Calcination effect to the physical and optical properties of Zn$_2$SiO$_4$ composite prepared by impregnation of ZnO on SiO$_2$ amorphous nanoparticles

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Abstract. In this study, Zn$_2$SiO$_4$ composite-based ceramic was synthesised using amorphous SiO$_2$ nanoparticles as a silicon source. The amorphous SiO$_2$ nanoparticles which obtained from a simple precipitation process were mixed with aqueous zinc nitrate. Amorphous SiO$_2$ nanoparticles were encapsulated by the zinc source in aqueous solution, dried, and subjected to calcination. The underwent calcination showed the changing of phases, morphology, and size with increased temperatures. During calcination, ZnO phase appeared at the beginning of heating temperature and Zn$_2$SiO$_4$ phase started to emerge at 800 °C onwards, as shown by XRD patterns. The optical band gap analysis of Zn$_2$SiO$_4$ composite was determined to be within the range of 3.12 to 3.19 eV. The diffusion of zinc ions into SiO$_2$ nanoparticles with high surface area also reduced the phase formation temperature for Zn$_2$SiO$_4$, compared to a conventional solid state method. This optical characteristic is expected to be a potential candidate for phosphor materials in opto-electronic devices application.

1. Introduction
The synthesis of zinc silicate (Zn$_2$SiO$_4$) composite has gained more interest due to the combination of the optical properties of both zinc oxide (ZnO) and silica (SiO$_2$) components. This composite system merges the low band gap ZnO with the high band gap SiO$_2$. The formation of ZnO phase during the synthesis depends on the preparation method, sintering temperature, and the ratio of ZnO and SiO$_2$. As for silicate-based phosphor, the selection of silica precursors can certainly influence the final properties of the targeted materials.

Conventionally, ZnO and SiO$_2$ powders are used with appropriate stoichiometry and heated at a high temperature to form Zn$_2$SiO$_4$. Several precursor materials of SiO$_2$ have been used whether from natural source or laboratory grade SiO$_2$. Soda lime silica produced from waste materials [1, 2], silicon alkoxide, or tetraethyl orthosilicate (TEOS) [3], and commercialized mesoporous SiO$_2$ [4] are among common SiO$_2$ sources. The conventional melt quenching of soda lime silica, mixed with ZnO, produces a glass precursor, which is then subjected to heat treatment to form Zn$_2$SiO$_4$-based glass-ceramic. The drawback of using soda lime silica is the presence of other elements in the glass, which can affect the properties of the final materials [5]. TEOS is a liquid SiO$_2$ precursor that needs to be added into a mixture of water and alcohol solvent for the Si ions to react with zinc ions. A suitable ratio of water to alcohol solvent is required for the hydrolysis of TEOS to occur and to form Zn$_2$SiO$_4$. Consequently, the hydrophobic properties [6] and toxicity of TEOS [7] become drawbacks to those that have specific requirements during production. With a simple route of reaction, porous silica
powder can be synthesised from the reaction of water glass with weak acids, such as alcohol (ethanol), as demonstrated by [8] and [9]. The resulting porous silica particles can offer an advantage when used as a precursor during the formation of the $\text{Zn}_2\text{SiO}_4$ phase. Porous silica made of water glass has different porosity characteristics due to the reaction with a weak acid.

Several methods for synthesising $\text{Zn}_2\text{SiO}_4$ composite have been reported by previous researchers. Sohn, Ramakrishna and Diao [10-12] performed solid state reaction that involving high temperature heat treatment with certain soaking time in order to form whether single phase or composite of $\text{Zn}_2\text{SiO}_4$. Sol- gel method that used zinc salts, such as nitrate and acetate, and alkoxy derivatives of silicon, such as tetraethyl orthosilicate are often reported in preparation of $\text{Zn}_2\text{SiO}_4$ composite [13]. There are also variations of synthesis arrangements for the sol-gel method. Some researchers may produce solid $\text{SiO}_2$ first, then mix it with zinc precursor, or vice versa, or combine a mixture of $\text{ZnO}$ and $\text{SiO}_2$ sources together. Silica in the form of tetraethoxysilane (TEOS) is usually mixed with the aqueous solution of zinc, particularly zinc nitrate ($\text{Zn(NO}_3)_2$) and zinc acetate ($\text{Zn(CH}_3\text{COO})_2$). The prepared nanoparticles would appear amorphous prior to calcination. The simultaneous mixing of precursors will cause the ZnO nanoparticles to be homogeneously dispersed in the silica matrix and retained at a certain temperature. Calcination of the amorphous phase of $\text{ZnO-SiO}_2$ composite start to form $\text{Zn}_2\text{SiO}_4$ composite phase at 800 °C that as in the form of thin films [14] and at 900 °C for powder form [15,16].

Hydrothermal and vapour method are both often been used for $\text{Zn}_2\text{SiO}_4$ composite preparation. In hydrothermal method, water, or an aqueous mixture is used as the reaction medium. The experiments is regularly conducted in a Teflon-lined autoclave to which raw materials or precursors are loaded and heated at a temperature that ranges between 100 to 370 °C. The high pressure in the Teflon-lined container can reduce the formation temperature of $\text{Zn}_2\text{SiO}_4$ phase [17]. A gas phase is used as the reaction medium in a vapour method. The raw materials, or precursors are sprayed, or vaporized into a gas at temperatures that range between 300 to 1300 °C to produce solid materials. The vapour method contributes to low crystalline, or amorphous-like α-phase $\text{Zn}_2\text{SiO}_4$ and calcination should be done to turn it into a crystalline phase [18].

The application of impregnation techniques using silica begin with the preparation of metal-supported catalysts. Using this method, the filling of the surface pores can initiate the adsorption of metal ions. The impregnation or absorption of metals on silica as a substrate have been reported by Clause, Chaneac and Bonneviot [19-21]. They emphasized that the interactions between metal ions in a solution, or mobile medium with the solid surface of support material can strongly influence the size distribution and stability of the oxide particles during calcination process. The interaction of metal ions in a solution at the solid-liquid interface lead to deposition precipitation with addition of electrostatic adsorption of cations and counteranion without modification of support surface.

This study was focused on synthesising $\text{Zn}_2\text{SiO}_4$ composite using a wet impregnation method. This method applied amorphous silica nanoparticles as a morphology-controlling template to perform chemical reactions or for new phase formation when combined with other ions; in this case, zinc ions.

2. Materials and method

$\text{Zn}_2\text{SiO}_4$ composite powders were produced by mixing amorphous silica nanoparticles that were produced based on the method established elsewhere [22], with zinc nitrate hexahydrate. First, 1.20 g of amorphous silica nanoparticles was dispersed in 200 mL of deionized (D.I.) water using an ultrasonic agitator for 30 minutes. Next, the amorphous silica nanoparticle dispersion was mixed with a solution of 11.89 g of zinc nitrate hexahydrate (Sigma Aldrich), which was already dissolved in 200 mL of deionized water. This mixture was stirred for 2 hours using a magnetic stirrer. The ratio of the precursor concentration of $\text{Zn}:\text{Si}$ is 2:1. Then, the mixed solution was poured into a petri dish and dried in an oven for 24 hours at 120 °C. The dried powder was ground using a mortar grinder and finally, heat treated at 600, 700, 800, 900, and 1000 °C at 10 °C/min heating rate for 3 hours.

To study the structural characteristic of synthesized $\text{Zn}_2\text{SiO}_4$ composite powders, the sample powder were characterized by X-ray diffraction (XRD) (PANalytical X’pert PRO PW 3040 MPD), thermal analysis (Mettler Toledo TGA/DSC 1HT), field emission scanning electron microscopy (FESEM) (FEI NOVA NanoSEM 230) and transmission electron microscopy (TEM) (Hitachi H-7100). In addition, the optical property was characterized using UV-Visible (Shimadzu UV-3600) spectrometer for optical absorption and band gap determination.
3. Results and discussion

3.1. XRD analysis

The effect of calcination on the structure of ZnO-Zn$_2$SiO$_4$ composite was investigated using XRD spectrum of each sample at temperatures ranging from 600 to 1000 °C. Figures 1 shows the XRD spectra of the ZnO-Zn$_2$SiO$_4$ composite for Zn:Si ratios of 2:1. The wurtzite structure of the ZnO nanocrystals was observed at 600 °C. Nine major diffraction peaks were positioned at 2θ values of 31.79°, 34.44°, 36.27°, 47.56°, 56.61°, 62.88°, 66.39°, 67.96°, and 69.10°, registered as JCPDS No. 89-0510. The lattice constant was obtained using the standard formula for tetragonal crystal system $1/d^2 = ((h^2+k^2)/a^2)+(l^2/c^2)$, where h,k,l are Miller index, d is inter planar spacing obtained from Bragg’s law, and a,b,c are the lattice constant. The lattice constant calculated from the XRD spectrum gave the value of $a = 3.249$ Å and $c = 5.206$ Å, which were similar to ZnO hexagonal wurtzite structure [23]. With the progression of sintering temperature from 600 to 700 °C, the diffraction peaks had broadened. Usually, the temperature gain will increase the crystallite size due to the agglomeration. The changes in FWHM can be influenced by the changes of crystallite size and strain due to the temperature increase. This was shown by the reduction of FWHM peak intensity. From the data, the progressive sintering temperature, from 600 to 700 °C, had expanded the FWHM of the diffraction peaks. Therefore, the diffraction peak broadening of ZnO nanocrystals was not affected by the increasing particle size. Strain could also promote the broadening of XRD peaks when the temperature was increased. As a result, the diffraction peaks of ZnO were shifted towards the higher angle side, with increases in temperature from 600 to 700 °C. Thus, the broadening and shifting of the diffraction spectrum peaks of ZnO nanocrystals could be linked to the strain induced in the ZnO nanocrystals by the surrounding silica network [24].

The formation of Zn$_2$SiO$_4$ phase was initiated at 700 °C due to the presence of peak shoulder and peak broadening, but appeared clearly at 800 °C. The major diffraction peaks were positioned at 21.81°, 22.19°, 25.67°, 31.64°, 34.13°, 38.94°, 42.26°, 43.73°, 45.16°, 46.30°, 47.05°, 54.43°, 56.67°, 57.72°, 59.64°, 61.00°, 62.94°, 66.42°, and 68.03°, registered as α-Zn$_2$SiO$_4$ crystal phase of JCPDF No. 37- 1485. The formation of Zn$_2$SiO$_4$ peak from 700 °C onwards was influenced by the ZnO phase that became a dominant diffusing phase during calcination [25]. This formation temperature is lower than conventional solid state reaction temperature that required the heating temperature between 900 to 1500 °C for several hours for the mixture of precursor to melt, followed by heat treatment in order to get the crystalline phase [17].

![Figure 1. XRD pattern of calcined ZnO-Zn$_2$SiO$_4$ composite between 600 to 1000 °C.](image-url)
3.2. Thermal analysis

Figure 2 show the TG and DTG curves for Zn$_2$SiO$_4$ composite. Weight loss became an approximate constant value beginning at 300 °C. Additionally, similar four sharp peaks were observed in the DTG curves for all samples with different ratios of zinc. The first and second peak at approximately 78 to 88 °C and 123 to 136 °C can be attributed to dehydration water and dehydroxylation of zinc hydroxide, which would normally evaporate at this temperature. The third and fourth peak, which appeared in the range of 219 to 220 °C and 278 to 291 °C, were caused by the decomposition of nitrate to nitrogen oxide, and the release of oxygen gas, as identified by Biswick [26]. The decomposition of these substances was accompanied by the abrupt weight loss at approximately 300 °C for all samples.

The ZnO phase was already formed at this temperature after the decomposition of the organic substances, up to 600 °C, as shown by the XRD patterns. The particle weight remained constant from 600 °C up to 1200 °C. The subsequent minor weight losses before constant weight began were observed between 300 °C to 600 °C. These weight losses could be related to the decomposition of oxygen due to the initial formation of Zn$_2$SiO$_4$ phase.

![Thermogravimetric (TG) and derivative thermogravimetric (DTG) curves of Zn$_2$SiO$_4$ composite.](image)

3.3. FESEM and TEM analysis

The morphological changes of the Zn$_2$SiO$_4$ composite, with the progression of calcination temperature, were studied using FESEM. Figure 3 shows FESEM images of ZnO-Zn$_2$SiO$_4$ composite calcined between 600 to 1000 °C. It is clear that these composites exhibited spherical morphology at 600 and 700 °C, but at temperatures of higher than 800 °C, the curvature of the spherical particles that are touching each other will be diffused first, and start to form the neck-like or dumbbell structures [27]. The small spherical particles have a vast surface area compared to a similar volume of bulk solid. The surface properties of bulk and nanosize particles are different because a nanosize particle has a large surface to volume ratio and small radius of curvature. The FESEM images were analysed using image processing and analysis software, ImageJ in order determine the particles size distribution. The overall morphology of Zn$_2$SiO$_4$ composite, upon annealing at 600 °C, showed the spherical form with an average diameter of 69.68 ± 3.20 nm. Some parts of the composite displayed individual particles, while other parts showed agglomeration. Burned off residual organic groups, such as nitrates, could lead to the encapsulation of ZnO on the surface of SiO$_2$ nanoparticles [28]. Further increased of temperature lead to diffusion and particle size cannot be precisely estimated from annealing at 700 °C (Figure 3 (b)). Eventually, the pores would also be closed by the diffusion of zinc ions at higher than 700 °C.

Figure 4 shows the TEM images of Zn$_2$SiO$_4$ composite at different calcination temperatures. These images show a nearly dense core that became darker than the layer near the surface of the particles. The zinc oxide encapsulated the silica core, which can be seen as a semi-transparent layer at the edge of the particle boundary, at sintering temperature of 600 °C. With further increase of sintering temperature,
the cores of the particles became darker and the semi-transparent layer near the surface was reduced [29]. Concurrently, interparticle diffusion was observed, with the near-to-polyhedral shape morphology forming at 8000 °C (Figure 3 (c)). Subsequently, the continuous diffusion between particles led to the formation of larger particles at 1000 °C (Figure 4 (b)).

Figure 3. FESEM images of ZnO-Zn$_2$SiO$_4$ composite sintered at temperature of (a) 600 °C, (b) 700° C and (c) 800 °C.

3.4. UV-vis analysis
The UV-visible spectroscopy measurement of the Zn$_2$SiO$_4$ composite samples heat treated at various temperatures were recorded in the spectral region of 200 to 800 nm. Figure 5 show the absorption spectra of the ZnO-Zn$_2$SiO$_4$ composite sintered at various temperatures. The maximum absorption edge of the heat treated samples was at approximately 380 nm. This sharp absorption edge corresponds to the characteristics of the crystalline phase of ZnO. For sample calcined at 600 °C, lower absorption intensity was observed. When the calcination temperature was increased higher than 800 °C, a decrease was observed in the absorption peak intensity, along with the shift of the peak towards lower wavelength. Another absorption edge was observed between 260 and 280 nm for the sample sintered at higher than 800 °C. Uegaito [31] linked the emerging shoulder peak at 255 nm as absorption edge from Zn$_2$SiO$_4$ phase after annealing at higher than 800 °C. Omri and Sharma [32,33] also made the same reference for this particular absorption edge. However, in this study, the minimum observed wavelength in the spectrum was at 220 nm, which made the existence of the small absorption edge possible. Based on the previously discussed XRD, FESEM, and TEM results, the decrease in the UV-vis absorption peak intensity can be attributed to the reducing of ZnO phase and the formation of Zn$_2$SiO$_4$ crystal [34].

Figure 4. TEM images of ZnO-Zn$_2$SiO$_4$ composite sintered at temperature of (a) 600 °C and (b) 1000 °C.
Figure 5. UV-Vis absorption spectra for $\text{Zn}_2\text{SiO}_4$ composite sample, with Zn:Si ratio of 2:1 using SiO90m, sintered at 600 to 1000 °C.

Figure 6. Tauc plot of $(\alpha h\nu)^2$ vs. energy for $\text{Zn}_2\text{SiO}_4$ composite, with Zn:Si ratio of 2:1, sintered at 600 to 1000 °C using SiO90m.

The optical band gap was determined from the relationship between the absorption coefficient and photon energy, which is associated with an electronic transition. The relationship is given by Equation $(\alpha h\nu)^n = A(h\nu - E_g)$, where $A$ is a constant, the photon energy is denoted by $h\nu$, and $E_g$ is the optical energy band. The $n = \frac{1}{2}$ which is direct allowed transition is used to determined optical band gap value from the best linear fit by extrapolating the linear parts of $(\alpha h\nu)^n$ vs. $h\nu$ curves [35,36]. The extrapolation of linear region from individual plot lines to $x$-axis intercept will give optical band gap value as shows in Figure 6. The optical band gaps of Zn2SiO4 composites had ranged between 3.12 and 3.19 eV, which was lower compared to the bulk band gap for ZnO crystal phase at 3.37 eV. The lower optical band gap value obtained in this study was due to the optical confinement effect of the formation of ZnO layers on the silica nanoparticles [37,38]. The increasing heat treatment temperature would definitely affect the ZnO layer, which would start to diffuse within the SiO2 nanoparticles. In this study, higher calcination temperature had increased the particle size and the porous particles became denser, as shown in FESEM and TEM micrographs. This condition would lead to higher absorbance of photons [39].
4. Conclusions

In this study, ZnO-Zn$_2$SiO$_4$ composite were successfully prepared by the wet chemical impregnation method followed by calcination. Amorphous SiO$_2$ nanoparticles were chosen as the precursors and mixed with aqueous zinc nitrate. Results from the XRD analyses showed the formation of ZnO-Zn$_2$SiO$_4$ composite when the temperature was increased to higher than 800 °C, which was lower than conventional solid state sintering temperature. The impregnation of zinc ions with amorphous SiO$_2$ nanoparticles had significantly reduced the formation temperature of Zn$_2$SiO$_4$ phase. Based on the morphological analyses from FESEM and TEM, as the temperature increases, the spherical particles exhibited interconnections between the particles. The UV-Vis measurements showed the values of the calculated optical band gap of ZnO to be in the range of 3.12 to 3.19 eV. Conclusively, the synthesised Zn$_2$SiO$_4$ composite had shown a promising properties for phosphor materials. Its optical properties appeared suitable to be further developed for applications in photonic and opto-electronic materials.

5. References

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Acknowledgments
The authors gratefully acknowledged the financial support for this study from the Ministry of Education Malaysia, Universiti Malaysia Terengganu and Universiti Putra Malaysia.