TiO_2 and transmittance of polymers.

The immobilization on TiO_2 catalyst can be performed on supporting materials such as polymer-sized, lightweight, transparent and thermoplastic. (Isnaini, et al., 2011; Arutanti and Abdullah, 2009). With thermoplastic properties on polymer allows the catalyst materials attached to the polymer surface when heating around the point of Heat Deflection Temperature (HDT) of polymer. Thus, immobilization of the catalyst on the surface of polymer materials can be done through thermal milling techniques.

In principle, this technique involves the process of mixing materials and giving thermal energy. In thermal immobilization mechanism, polymer surface will be softened, so that the a number of TiO_2 catalyst can be attached to the polymer surface. Mass of TiO_2 dan trasmittance of polymer can be controlled by adjusting operating parameters such as temperature and duration during the milling process.

In this paper, we will assess two type of milling system modifications, namely the modification of cylindrical milling equipment with heater and milling equipment with electric oven. It is done in order to the temperature and duration of immobilization can be well controlled.

METHOD

This research uses two types of milling equipment, namely the cylindrical milling with heater and equipment milling using electric oven. A cylindrical tube in milling tool acts as container to mix the catalyst materials and buffer materials. This tube connected to the motor that rotates at a constant speed during the immobilization process in progress. Inside the tube, there are two non-magnetic stainless steel cylindrical rods that acts as a stirring bar and give the effect of of collisions on the mixture of catalyst and polymer.

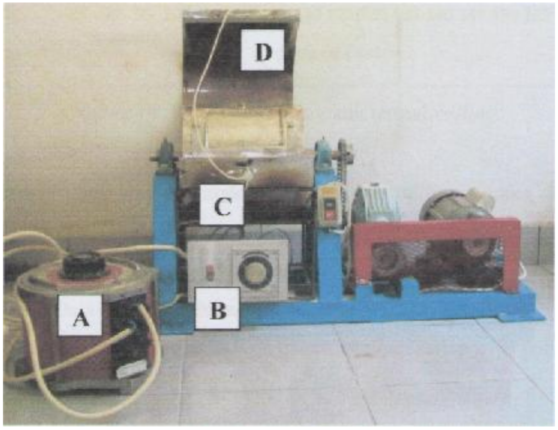

Figure 1. Cylindrical milling systems equipped with a heater circuit: A. Variac; B. temperature controller; C. thermocouple; D. slab of stainless steel (Amalia, et al., 2011).

For controlling the heating temperature during immobilization process, cylindrical milling connected with heater that integrated with temperature controller and a voltage regulator (variac). Cylindrical milling, heater, variac as well as temperature control was shown in Figure 1. It is difficult to control the temperature during immobilization process TiO_2 on the surface of the polymer. This is a major reason why it is necessary to modify the system simpler.

Electric oven can be modified to become a simple milling tool. Electric oven which used having the shaft in the center of the oven that allows rotisserie can be rotate. This research used an electric oven type KBO 190 RAW merk Kirin with specifications as summarized in Table 1.

Table 1. Spesification of Electric Oven with Kirin Merk *KBO 190 RAW Type*

Electric voltage	220 volt
Power consumption	400 – 800 watt
Capasity	19 litres
Dimension	(495 ´ 363 ´ 346) mm
Temperature maks	250 °C
Maximum *timer*	60 minutes

Electric oven is complemented with a pair of heater plates in the oven on the top and bottom can be arranged. In addition, the oven is equipped with temperature controller and timer, so that the temperature and the duration of heating can be set automatically.

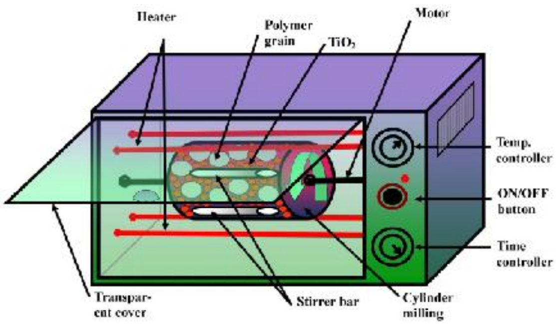

Figure 2. Components of the electric oven which play an important role in the milling process (Aliah, 2012)

The main components of the electric oven that play a role in the milling process were shown in Figure 2. The testing process on the electric oven was done by controlling the temperature in the range of 110 ° C and the timer is set to 60 minutes. The real oven temperature was measured using an infrared thermometer.

After comparing the efficiency of setting the temperature and time of milling, a more efficient system will be used in further research. Several studies have been reported related to this issue, including the selection of polymer materials as a buffer materials on TiO_2 catalyst,optimization of temperature and time milling on the photocatalyst fabrication and testing of photodegradation on organic compounds of methylene blue (MB) using TiO_2 material

RESULT AND DISCUSSION

Figure 3 displays the thermal characteristic curve of cylindrical milling. The graph shows the relationship between the input voltage and the temperature of the cylinder milling equipment. Heater temperature on the thermostat is set at 200 ° C and the elevated input voltages of 10 volts every 5 minutes. With knowing this thermal characteristic curve, the milling process on the desired temperature can be done simply by adjusting the input voltage on Variac.

As well as conventional cylinder milling systems that use a gas stove as a heater, cylindrical milling that modified by adding heater circuit is also still leaves several problems. The addition of heater on cylindrical milling cannot guarantee the immobilization process takes place at a controlled temperature. Moreover, it takes a long time on each immobilization process. In one of the experiment the testing this system, the milling process is carried at a temperature of 110 ° C for 60 minutes.

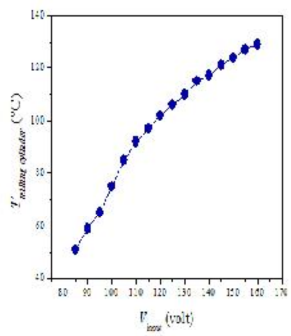

Figure 3. Thermal characteristics of cylindrical milling equipped with heater circuit.

Based on the thermal characteristic curves in Figure 3, is required the input voltage of 130 volts so that milling cylinder temperature reaches the range of 110 ° C. To determine the consistency of temperature, observations was carried by measure the temperature as a function of time. The equipment was controlled by setting parameter input voltage of 130 V and a temperature of 110 ° C. Results obtained are shown in Figure 4.

By using this cylinderl milling, it takes 90 minutes to allow the the milling cylinder reaches a temperature of 110 °C. In addition, the milling temperature increased for 60 minutes the milling process. The cooling process ongoing much longer, takes 70 minutes to reach a room temperature. Thus, it took 220 minutes for each immobilization process using the milling cylinder.

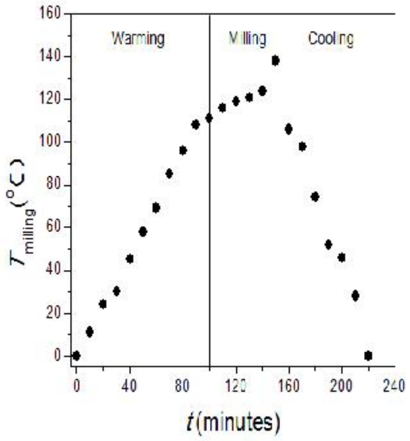

Figure 4. Thermal condition of milling cylinder as time function (V_{input} = 130 V; $T_{milling}$= 110 °C; $t_{milling}$= 60 minutes).

Figure 5. Milling cylinder based electric oven: a. electric oven, b. electric oven with milling tubes that can be rotates in the middle of the oven, c. milling cylinder.

A modification on electric oven is done by adding a hollow cylinder in the center of the oven. This cylinder serves as the place of mixing process the material. This cylinder is equipped with two cylindrical solid rod therein. Electric oven, cylindrical stirrer and stirrer cylinder placement in the electric oven is shown in Figure 5.

In the process of immobilization, a cylindrical tube mounted on the tool holder roast. The holder connected to an electric motor that can rotate at a constant speed. Temperature settings at once time setting automatically becomes the main advantages of this device, so that the immobilization process can be well controlled.

The use of modified electric oven can improve temperature control system on the process of immobilization. In addition, the time required to reach the desired temperature and the duration of the cooling process after milling taking place with a fairly short. Temperature conditions as a function of time is shown in Figure 6.

In this test, only takes about 5 minutes to raise the temperature in the oven from room temperature to the desired temperature (110 ° C). As long as the following 55 minutes, the temperature of milling cylinder fluctuated between 110-114 ° C. After the time reached 60 minutes, the oven automatically power off which indicates the start of the cooling process in the oven chamber. Within 5 minutes, the oven temperature back to room temperature. Thus, it takes only about 65 minutes total time for this immobilization process (Aliah, 2012).

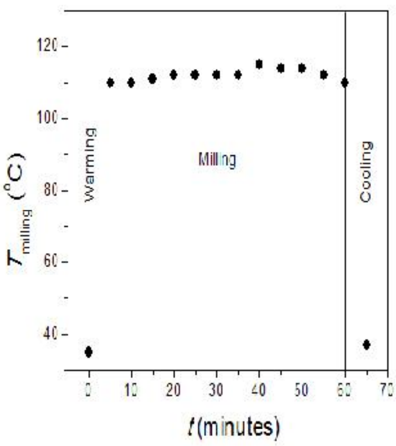

Figure 6. The temperature of electric oven modified as a function of time in the process of milling ($T_{milling}$ = 110 ° C; $t_{milling}$ = 60 minutes).

Based on description above, the modified electric oven into the milling equipment can overcome the problem of controlling the temperature and time milling. Automatic control is expected to improve the quality of the attachment of the catalyst on the surface of the polymer. Catalysts are expected having good transmittance and the amount of catalyst on surface of polymer quite a lot to decompose organic pollutants compounds.

Table 2. Density and transmittance of polymer before and after coated with TiO_2

Polymers	Density (g/cm^3)		Transmittance (%)	
	Without TiO_2	With TiO_2	Without TiO_2	With TiO_2
PS	0.950	1.081	74	61
LLDPE	0.920	0.980	56	52
PP	0.855	0.872	62	58

The modified electric oven was have used in choosing the appropriate thermoplastic polymers as a buffer materials for TiO_2 photocatalyst. In this research, we choose three kind of thermoplastic polymer, namely polysterene (PS), low lower density polyethylene (LLDPE), and polypropelene (PP). The selection of polymer refers to the properties of polymer, namely lightweight, transparent, and thermoplastic. The density and the transparancy of the three types of polymer thermoplastic polymer become a reference in the selection the buffer of TiO_2 catalyst.

Based on data in Table 2, from the three types of polymers which were tested, PP polymers provide optimum results, in which its density low enough so floats well on the water surface and shows good transmittance properties (Aliah, 2012). Thus, the polymer PP has great potential to be used as a buffer materials for TiO_2 photocatalyst.

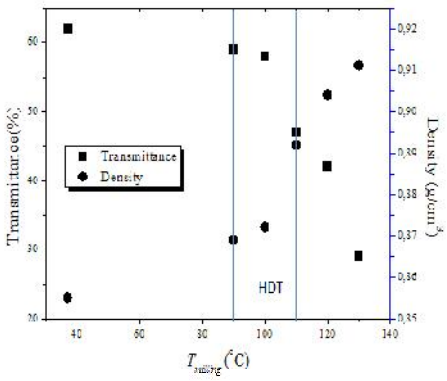

Figure 7. Effect of temperature on the transmittance and the density of the PP-coated TiO for 60 minutes milling process (Aliah, et al., 2012)

In order to obtain the optimum operating temperature on the modified electric oven, the milling temperature optimization experiments had been conducted. Optimization carried out by varying the temperature in the range 90 ° C to 130 ° C at the milling process for layering TiO_2 catalyst on the surface of the polymer PP. Based on the values transmittance and the density PP-coated TiO_2, obtained a graph of temperature milling with both variables as shown in Figure 7.

Heat deflection Temperature (HDT) PP polymers in the range of 100-115 ° C. We can predict that the optimum temperature is around the point of the HDT. This prediction is confirmed by the test data photodegradation of photocatalyst TiO_2 materials which has been coated on surface of polymer PP with temperatures varying between 90 °C to 130 °C. In Figure 8, we know that the test sample using a polymer coating layered with a temperature of 100 ° C resulted in the highest reaction rate constant. In other words, the optimum milling process taking place at a temperature of 100 °C. These data are relevant to the data the transmittance and the density of the shown in Figure 7.

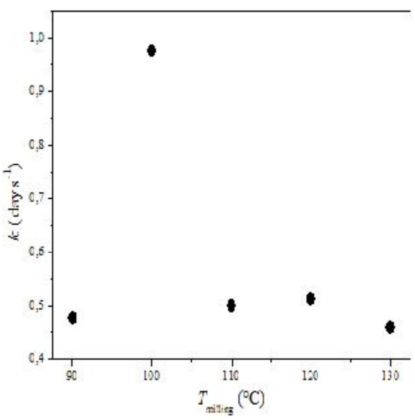

Figure 8. Effect of temperature on the reaction kinetic of photodegradation PP-coated TiO_2

Furthermore, optimization of the duration of milling carried out by varying milling time between 0 to 120 minutes at a temperature of 100 °C. The influence of milling duration on the transmittance and the density were shown in Figure 9. The optimum conditions predicted to occur in the operation of milling with the duration for 90 minutes on a milling temperature of 100 °C. In this condition, the transmittance and the density of the grains PP-coated TiO_2 were respectively by 56% and 0.884 g cm^{-3}. This prediction is also reinforced by the value of the reaction kinetics on photo degradation of organic compounds.

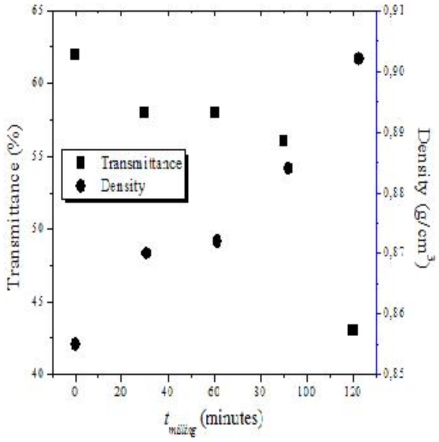

Figure 9. The effect of the duration of milling on the transmittance and the density of the PP-coated TiO at milling temperature of 100 °C (Aliah, et al., 2012).

The graph in Figure 10 shows the effect of duration of milling on the the reaction kinetics on TiO_2 photocatalytic process. The MB solution which tested using the catalyst with a duration of milling for 90 minutes can decompose organic compounds fastest compared to other test solutions. Thus, the use of a modified electric oven into a grinding tool to fabricate TiO_2-coated PP catalysts work optimally at temperatures of 100 ° C for 90 minutes.

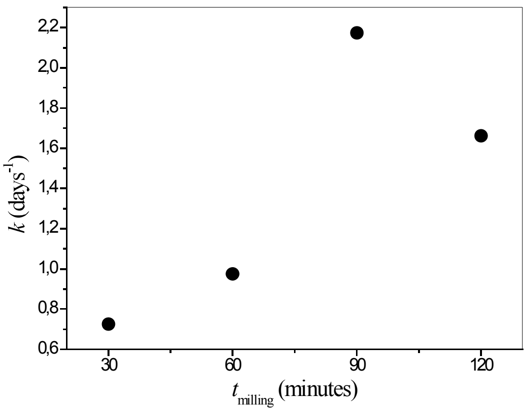

Figure 10. Effect of $t_{milling}$ on the reaction kinetics of TiO_2 photocatalytic process

Figure 11 shows the color of test solutions before and during the process of photodegradation. The reduction of MB concentrations is shown qualitatively from the reduction of the color of test solutions. Decomposition of compound MB through photocatalytic process shows the breaking of the chemical bond. A long-chain compounds derived from color form a simple chain of chemical bonds. (Aliah, 2012).

0^{th} days 5^{th} days

Figure 11. The MB test solution (from left to right) on days 0, 1, 2, 3, 4, 5 (the catalyst with $T_{milling}$ = 100 °C, $m_{milling}$ = 90 minutes, $m_{catalyst}$ = 9.0 g, $C_{0\ MB}$ = 2,60×10-5 M)

CONCLUSION

Modifications of milling tools from electric oven with Kirin Merk type KBO RAW 190 are fully integrated with temperature control and timer. It can improve the temperature control system during the immobilization process. Setting the temperature and duration of heating can automatically improve the quality of attachment of the catalyst on the surface of a thermoplastic polymer.

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