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The Morphology Characterization of Nitrogen and Magnesium Doped with Titanium Dioxide (TiO_2) using Field Emission Scanning Electron (FESEM) for Photocatalytic Study

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ABSTRACT

Previous studies attempted to modify the electronic structure of TiO_2 in order to broaden the sensitivity to visible light region from the UV light region to visible light region. In this matter, it is very important to identify the effect of parameters that will be affecting the production of photocatalyst as it will also eventually affect the photocatalytic activity. Some of the advantages of dopants are they are effective and efficient. Some of the parameters that could be identify are such as effect of metal and non-metal dopant, the dopant concentration, loading of photocatalyst during photocatalytic activity, calcination temperature during the heating treatment, initial concentration of pollutant and others. In this study, the parameters that will be studied are including type of dopant (metal and non-metal dopant), calcination temperature, and the dopant concentration. In this paper, the temperature was varied for 300°C, 500°Cand 700°C by using different weight percentage which are 0.5 wt. %, 0.7 wt. % and 0.9 wt. %. From FESEM result, shows the temperature affect the morphology of Nitrogen and Magnesium-Doped with Titanium Dioxide (TiO_2).

Key words: FESEM, N-TiO_2, Mg-TiO_2, photocatalytic

1. INTRODUCTION

Doping technique has been applied in photocatalytic reaction in order to overcome the limitations of using pure TiO_2. The limitations of pure TiO_2 are including the wide band gap, incapability to be activated under visible light and also the thermal stability [1]. Besides, this method has become a success to some of the researchers when dopants is introduced to the TiO_2. Non-metal dopants normally used are nitrogen, chlorine, sulphur and fluoride as the dopants. Among these dopants, nitrogen showed a great result in achieving the efficient photocatalysis process. This is due to the small atom size of nitrogen that make it easier to be introduced into the TiO_2 molecule. This is agreed by Yang et al. [2], which claimed that nitrogen is the most effective substitution as a non-metal dopants due to a similar size to oxygen atom as well as low ionization energy, and it is also easy to be introduced into TiO_2 lattice and substitute O, which will results in having a higher photocatalytic activity. Nitrogen has been widely used as dopant due to the comparable size and electronegativity to the oxygen and it is the most suitable element to reduce the band gap width of TiO_2[3]. While Avasalara et al.[4]reported that magnesium doping with TiO_2 may synthesized a better photocatalyst and effect of dopant size on photocatalytic activity. Hence, magnesium is chosen as a dopant and photocatalytic activity.

2. MATERIALS AND METHODS

In this research, several chemicals were used and bought from Sigma-Aldrich. Magnesium chloride 6 hydrate from Bendosen and ammonium nitrate 99 % from Emory were bought from SaintifikBersatu (M) Sdn, Bhd. Company. Ethanol, 95 %, a laboratory grade from HmbG chemical was used as solvent. Acetic acid glacial, 100 %, AR from Emory was used.

In this study, nitrogen-doped TiO_2 and magnesium-doped TiO_2 were prepared using sol-gel method. The samples will be varied at different weight. % (0.5 wt. %, 0.7 wt. % and 0.9 wt. %) for each dopant. Parameters that involved in this experiment are weight concentration of dopants (0.5 wt. %, 0.7 wt. % and 0.9 wt. %), type of dopants (nitrogen and magnesium), calcination temperature (300°C, 500°Cand 700°C). The refrigerator centrifuge was used in order to obtain the sol gel from the solution. At 9000 rpm for 10 minutes, the samples were centrifuged, excess liquid was removed and sol gel was collected. Next, for the heat treatment process, a furnace box (model 524120-P) was used. Different calcination temperature of 300 °C, 500 °C, and 700 °C were applied on the sample for 1 hour. Then the samples were characterized with Field Emission-Scanning Electron Microscope (FESEM).

3. RESULTS AND DISCUSSIONS

All the samples that undergo the sol-gel process were analyzed by using FESEM in order to identify the shape of the samples. From the analysis result, most of the samples (at different calcination temperature) were having an almost spherical shape but with different average particles sizes. Some of the samples have non-spherical shapes. The difference that can be observed from all the result at 500 °C calcination temperature, the particles of the samples were found bigger compared to the particles at 300 °C, and this might be due to the increasing of calcination temperature during the synthesis process. Bigger particle sizes will give smaller surface area of photocatalyst, thus, it will eventually affect the efficiency of photodegradation of Reactive Black 5.

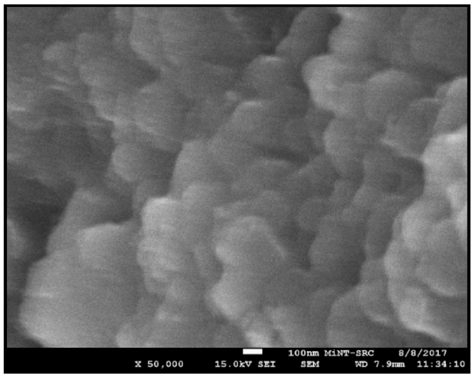

Figure 1: Morphology of TiO_2 without dopants

Figure 1 showed FESEM analysis of undoped TiO_2. From the FESEM image, it can be seen that the particles of TiO2 are agglomerated and bigger in size. Behnajady et al, [5]also found that pure TiO_2 is bigger in size and not uniform in size compared to the Mg-TiO_2 sample in their study. This statement also can be supported by Ganesh et al., [6] which found that the samples of pure TiO_2 were in agglomerated, different sizes and larger particles compared to the doped samples.

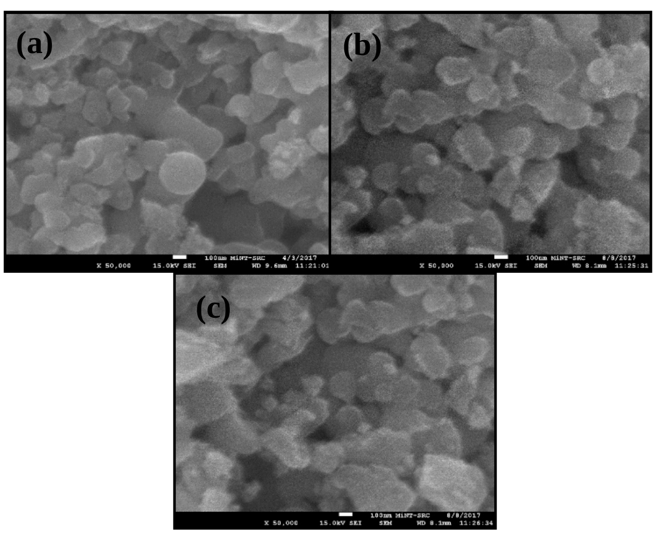

Figure2: Morphology of TiO_2 doped with (a) 0.5wt. % Mg (b) 0.7wt. % Mg and (c) 0.9wt. % Mg at 300°C

Figure 2 showed the FESEM analysis of 0.5wt. % Mg-TiO_2, 0.7wt. % Mg-TiO_2, 0.9wt. % Mg-TiO_2, at calcination temperature of 300°C. As seen clearly in the figure 2, it can be observed that basically, there is not much porosity can be seen, the particles size also is less agglomerated to each other compared to the pure TiO_2. Shivaraju*et al*, [7] also found the SEM image of Mg-TiO_2 in their research to be in spherical, granulated and smaller particles formed. There is not many different can be seen for the sample when different weight percentage content of magnesium was applied.

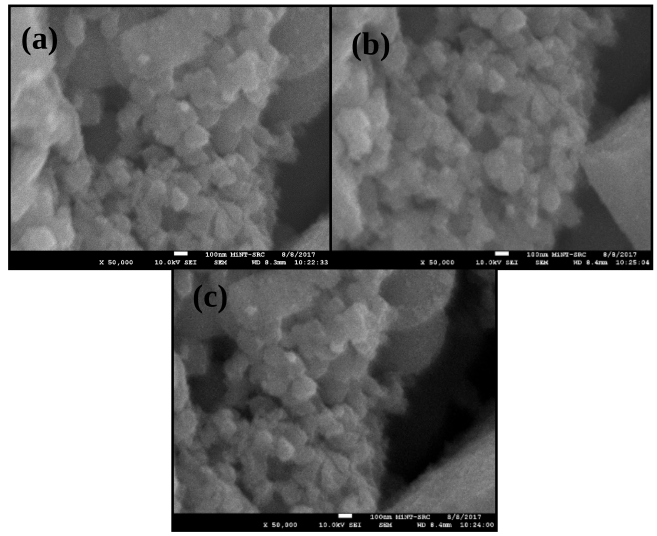

Figure3: Morphology of TiO_2 doped with (a) 0.5wt. % N (b) 0.7wt. % N and (c) 0.9wt. % N at 300°C

The figure 3 shown the morphological analysis of 0.5wt. % N-TiO_2, 0.7wt. % N-TiO_2, 0.9wt. % N-TiO_2, calcined at 300°C. As it can be seen, the morphological shape shown almost spherical for the most of the particles in each sample same as the figure 3. The particles were observed smaller than the pure TiO_2 and it can be seen that the particles were having porous structure compared to the undoped TiO_2. The same result was reported by [8]–[9].

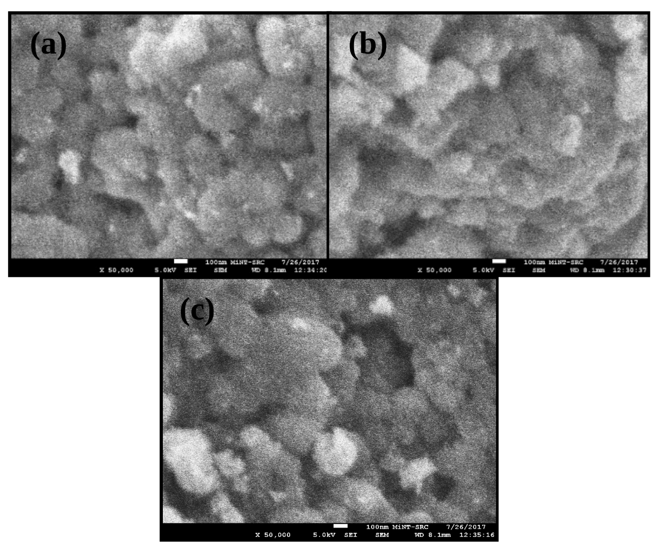

Figure4: Morphology of TiO_2 doped with (a) 0.5wt. % Mg (b) 0.7wt. % Mg and (c) 0.9wt. % Mg at 500°C

Figure 4 and 5 showed FESEM analysis of samples at calcination temperature of 500°C. The size of the particles for both figures were observed bigger than the samples that calcinated at 300°C. This might be due to the increasing of calcination temperature. Also found that as the calcination temperature increased, the size particles become bigger. It is observed that, the grain size is almost same and without much porosity. However, the shape of the particles still in sphere and some agglomerated particles can be seen in the FESEM images. Big particles size with small surface area and agglomerated particles can be the reason of an unfavorable degradation process.

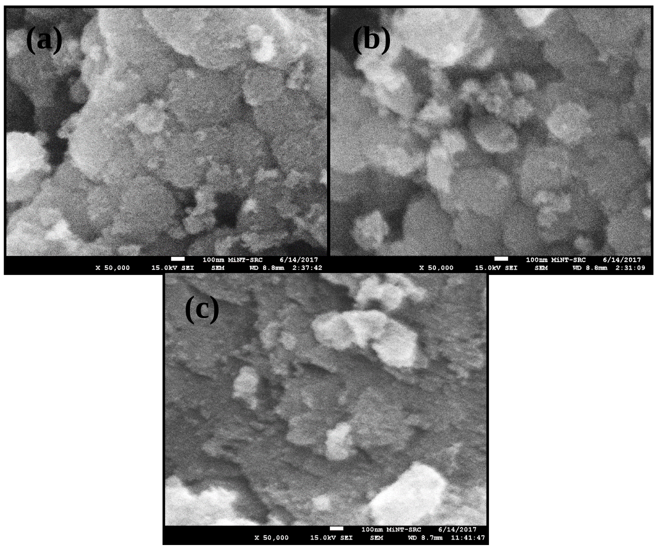

Figure5: Morphology of TiO_2 doped with (a) 0.5wt. % N (b) 0.7wt. % N and (c) 0.9wt. % N at 500°C

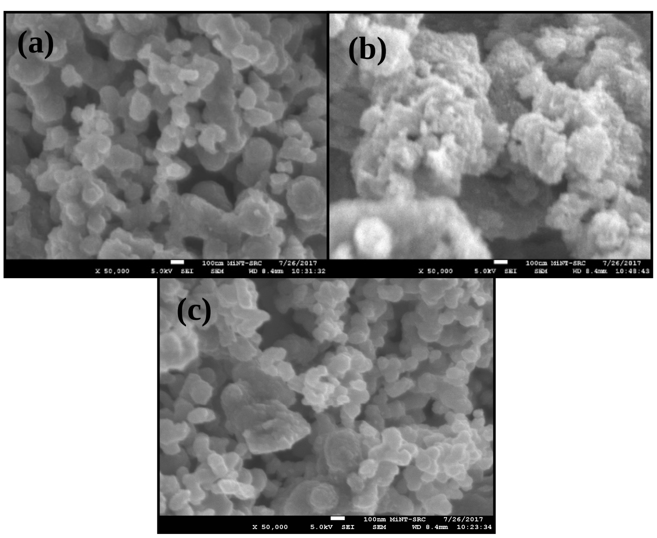

Figure 6: Morphology of TiO_2 doped with (a) 0.5wt. % Mg (b) 0.7wt. % Mg and (c) 0.9wt. % Mg at 700°C

Samples that undergo calcination of 700°C showed on Figure 6 and 7, from the observation, it can be seen that the prepared samples, due to their small grain size, have agglomerated to larger particles. Most of them showed spherical shape and uniform size. Samples calcinated at 700 °C also found has a porous structure in comparison to the undoped sample. Behnajady*et al*, [5]reported the same result when Mg-TiO_2 sample were calcinated at higher temperature of 650 °C. The sample were observed to have smaller grains size and large agglomerated particles were formed. Nolan *et al*[10] also found that the N-TiO_2 sample calcinated at higher temperature of 550 °Chas a porous structure and agglomerated particles are also found to be smaller than pure TiO_2 in their research.

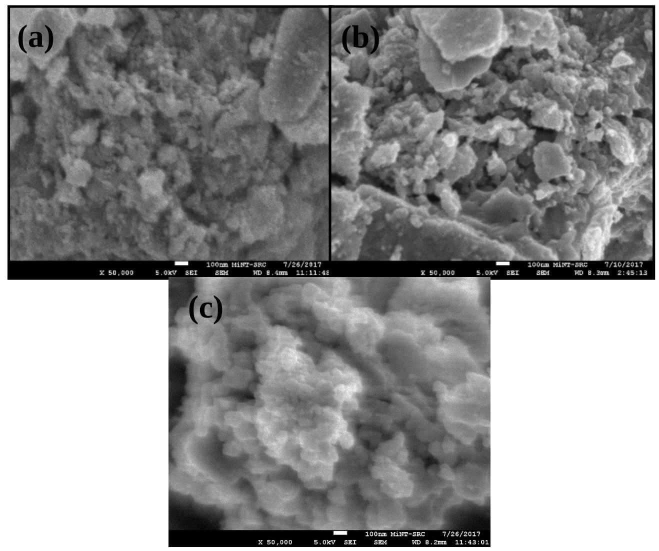

Figure 7: Morphology of TiO_2 doped with (a) 0.5wt. % N (b) 0.7wt. % N and (c) 0.9wt. % N at 700°C

4. CONCLUSION

FESEM analysis has been showed that majority of the particles were in almost sphere shape. Shape particle of photocatalyst that calcinated under 300°C showed sphere and non-agglomerated. While photocatalyst at 500°C showed a bit agglomerated and larger size. Photocatalyst at 700°C showed sphere shape. The undoped TiO_2 were found to be at larges particles size compared to the doped samples.

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