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Characterisation of Doped Titanium Dioxide (TiO_2) at Different Calcination Temperature Using X-Ray Diffraction (XRD)

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Titanium dioxide (TiO_2) is one of the most powerful semiconductors used in wastewater treatment. However, TiO_2 has a wide band gap that only can be activated by using UV light. To overcome the weakness of this photocatalyst, the doping technique by using the sol-gel method was applied to the TiO_2. The sol-gel method is easy, low cost and can be conducted at low temperature. Through this method, the band gap can be narrow and the doped TiO_2 can be activated using visible light. The objectives of this research are to study the relationship between variable parameters and the sample characterisations, to analyse the characteristics of modified photocatalysts doped nitrogen and magnesium on the phase. In this research project, nitrogen and magnesium will be used for doping techniques with TiO_2. Calcination temperature will be varied at 300°C, 500°C and 700°C to analyse the phase and characteristics of samples at different calcination temperatures. Parameters that are involved in this research include the amount of doping which is varied at 0.5 wt.%, 0.7 wt.% and 0.9 wt.% and type of dopant used (nitrogen and magnesium). The sample will be characterised by using X-Ray Diffraction (XRD).

Keywords: nitrogen; magnesium; TiO_2; XRD

I. INTRODUCTION

Titanium dioxide is made up of TiO_6 octahedral. This arrangement of octahedral makes the structure consists of three different polymorphs which are anatase, rutile and brookite (Nolan *et al.,* 2011). The rutile part is thermodynamically stable, while anatase and brookite are categorised as metastable. In photocatalysis, TiO_2 is often used as the catalyst due to the cheap raw materials, easy to obtain and also environmentally friendly. No side product will harm the environment discharged from the process as semiconductor catalysts such as TiO_2 and ZnO have been widely used to mineralise harmful organic pollutants in wastewater into harmless inorganic non-toxic compounds such as carbon dioxide (CO_2), hydrochloric (HCl) and water, thus it is very recommended to be applied in the wastewater treatment process (Mondal & Sharma, 2005).

Despite all the advantages of this photocatalyst, titanium dioxide is capable to act as a sensitiser for the light which will reduce the redox process due to its electronic structure. The best part is that the TiO_2 can be chemically activated by light, thus in the industry, water treatment, air-cleaning or self-cleaning for the building often used TiO_2 which helps the cleaning process become easier.

However, TiO_2 only can be applied when the Ultra Violet (UV) light is present to activate the electrons because of the high band gap energy (3.2 eV), which will cause the process to become very costly (Sikirman *et al.,* 2014). In that case, the band gap energy needs to be narrowed to enable the process to operate under visible light ($400\ nm < \lambda < 500\ nm$) by modifying the photocatalyst. One of the easiest ways to reduce the band gap energy is by using dopants with suitable elements. There are other ways including sputtering, ion implantation, pulsed laser deposition, hydrothermal and solvothermal synthesis but the sol-gel method will be chosen as the method is easy to apply than the others as the process only needs to operate at a low temperature normally less than

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700 °C and low cost involved.

A non-metal dopant, for example, nitrogen is considered the most effective dopant to be incorporated with TiO_2 as the size is smaller and the source is cheaper. Besides, much research showed a great result when nitrogen-doped TiO_2 is used on methylene blue and methyl orange. Thus, in this study, nitrogen-doped TiO_2 will be applied to Reactive Black 5 dye and the result will be analysed (Segne *et al.,* 2011). Some researchers believed that the nitrogen atoms substitute the oxygen atom in the lattice of the TiO_2 molecule (Nolan *et al.,* 2011). Magnesium is one of the abundant alkaline earth metals and is less hazardous to be used in the water treatment process which will cause a more environmentally friendly process. Magnesium is a type of metal dopant and a study of doping with magnesium ions that have a nearly equivalent atomic radius to titanium, may give a better insight into doping process in detail and the effect of dopant size on the photocatalytic activity. Besides, inserting metal ions into the TiO_2 structure can decrease the band gap and from the research, it is found that metals can lower the electron-hole recombination rate and trap the electrons (Behnajady & Tohidi, 2013).

II. MATERIALS AND METHOD

In this research, Titanium (IV) isopropoxide (TTIP) and Reactive Black 5 with dye content ≥50% were bought from Sigma-Aldrich. Magnesium Chloride 6 Hydrate from Bendosen and Ammonium Nitrate 99% from Emory. While Ethanol 95%, a laboratory grade from HmbG and Acetic acid glacial 100% from Emory were used.

Initially, 45 ml of deionised water and 5 ml of acetic acid were mixed and labelled as solution A. Solution B was prepared in another beaker by diluting 15 ml of Titanium (IV) isopropoxide (TTIP) with 5 ml of ethanol. Solution B was added dropwise into solution A for 30 minutes. Different amount of magnesium chloride and nitrogen nitrate was added into solution B to vary the weight percentage of magnesium and nitrogen as the dopants. Next, the solution was centrifuged for 15 minutes at 9000 rpm and the liquid layer was removed and sol-gel was obtained.

The sol-gel was left at room temperature for 12 hours as a pre-ageing process before ageing in the oven at a temperature of 110 °C for 17 hours to remove excess solvents. The powder obtained was grounded finely and calcinated in a box furnace (Model 524120-P) at 300 °C, 500 °C and 700 °C for 1 hour for heating treatment to complete the preparation of the photocatalyst. Then all the samples were analysed using XRD. Dye samples were taken at every 15 minutes interval time. All the samples are labelled as 0.5 wt. % Mg-TiO_2, 0.7 wt.% Mg-TiO_2, 0.9 wt. % Mg-TiO_2, 0.5 wt. % N-TiO_2, 0.70 wt. % N-TiO_2 and 0.9 wt.% N-TiO_2.

All the samples were characterised and analysed to identify the phase structure using X-ray Diffraction (XRD) (Bruker D8 Advance Machine). All the results and data were recorded and tabulated.

III. RESULTS AND DISCUSSION

The phase composite and estimation of crystallite size for all samples were analysed and evaluated by using X-ray diffraction (XRD) analysis.

After the XRD analysis is done, the results will be analysed and the estimated crystallite size of all samples (0.5wt.% N-TiO_2, 0.7wt.% N-TiO_2, 0.9wt.% N-TiO_2, 0.5wt.% Mg-TiO_2, 0.7wt.% Mg-TiO_2 and 0.9wt.% Mg-TiO_2) at different calcination temperature of 300 °C, 500 °C and 700 °C calculated by using the Scherer's equation as follows:

$$D = K\lambda/\beta\cos\theta \quad (1)$$

Where, K=0.9 is the Scherer constant, λ = 0.15406 nm is the X-ray wavelength, β as the peak width of half maximum in radian, and θ is the Bragg diffraction angle. The rutile and anatase phase content (%) can be calculated by using the equation;

$$\% \text{ Rutile phase} = \frac{1}{[\left(I_A/I_R\right)0.884+1]} \quad (2)$$

$$\% \text{ Anatase phase} = \frac{1}{[\left(\frac{I_R}{I_A}\right)1.26+1]} \quad (3)$$

I_A refer to the intensity of the strongest anatase reflection. While I_R is the intensity of the strongest rutile reflection.

Table 1. The particle size of samples at different calcination temperature for N-TiO_2 and Mg-TiO_2

Samples name	(wt.%)	Calcination Temperature (°C)	Anatase Phase Content (%)	Rutile Phase Content (%)	Estimated Anatase Crystallites Size (nm)	Estimated Rutile Crystallites Size (nm)
N- TiO_2	0.5	300	100	-	7.57	-
	0.7	300	100	-	11.43	-
	0.9	300	100	-	7.54	-
	0.5	500	100	-	12.80	-
	0.7	500	100	-	10.41	-
	0.9	500	100	-	15.71	-
	0.5	700	75	25	55.56	118.57
	0.7	700	69	31	41.92	71.23
	0.9	700	80	20	55.56	89.02

Samples name	(wt.%)	Calcination Temperature (°C)	Anatase Phase Content (%)	Rutile Phase Content (%)	Estimated Anatase Crystallites Size (nm)	Estimated Rutile Crystallites Size (nm)
Mg- TiO_2	0.5	300	100	-	7.56	-
	0.7	300	100	-	13.89	-
	0.9	300	100	-	6.94	-
	0.5	500	100	-	13.94	-
	0.7	500	100	-	27.69	-
	0.9	500	100	-	12.79	-
	0.5	700	96	4	41.67	89.01
	0.7	700	82	18	41.59	59.35
	0.9	700	97	3	41.67	29.67

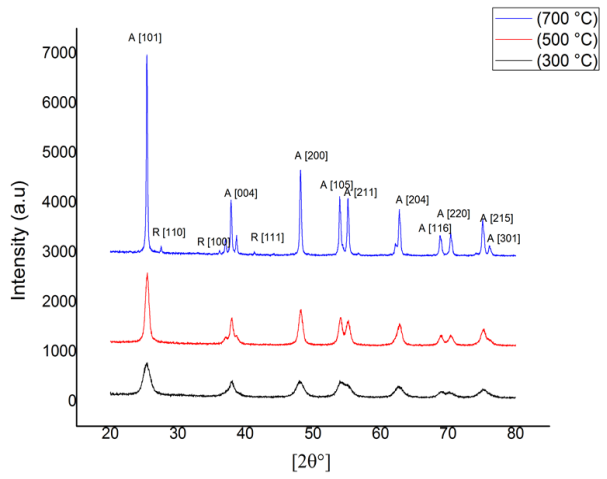

Figure 1. Comparison XRD analysis of sample 0.5wt. % Mg-TiO_2, prepared at (a) 300 °C, (b) 500 °C, and (c) 700°C

Calcination temperature can affect the physical properties of the gels by polymerisation, coarsening and phase transformation (Hsiang & Lin, 2007). Figure 1 showed the XRD analysis of sample 0.5wt. % Mg-TiO_2, prepared at varying temperatures from 300 °C to 700 °C. From the analysis, it can be seen that at temperatures 300 °C and 500 °C, only a single anatase phase occurred and at 700 °C, the peaks of rutile can be observed. A study conducted by Kumar *et al.* (2016), found that at a lower temperature (300 °C -500 °C) only the anatase phase was observed and at 600 °C the rutile peaks started to appear. Peaks at a calcination temperature of 300 °C showed wider than the peaks at a temperature of 500°C and 700 °C. The anatase phase can be observed at peaks of 2θ = 24.40°, 38.00°, 48.00°, 54.00°, 62.90°, 69.00°, and 75.14°. As the calcination temperature was increased up to 500 °C, the peaks of the anatase phase become defined and can be observed especially at 2θ = 54°and 55.12°, also at 2θ =68.9°and 70.4°. The peaks of the rutile phase can be observed at 2θ = 27.45°, 36.09°,

41.23°, 54.32°, 56.64°, 62.74°, 64.04°, 69.01° and 69.79°. From the analysis, it also can be seen that the peaks at 700 °C become narrower compared to peaks at 300 °C and 500 °C. This is because of the improvement of TiO_2 crystallinity associated with the anatase crystals' growth. According to Mozia (2008), during the heat treatment of TiO_2, dehydration takes place and as a result, the crystals grow to a size larger than those of the original particles. Besides, the increase of the heat treatment temperature up to 700 °C led to the phase transformation from thermodynamically metastable anatase to the most stable form of TiO_2, rutile. The estimated crystallite sizes can be calculated by using Scherer's equation (Equation (1)) and the percentage of anatase and rutile phase can be found by applying Equations (2) and (3). From table 1, the estimated crystallite size for the sample is 0.5 wt.% Mg-TiO_2 at 300 °C has the smallest size compared to 500 °C and 700 °C which are 7.56 nm, 13. 94 nm, 41.67 nm (anatase) and 89.01 nm (rutile), respectively. The size of crystallites increases as the calcination increases which coincides with the statement made by Mozia (2008) and Aphairaj *et al.* (2011). At heat treatment of 700 °C, the samples contain 96 % of anatase and 4 % of rutile.

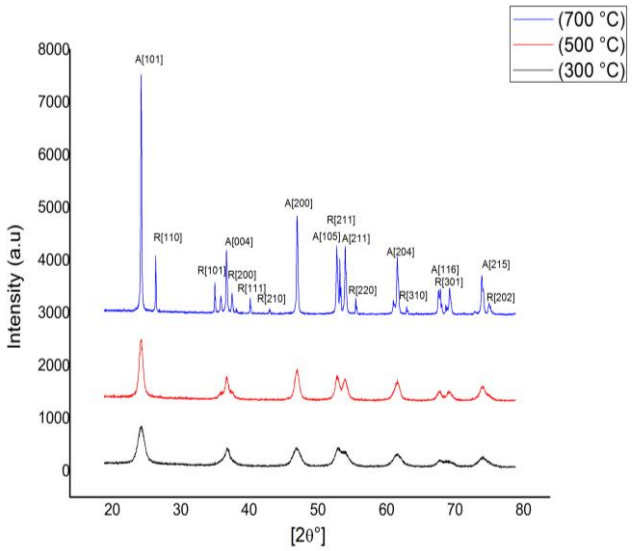

Figure 2. Comparison XRD analysis of sample 0.7wt. % Mg-TiO_2, prepared at (a) 300 °C, (b) 500 °C, and (c) 700 °C

Figure 2 showed the XRD analysis of 0.7wt. % Mg-TiO_2 prepared at 300 °C to 700 °C. There are not many differences that can be seen from the previous pattern analysis. Anatase peaks of the two different temperatures were observed at the same point as the previous sample of 0.5wt. % Mg-TiO_2. From Figure 2, it is observed that the peaks of 500 °C are defined and can be seen compared to 300 °C. It is also found that the peak was wider when 300 °C temperature was applied and become narrowed as the temperature at 500 °C and 700 °C. At 700 °C, rutile peaks can be observed clearly and the peak's intensity is higher compared to the previous samples. It can be related to the experiment conducted by Cai *et al.* (2016), in which the rutile transformation may start at 600 °C. However, the anatase and rutile content of this sample is 82 % and 18 % which showed higher rutile content compared to the 0.5 wt. % Mg-TiO_2. João Gomes *et al.* (2012) found that the rutile peaks started to appear when the calcination temperature is at 350 °C. The estimated crystallite sizes are 13.89 nm, 27. 69 nm, 41. 59 nm (anatase) and 59.35 nm (rutile), respectively for 300 °C, 500 °C and 700 °C. The same results were found by Cai *et al.* (2016), when the calcination temperature is higher (600 °C), the crystal size was found bigger compared to the lower calcination temperature (400 °C). Hanaor and Sorrel (2011) explained that during the transition of anatase to the rutile phase, significant grain growth will occur and resulting in large rutile grains and small anatase grains. At 2θ = 54.32°, a rutile peak can be observed appearing from the anatase peak at 2θ = 53.90°. This is because, the rutile crystallites may grow as an overlayer of rutile on anatase particles or the rutile may form in the bulk of anatase grains and leave a surface layer of anatase on rutile particles, and this process can be referred to as encapsulation. The peaks of rutile of sample 0.7 wt. % Mg-TiO_2 were found to have higher relative intensities compared to the samples of 0.5 wt. % Mg-TiO_2 may be due to the effects of different amounts of dopants supplied during the preparation of the photocatalyst. The photocatalytic activity is directly proportional to the dopant concentration but at a certain level, the photocatalytic activity will be decreasing due to the active site and hydroxyl ion that cause agglomeration of the catalyst.

Figure 3 showed XRD analysis for sample 0.9 wt. % Mg-TiO_2 at calcination temperature of 300 °C, 500 °C, and 700 °C. At 300 °C and 500 °C, only a single anatase phase was found. At 700 °C, the mixed phase of anatase-rutile was found. From these results, it can be said that the formation of the rutile phase started at a temperature of 600 °C. The peaks of

anatase are significantly defined at 500 °C and 700 °C, this may be attributed to the improvements in the crystallinity of the grains (Yu & Wang, 2010). The broad peaks at 300 °C were also found to be narrowed when the calcination temperature is increasing, which indicates the size of the grains becomes bigger at a higher temperature. Wetchakun *et al.* (2012), studied the influence of calcination temperature on the transformation of rutile from anatase, from the research, it was found that the temperature of anatase to rutile transformation was found between 500 °C and 600 °C. They also found that the crystallite sizes increased at a higher temperature. Yu and Wang (2009) explained that the phase transformation caused the growth of grains size due to the heat of phase transformation to accelerate the grain growth. For this sample, the estimated crystallite sizes calculated using Scherer's equation are 6.94 nm, 12. 79 nm, 41.59 nm (anatase) and 29.67 nm (rutile) for 300 °C, 500 °C and 700 °C each. From the XRD analysis, the percentage of anatase and rutile phase content was also calculated and it is found the content of mixed-phase anatase- rutile is 93 % and 3 %, respectively.

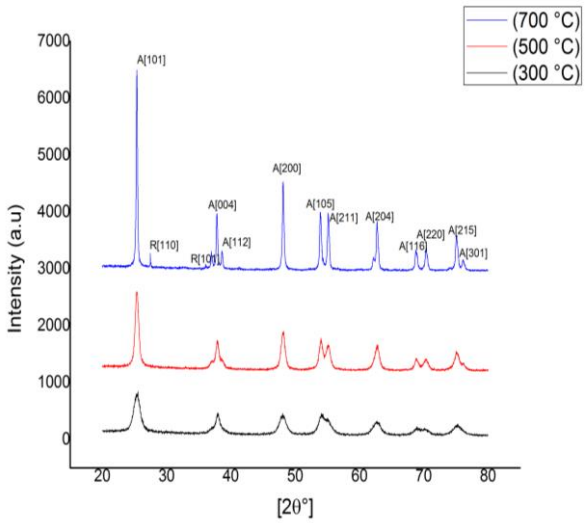

Figure 3. Comparison XRD analysis of sample 0.9 wt. % Mg-TiO_2, prepared at varying temperatures of 300 °C, 500 °C and 700 °C

Based on Figure 4, a different phase of photocatalyst can be seen on the XRD data of the photocatalyst sample with 0.5wt. % N-TiO_2 where each of them was calcined at temperatures of 300 °C, 500 °C and 700 °C respectively. From the graph, it can be seen at calcination temperatures of 300 °C and 500 °C, the anatase phase was observed and at 700 °C rutile phase can be observed from the graph. At 300 °C, the anatase phase can be seen and at 500 °C, defined peaks of anatase can be observed at 2θ = 25.40, 38.00, 48.00, 54.00, 55.20, 62.72, 68.80, 70.26 and 75.14°. As the higher calcination temperature was applied, the rutile peaks already formed at 700 °C, where the peaks can be observed at 2θ = 27.58, 36.22, 41.38, 54.46, 64.16, and 69.92°. Thus, it can be said the formation of rutile may start at 600 °C. From the XRD graph, at a lower temperature, broad peaks can be seen at 2θ = 48.00, 54.00, 55.20, 62.72, 68.8, 70.26 and 75.14°which indicates small crystallite sizes. As the calcination temperature gets higher, the peaks become narrowed and stronger intensities could be observed, which means that the crystallite sizes have grown due to the heat treatment applied and the crystallinity has improved as the calcination temperature increases. The estimated crystallite sizes were calculated and tabulated as in table 1. At 300 °C, the estimated crystallite size is 7.57 nm, at 500 °C, 12.8 nm and 700 °C, the estimated crystallite sizes are 55.56 nm (anatase) and 118.57 nm (rutile). The anatase-rutile phase content for this sample was calculated and it was found that 75 % of anatase and 25 % rutile were present in the sample.

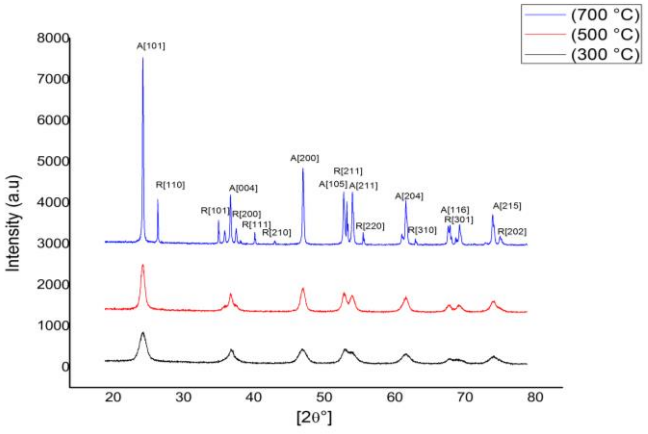

Figure 4. Comparison XRD analysis of sample 0.5wt. % N-TiO_2, prepared at varying temperatures of 300°C, 500°C and 700°C

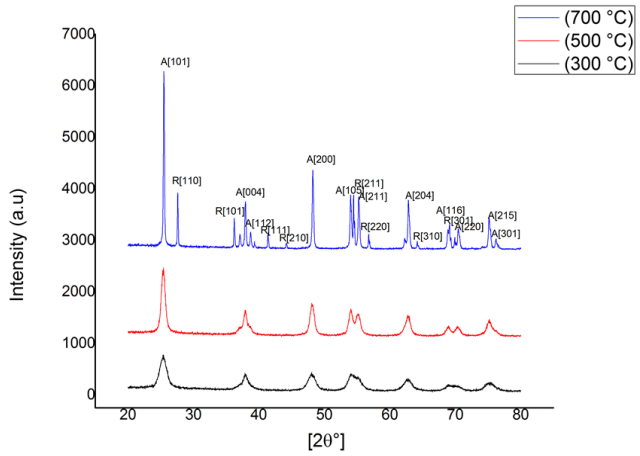

Figure 5. Comparison XRD analysis of sample 0.7 wt. % N-TiO_2, prepared at varying temperatures of 300°C, 500°C and 700°C

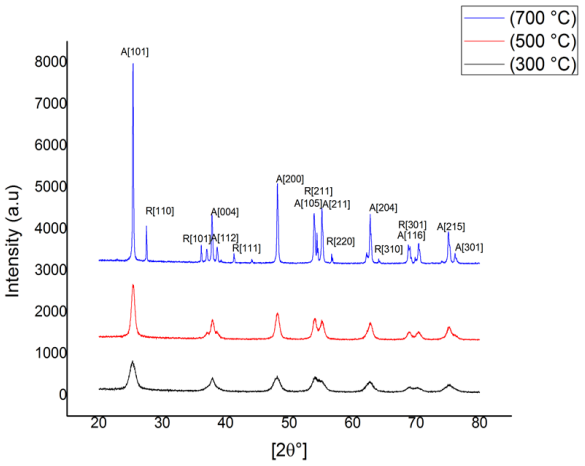

Figure 6. Comparison XRD analysis of sample 0.9 wt. % N-TiO_2, prepared at varying temperatures of 300 °C, 500 °C and 700 °C

XRD results for the sample of 0.7 wt. % N-TiO_2 (refer to Figure 5) showed almost the same result as the 0.5 wt.% N-TiO_2 where anatase phase can be seen at a calcination temperature of 300 °C and developed into defined peaks at 500 °C. The rutile phase can be seen at the calcination temperature of 700 °C. Rutile peaks can be observed clearly at 2θ = 27.45, 36.08, 41.23, 54.32, 56.64, 62.74, 64.04, 69.01, and 69.79°. The same observation as the 0.5 wt. % N-TiO_2 sample, broad peaks were found at a lower temperature (300 °C) indicating smaller crystallite sizes and narrowed peaks were observed at a higher temperature which indicated larger crystallite sizes. The estimated crystallite sizes were calculated and it is found at 300 °C, the crystallite size is 11.43 nm, at 500 °C, the crystallite size is 10.41 nm and at 700 °C, crystallite sizes for anatase and rutile are 41.92 nm and 71.23 nm respectively. It was found that the phase content of anatase-rutile is 69 % and 31 %. It was reported that the mixture phase content of anatase-rutile with 70 % and 30 % exhibited higher photodegradation performance than the P25 TiO_2 (Hanaor & Sorrel, 2011) .

Sample 0.9 wt. % N-TiO_2 were analysed using XRD and the result is shown as referred in Figure 6. The sample prepared at 300 °C and 500 °C showed that there was the presence of the anatase phase. At peaks of 2θ = 25.35, 36.88, 37.79, 48.08, 53.92, 55.12, 62.73, 68.59, 70.36, 75.01 and 76.08°, the anatase phase can be seen clearly at all calcination temperature. However, at 300 °C, the peak intensities of anatase can be observed weak indicating a lower crystallinity phase. As the calcination increased, the intensities of the peaks appeared to be stronger and defined, this may be attributed to the improvement of anatase crystallinity. The analysis also showed that when calcination temperature is at 300 °C, the peaks are wider compared to the peaks at a calcination temperature of 500 °C and 700 °C which indicates that the crystallite size becomes bigger as the temperature increases. The estimated crystallite sizes were calculated for temperatures 300 °C, 500 °C and 700 °C which are 7.54 nm, 15. 17 nm, 55.56 nm (anatase) and 89.02 nm (rutile), respectively. For this sample, the anatase phase content is 80 % and the rutile phase content is 20 % which is similar to the sample of 0.7 wt. % Mg-TiO_2 and the commercial P25 TiO_2 (Hanaour & Sorrel, 2011).

From the XRD analysis, the size of a particle of the samples can be calculated using Scherer's equation and it's also important to do this analysis to identify the phase for each sample. This is because the phase and particle size of the samples is affected significantly by the changes in the calcination temperature during the heat treatment process. From the analysis, it was found only a single phase of anatase

was present at a calcination temperature of 300 °C and 500 °C, and the rutile phase may start to appear at a calcination temperature of 600 °C. At calcination temperature of 300 °C, the peaks intensity of the XRD is weaker compared to the peaks intensity at 500 °C and 700 °C. As the temperature is increasing, the crystallisation of the anatase phase is enhanced (Behnajady & Tohidi, 2013; Aphairaj *et. al.,* 2011; Wetchakun *et. al.,* 2012; Yudoyono *et al.,* 2016). Wide peaks at lower temperatures are attributed to the smaller size of grains (Cai *et. al.,* 2013; Yu & Wang, 2010). At the end of the XRD analysis, it can be concluded as the calcination temperature increasing, the size particle is also increases. The reason is that at higher calcination temperatures, the formed crystallites are larger, which can be attributed to the thermally promoted crystallite growth, also during the heat treatment of TiO_2, dehydration takes place and as a result, the crystals grow to a size larger than those of the original particles (Mozia, 2008; Sheena *et al.,* 2014).

IV. CONCLUSION

XRD analysis has confirmed the photocatalyst that undergoes calcination temperatures of 300 °C and 500 °C to be an anatase structure. A rutile phase has been observed from the XRD analysis for the photocatalyst that calcinated at 700 °C. The rutile peaks were predicted to start to form at a temperature of 600 °C while photocatalyst that undergoes calcination temperature at 500 °C showed anatase structure.

V. ACKNOWLEDGEMENT

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