Biogenic synthesis of magnesium oxide nanoparticles using Aloe barbadensis leaf latex extract

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Abstract. Biological methods are employed to yield less or non-toxic MgO nanoparticles to utilize them in biological applications. Among various biosynthesis approaches, plant extracts with phytochemicals, especially from leaves, are widely used to fabricate MgO nanoparticles, due to their high availability, rapid synthesis and ability to yield smaller stable nanoparticles. Aloe barbadensis is a succulent xerophytic plant with unique characteristics to withhold water in its leaf named parenchymal gel, which is protected by a chloroplast containing thick latex, to avoid transpiration in high temperature condition of the desert. These latex contains phytochemicals such as flavanol, quercetin, Kaempferol, myricetin and fisetin, along with other common phytochemicals such as phenols and terpenoids, that are essential for nanoparticle formation. Further, these compounds also possess enhanced biological properties. Thus, the aim of the present study is to obtain crude phytochemical extracts from Aloe barbadensis latex and utilize them as reducing and stabilizing agent for the smaller MgO nanoparticle formation. In addition, the parameters which affect the formation of nanoparticles are identified and optimized to yield smaller MgO nanoparticles with phytochemicals as surface functional groups, to be beneficial in biomedical applications.

Keywords: Aloe barbadensis; Biogenic synthesis; MgO nanoparticles; Leaf latex; Phytochemicals; Functional groups

1. Introduction

Magnesium oxide (MgO) nanoparticles are currently popular among researchers, due to their exclusive biological, electronical and thermal properties [1]. This has led to the search of various synthesis approaches to yield smaller sized MgO nanoparticles with distinct morphologies [2]. Chemical approaches such as sol-gel, hydrothermal, co-precipitation and solvothermal methods are under extensive research to yield smaller sized nanoparticles [3-6]. However, the toxicity of these chemicals derived MgO nanoparticles are the major limitation, while utilizing these nanoparticles for biological applications [7]. Thus, biological methods are employed to yield less or non-toxic MgO nanoparticles to utilize them in biological applications [8]. Algae [8] and fungi [9] are used for the biosynthesis of MgO nanoparticles, however the time required for synthesis and limited availability of the organisms are the drawbacks to utilize them for large scale biological applications. Thus, plant extracts with phytochemicals, especially from leaves, are widely used to fabricate MgO nanoparticles, due to their high availability, rapid synthesis and ability to yield smaller stable nanoparticles [10-12].

Aloe barbadensis is an evergreen perennial succulent xerophytic plant that grows widely in places, where ground water is low [12]. The evolution to sustain the climatic conditions in the desert...
(xerophytic) ecosystem, these plants possess unique characteristics to withhold water in its leaf named parenchymal gel to avoid transpiration in high temperature condition of the desert [13]. Thus, the normal leaves in Aloe barbadensis are modified into pseudo-leaves with high water content (gel) [14], which is protected by a chloroplast containing thick latex [15]. These latex contains phytochemicals such as flavanol, quercetin, Kaempferol, myricetin and fisetin [16], along with other common phytochemicals such as phenols [17] and terpenoids [18]. Both the parenchymal gel as well as latex is highly beneficial in biomedical applications, due to the presence of unique phytochemical combinations [19, 20]. Several metal oxide nanoparticles have been synthesized previously by using phytochemical extracts as reducing and stabilizing agents [21-23]. Thus, the aim of the present study is to obtain crude phytochemical extracts from Aloe barbadensis latex and utilize them as reducing and stabilizing agent for the smaller MgO nanoparticle formation. In addition, the parameters which affect the formation of nanoparticles are identified and optimized to yield smaller MgO nanoparticles with phytochemicals as surface functional groups, to be beneficial in biomedical applications.

2. Methodology

2.1. Materials

The magnesium nitrate hexahydrate (Mg (NO₃)₂•6H₂O) with molecular weight of 256.41 g/mol and purity of 99.8% was used as magnesium precursor and are purchased from Alfa Acer® (Singapore). The Aloe barbadensis latexes were collected from the local farm in Miri, Sarawak, Malaysia. The ethanol for the phytochemical extraction was purchased from Sigma-Aldrich® (Singapore) with molecular weight of 46.069 g/mol and 70% purity.

2.2. Preparation of Aloe latex powder

The Aloe barbadensis leaf latex was collected from local farm and cleaned with distilled water. The latex was chopped into small pieces and homogenized in a bio-dryer for a day. Later, the chopped latexes were blended using a blender to obtain fine powders as shown in Figure 1. However, the latex contains water molecules along with leaf layer, which makes the powder to be rich in water content. The water molecules can be removed by freeze-drying the latex powder at -40°C and 0.133 mBar vacuum using Fisher brand Labconco® 1.5 L freeze dry system as shown in Figure 1. The resultant latex powders were stored in a desiccator for further experiment.

![Figure 1. Processes involved in Aloe latex powder preparation](image)

2.3. Preparation of Aloe solid and liquid extract

20 grams of latex powder were added to 100 ml of distilled water and 100 ml of ethanol in a 250 ml conical flask [24]. The mixture in the conical flask was allowed to constantly stir (450 rpm) for 24 h at room temperature for complete elucidation of active materials and to dissolve them in the solvent as shown in Figure 2. Later, the latex powder extract is filtered using vacuum filtration method, where both the residues and filtrate was collected as in Figure 2. The collected residues (solid state) and the filtrate (aqueous state) are subjected to liquid-liquid extract purification process via the rotary vacuum evaporator for 3 h to eliminate solvent (ethanol) solvent from the aqueous phytochemical extract using a water bath at 50°C of temperature as displayed in Figure 2. The resultant solid residue and the aqueous filtrate containing phytochemicals, that are free from ethanol, was collected and used for MgO nanoparticle synthesis.
2.4. Preparation of MgO nanoparticles
Both solid and liquid extracts of Aloe leaf latex powder were used for the biosynthesis of smaller MgO nanoparticles. The parameters that affect the synthesis such as reaction time, extract ratio, temperature and precursor concentration was optimized using one-factor-at-a-time (OFAT) approach. Table 1 shows the design of experiment used for the optimization of parameters. In each step of the OFAT analysis, the parameters were optimized, and the optimized parameter was included in the next step. The flowchart for the synthesis of MgO with both extracts are shown in Figure 3.

| Sample name | Fixed parameters | Parameter to be optimized | Extract ratio | Temperature (°C) | Precursor concentration (mol/L) |
|-------------|------------------|---------------------------|---------------|------------------|---------------------------------|
| SE1/LE1     | 60°C temperature (fixed) | 0.01M precursor (fixed) | 1:10          | 40               | 0.01                            |
| SE2/LE2     | 60°C temperature (fixed) | 0.01M precursor (fixed) | 1:20          | 50               | 0.055                           |
| SE3/LE3     | 60°C temperature (fixed) | 0.01M precursor (fixed) | 1:30          | 60               | 0.10                           |
| SE4/LE4     | 60°C temperature (fixed) | 0.01M precursor (fixed) | 1:40          | 70               | 0.15                           |
| SE5/LE5     | 60°C temperature (fixed) | 0.01M precursor (fixed) | 1:50          |                   |                                 |
| ST1         | 1:10 extract ratio (selected) | 0.01M precursor (fixed) | 40            |                   |                                 |
| ST2         | 1:10 extract ratio (selected) | 0.01M precursor (fixed) | 50            |                   |                                 |
| ST3         | 1:10 extract ratio (selected) | 0.01M precursor (fixed) | 60            |                   |                                 |
| ST4         | 1:10 extract ratio (selected) | 0.01M precursor (fixed) | 70            |                   |                                 |
| SP1         | 60°C temperature (selected) | 1:10 extract ratio (fixed) | 40            | 0.01             |                                 |
| SP2         | 60°C temperature (selected) | 1:10 extract ratio (fixed) | 50            | 0.055            |                                 |
| SP3         | 60°C temperature (selected) | 1:10 extract ratio (fixed) | 60            | 0.10             |                                 |
| SP4         | 60°C temperature (selected) | 1:10 extract ratio (fixed) | 70            | 0.15             |                                 |
| LT1         | 1:50 extract ratio (selected) | 0.01M precursor (fixed) | 40            |                   |                                 |
| LT2         | 1:50 extract ratio (selected) | 0.01M precursor (fixed) | 50            |                   |                                 |
| LT3         | 1:50 extract ratio (selected) | 0.01M precursor (fixed) | 60            |                   |                                 |
| LT4         | 1:50 extract ratio (selected) | 0.01M precursor (fixed) | 70            |                   |                                 |
| LP1         | 60°C temperature (selected) | 1:50 extract ratio (fixed) | 40            | 0.01             |                                 |
| LP2         | 60°C temperature (selected) | 1:50 extract ratio (fixed) | 50            | 0.055            |                                 |
| LP3         | 60°C temperature (selected) | 1:50 extract ratio (fixed) | 60            | 0.10             |                                 |
| LP4         | 60°C temperature (selected) | 1:50 extract ratio (fixed) | 70            | 0.15             |                                 |
2.5. Characterization of MgO nanoparticles

The parameters were optimized based on the average particle size (particle size distribution) of the samples. Nano-ZS Zetasizer (Malvern Instruments, Worcestershire, UK) was used to analyze the average particle size and poly dispersity index of the biosynthesized MgO nanoparticles via dynamic light scattering (DLS) approach [25]. The formation of MgO nanoparticles via both solid and liquid Aloe extracts was confirmed by their surface plasmon resonance property via Perkin Elmer® Lambda 25 Ultraviolet-visible (UV-Vis) spectroscopy. The calcination temperature to obtain pure MgO nanoparticles from the solid extracts were identified via thermal degradation and heat flow patterns from Thermal gravimetric analyzer – Differential Scanning Calorimetry (TG-DSC), Mettler Toledo [26]. The functional groups present in the Aloe extracts and the MgO nanoparticles were identified using Thermo scientific NICOLET iS10 Fourier-Transform Infrared (FTIR) spectroscopy. Transmission electron microscope (TEM) from Tecnai™ G² Spirit BioTWIN, FEI company, is used to determine the size, shape and morphology of MgO nanoparticles and the analysis was performed at an accelerating voltage of 200 kV.

3. Results and discussions

Both solid and liquid extracts of Aloe latex powder were green in color, which upon adding to the precursor dissolved in distilled water, does not change in color. The heating process to form nanosized MgO particles has led to a pale green color of the powder (solid extract) and the filtrate (liquid extract) which denotes the formation of MgO nanoparticles and are confirmed via DLS analysis.

3.1. Optimization of reaction time

Initially, the reaction time to form the MgO nanoparticles was optimized with 60°C as temperature, 1:50 as extract (extract: distilled water) ratio and 0.01 M as precursor concentration. It is noteworthy that 23°C and 85°C of temperatures may lead to precipitation of particles, which may increase the size of nanoparticles by irregular disintegration of phytochemicals in the extract [27]. Further, it has been demonstrated in our previous study that temperatures below 60°C is not sufficient to provide energy for the formation and stabilization of nanoparticles. Thus, 60°C was selected as the optimum
temperature to optimize reaction time. Figure 4 (A) shows the average size of MgO nanoparticles formed via reaction time of 10 to 60 min. The smallest nanoparticle size of 78.82 nm was obtained at 30 min of reaction time, which was selected as the optimum reaction time for the formation of MgO nanoparticles using both solid and liquid Aloe latex powder extract. The selected reaction time was similar to the experiment performed by Gopalakrishnan et al. (2014), where chemical precipitation approach was used to obtain molybdenum sulfate quantum dots [28]. Further, MgO nanoparticles synthesized via other approaches such as sol-gel, microemulsion, co-precipitation, hydrothermal/solvothermal methods require a reaction time of minimum 2 hours [29]. Hence, 30 min was selected as the optimum reaction time for the fabrication of MgO nanoparticles and to optimize other parameters that affect nanoparticle formation capability of Aloe latex powder extracts.

3.2. Optimization of extract ratio

The addition of water with aqueous phytochemical extract (both solid and liquid) is a significant factor to be optimized, as it determines the concentration of plant extract (dilutions) in the reaction. A reaction time of 30 min, temperature of 60°C and 0.01 M of precursor concentrations were used as constant. Figure 4 (B) shows the average MgO nanoparticle size that is formed, while using water in the range of 10 to 50 parts along with 1 part of the extract. It was observed that liquid extract: an aqueous ratio of 1:50 yields smaller MgO nanoparticles with size of 85.05 nm. This may be due to the ability of phytochemical extract to reduce the particle size upon increasing reduction rate by decreasing concentrations [30]. The result of experiments performed by Ali et al. (2016) is exactly similar, where synthesis of ZnO nanoparticles was reported with variations in the concentrations of Aloe vera extract such as 10, 20, 30, 40 and 50% to be added in 100 mL of 0.25M ZnSO4 solution [31]. Similarly, a smaller MgO nanoparticle of 91.28 nm size was formed, when the extract ratio was 1:10 for the solid Aloe extract. The higher extract ratios yielded smaller nanoparticles, contrary to the liquid Aloe extract. However, it was reported that the increase in the concentration of solid extract may lead to decrement in the particle size of nanoparticles [32]. Thus, it is evident that both liquid and solid exhibits different mechanisms in the formation of nanoparticles, as there is a variation in the presence and quantity of certain phytochemicals.

3.3. Optimization of temperature

Figure 4 (C) shows the graph of average MgO nanoparticle size yielded by both solid and liquid extract at different temperatures ranging from 20 to 80°C. The precursor concentration of 0.01 M, a liquid extract ratio of 1:50 and reaction time of 30 min was used as constant. It is noteworthy that a smaller sized MgO nanoparticle was obtained between 50-60°C of reaction temperature for both solid and liquid extracts. MgO nanoparticle with an average size of 85.05 nm was obtained at 60°C, when liquid Aloe extract was used, whereas a further increase in the temperature has led to an increment in particle size. It is well known from previous literature that an increase in temperature will result in elevating diffusion coefficient, that reduces the reaction time required for the formation of stable nuclei and hence, reduces induction time. Typically, increasing temperature will increase the solubility and thus reduces the supersaturation. When the low supersaturation growth is more favour than nucleation, the growth rate increases and larger sized nanoparticles will be yielded [33]. Likewise, the effect of temperature in the average size of MgO nanoparticle was studied for solid Aloe extracts, which revealed that the nanoparticle size increases with an increase in temperature as explained in literatures [34]. A smaller nanoparticle size of 71.02 nm was obtained at 50°C, however, the PDI (supplementary information) revealed that the particles are polydispersed in nature. Thus, 60°C was selected as an optimum temperature to yield smaller MgO nanoparticles via both solid and liquid Aloe phytochemical extracts. These results can be attributed to the nucleation and growth of nanoparticles in a solution, where the particle morphology is affected by supersaturation, nucleation, and growth rates, colloidal stability, recrystallization, and aging process. Supersaturation is determined by the solution temperature, which has a predominant influence on the particle morphology and possesses high Gibbs free energy. The tendency of a system to lower its Gibbs free energy is the driving force in the processes of nucleation and growth of particles. At low temperature, a higher saturation result in a greater reduction of Gibbs free energy and this energy reduction will become the increased surface energy facilitating the continued nucleation to form smaller sized particles. The high temperature prefers a rapid hydrolysis and leads to a high supersaturation, which in turn lead to the formation of...
several nuclei. Thus, a rapid growth of nanoparticles size was observed at high temperatures [35], which has led to the selection of 60°C as the optimum reaction temperature for smaller sized MgO nanoparticle formation.

3.4. Optimization of precursor concentration

Figure 4 (D) shows the effect of different precursor concentration on the average size of MgO nanoparticles. It can be noted that an increment in the precursor concentration, increases the size of the nanoparticle. Further, the higher concentration of metal precursors enhances the size of the nanoparticle, independent of its nature. When liquid and solid Aloe latex powder extract were used with lower precursor concentration of 0.01 M, smaller average particle size of 85.05 nm and 72.3 nm was obtained, respectively. It was observed that lower precursor concentrations generally result in the formation of nanoparticles with smaller diameters, as precursors affect the crystallinity of the particles. Likewise, the higher precursor concentration may result in a higher nuclei density, which may lead to the formation of larger primary particles. These primary particles may lead to the agglomeration along the axis of the nanoparticle, which increases their size [36].

Figure 4. Optimization of MgO nanoparticle synthesis parameters (A) Reaction time, (B) Extract ratio, (C) Temperature and (D) Precursor concentration

3.5. Optimized parameters

The parameters such as reaction time, extract ratio, temperature and precursor concentration are optimized via OFAT approach. The optimized parameters are 30 min of reaction time, 60°C of reaction temperature and 0.01 M of precursor concentration for both Aloe (solid and liquid) extracts to yield smaller MgO nanoparticles. The optimized liquid extract to aqueous ratio is 1:50, whereas 1:10 ratio of solid extract: water was selected to obtain smaller MgO nanoparticles. Thus, it is evident that liquid and solid extract possesses significantly distinct phytochemicals, which alter their mechanism of nanoparticle formation. The MgO nanoparticles were synthesized using these optimized parameters for further characterizations.
3.6. Surface plasmon resonance analysis of MgO nanoparticles

Figure 5 (A) and (B) displays the UV and visible light absorbance spectra of the particles formed using liquid and solid Aloe latex powder extracts, respectively, magnesium nitrate as a precursor along with optimized parameters. The peak at ~280 nm in Figure 5 (A) and (B) represents the existence of oxide molecule in the particle. A miniscule peak at ~540 nm in Figure 5 (A) represents the presence of metal in the Aloe liquid extract yielded particles, which is absent in the particles formed by Aloe latex powder solid extract. Thus, it is evident that magnesium oxide is formed as colloidal particles in the liquid phytochemical extracts of Aloe latex powder. The results were similar to a previous study in which MgO nanoparticles are prepared via *Amaranthus tricolor* leaf extract and the optical absorbance of those nanosized particles are observed in the UV region (320 nm) [37]. The absence of peak in the visible region and the increase in a peak at 380 nm in Figure 5 (B) showed that hydroxide may be formed instead of oxide particles [38], while using Aloe solid latex powder extract. Hence, thermogravimetric analysis was performed to confirm the presence of metal hydroxide in the sample obtained from Aloe solid extracts.

![Figure 5. UV-Visible spectrum of (A) liquid extract and (B) solid extract of Aloe latex powder mediated MgO nanoparticles](image)

3.7. Thermogravimetric analysis of solid Aloe extracts synthesized nanoparticles

Figure 6 shows the TG-DSC data of particles yielded by the Aloe latex powder solid extract. The weight loss percentage and its corresponding heat loss or gain will be beneficial in identifying the molecules present in the samples [39]. In the first degradation stage from 130 to 240ºC, a weight loss of ~54% can be attributed to the evaporation of crystallizing water together with the impurities from phytochemicals present in the sample. In this process, most of the hydrogen molecule together with oxygen evaporates, which will lead to the transformation of magnesium hydroxide into MgO. The thermal degradation of transformation starts at 200ºC, followed by the occurrence of melting transition process. The degradation curve is not that obvious as phytochemicals’ impurities may have reduced the peak of crystallizing water evaporation. The second stage of weight loss which is approximately 4% from 230 to 410 ºC was due to the breaking of bonds and transformation of MgO into another crystallite form. In literature, the second stage weight loss of 3.5% from 250 to 400ºC is attributed to the crystallization of MgO nanoparticles [40]. The third stage weight loss of ~24% at 420 ºC corresponds to the phase change of the MgO. There is a deflection in the DSC curve, corresponds to the weight loss as this may be due to the occurrence of phase change [41]. There is no further significant weight loss was observed at high temperature after 480 ºC. Thus, it is evident that MgO nanoparticles can be formed via calcination and 350ºC for 4 hours was selected as optimum calcination temperature for the MgO formation from TG-DSC curve.
3.8. Functional group analysis of optimized MgO nanoparticles

Figure 7 (A) and (B) shows the FTIR spectra of solid and liquid Aloe latex extract, respectively, whereas Figure 7 (C) and (D) displays the FTIR spectra of MgO nanoparticles formed via the respective extracts. The FTIR spectral peaks present in the phytochemical extracts were depicted in the Table 2. The peaks at 3321.13, 2918.45, 2162.16, 1731.84, 1614.8, 1371.62, 1242.35, and 1316.45 cm⁻¹ in solid extract indicate the existence of N-H stretching of amines, C-H stretches of alkanes, -C≡C- stretches of alkynes, C=O stretches of aliphatic esters, N-H bends of amines, N-O symmetric stretching of nitroalkane, the C-N stretch of aliphatic amines, and C-O stretches of alcohols, carboxylic acids, esters and ethers. Similarly, the peaks at 3277.79, 2929.43, 1715, 1372.18, 1242.02 and 1024.04 cm⁻¹ in liquid extract indicate the existence of O-H stretches of the intramolecular hydrogen bond or phenols, C-H stretches of alkanes, C=O stretches ketones or saturated aliphatic, N-O symmetric stretches of nitroalkane, the C-N stretch of aliphatic amines and C-O stretching of alcohols, carboxylic acids, esters and ethers as reported by Kassama et al. (2015) [42]. The presence of these vibrational groups indicates the presence of phytochemicals such as flavanol, quercetin, Kaempferol, myricetin and fisetin in both extracts [16]. However, the changes in the peak intensity and peak shifts indicates that the quantity and combination of these phytochemical are different in solid and liquid extracts.

In Figure 7 (C) and (D), most of the peaks present in Figure (A) and (B) are absent, which represents the modification of functional group for the formation of MgO. Peaks corresponding to alcohol group, alkyne, primary amine and carboxylic acid are present in the MgO nanoparticles samples which may be formed from the phytochemicals and act as capping agent for the stabilization of nanosized particles. The peaks at 1065.82 and 872.64 Cm⁻¹ is due to the presence of Mg-O vibrational mode in the solid extract yielded particles [26], whereas the peak at 872.64 Cm⁻¹ is miniscule and 1065.82 Cm⁻¹ is too narrow in liquid extract yielded particles [43]. The vibration group related to metal oxide in the liquid extract yielded particles may not be detected by the FTIR spectroscopy as it is suspended in the liquid. Table 3 is the comparison of functional groups present in the extract and MgO nanoparticles. It is evident from Table 3 that the transformation of functional groups in the phytochemicals, during the synthesis is the significant factor that contributes to the formation of nanoparticles.
Figure 7. FTIR spectra of (A) Aloe latex solid extract, (B) Aloe latex liquid extract, (C) MgO nanoparticles from solid extract and (D) MgO nanoparticles from liquid extract

Table 2. FTIR peak values and functional groups of solid and liquid extracts of Aloe latex powder

| Solid extract | Peak values | Functional groups | Liquid extract | Peak values | Functional groups |
|---------------|-------------|-------------------|----------------|-------------|-------------------|
|               | 3321.13     | N-H (amines)      | 3277.79        | O-H stretch (alcohols, phenols) |
|               | 2918.45     | C-H stretch (alkanes) | 2929.43        | C-H stretch (alkanes) |
|               | 2850.27     | C-H stretch (alkanes) | 1715.00        | C=O stretch (ketones, saturated aliphatic) |
| 2162.16       | -C≡C- stretch (alkynes) | 1601.27        | N-H (amines) |
| 1731.84       | C=O stretch (aliphatic esters) | 1372.18        | N-O asymmetric stretch (nitroalkane) |
| 1614.80       | N-H(amines) | 1242.02        | C-N stretch (aliphatic amines) |
| 1371.62       | N-O asymmetric stretch (nitroalkane) | 1024.04        | C-O stretch (alcohols, carboxylic acids, esters, ethers) |
| 1316.45       | C-O stretch (alcohols, carboxylic acids, esters, ethers) | 1242.35        | C-N stretch (aliphatic amines) |
| 1019.72       | C-O stretch (aliphatic esters) | 1019.72        | - |

Table 3. Comparison of functional groups in the extracts and phytochemical extract synthesized MgO nanoparticles

| Frequency (cm⁻¹) | Bond Functional groups | ALSE | ALLE | ALSE synthesized MgO | ALLE synthesized MgO |
|------------------|------------------------|------|------|----------------------|----------------------|
| | | | | | |
| Wave Number (cm⁻¹) | Functional Group | Important Compounds | Present | Absent |
|-------------------|------------------|---------------------|---------|--------|
| 3500-3200         | O-H stretch, H bonded | Alcohols, phenols | -       | +      |
| 3400-3250         | N-H stretch      | Amines              | +       | +      |
| 3000-2850         | C-H stretch      | Alkanes             | +       | -      |
| 2260-2100         | C≡C- stretch     | Alkynes             | +       | +      |
| 1750-1735         | C=O stretch      | Esters, saturated aliphatic | + | - |
| 1715              | C=O stretch      | Ketones, saturated aliphatic | - | + |
| 1650-1580         | N-H bend         | 1° amines           | +       | +      |
| 1360-1290         | N-O symmetric stretch | Nitro compounds    | +       | -      |
| 1250-1020         | C-N stretch      | Aliphatic amines    | +       | +      |
| 1320-1000         | C-O stretch      | Alcohols, carboxylic acids, esters, ethers | + | - |
| 850-550           | C-Cl stretch     | Alkyl halides       | -       | +      |

+= present, -= absent, ALSE = Aloe latex solid extract, ALLE = Aloe latex liquid extract

### 3.9. Morphology analysis of optimized MgO nanoparticles

**Figure 8** shows the transmission electron micrographs of Aloe latex liquid extract (Figure 8 (A), (B) and (C)) synthesized MgO nanoparticles with optimized parameters and Aloe latex solid extract (Figure 8 (D), (E) and (F)) synthesized MgO nanoparticles with optimized parameters and calcinated at 350°C for 4 h. All the images reveal that the morphology of MgO nanoparticles formed via both Aloe extracts were spherical in shape. Figure 8 (A) show that the liquid extract synthesized MgO nanoparticles are agglomerated into a micro-sized spherical structure in the copper mesh grid used for TEM analysis. This emphasizes that the nanoparticles are needed to be dispersed in a more relevant solvent to de-agglomerate them, before using them for further applications. However, the size of the nanoparticle at 50 nm scale is noted to be in the range between 25-35 nm. The micrograph of solid extract synthesized MgO nanoparticles is different from the liquid extract synthesized counterparts, as the crude solid extract has led to the formation of translucent nanoparticles, which makes it tedious for the electrons to transmit through the particles. Thus, centered-dark field was used to analyze solid extract synthesized and calcinated MgO nanoparticles. The calcination provides has led to minimum agglomeration and the size of the nanoparticles are in the range of 39-60 nm. It is noteworthy that the liquid extract synthesized MgO nanoparticles are highly beneficial than solid extract counterparts, with respect to yield, monodispersity, rapid synthesis and less energy requirement aspects. The size of the liquid extract synthesized MgO nanoparticles are similar to our previous reports using different plant leaf extracts [37, 43, 44] and are smaller than the MgO nanoparticles fabricated via chemical synthesis approaches [26, 45, 46].
4. Conclusion

The present work reports the synthesis of MgO nanoparticles via phytochemicals that are extracted in a novel method from Aloe barbadensis latex and magnesium nitrate as precursor. The latex is a pseudo-leaf, which is a unique structure with water content in Aloe and hence, two types are phytochemical extracts such as liquid and solid are obtained from them. Both were utilized for the fabrication of MgO nanoparticles and the parameters such as reaction time, extract ratio (dilution), temperature and precursor concentration. The optimized parameters are 30 min of reaction time, 60°C of reaction temperature and 0.01 M of precursor concentration for both Aloe (solid and liquid) extracts to yield smaller MgO nanoparticles. The optimized liquid extract to aqueous ratio is 1:50, whereas 1:10 ratio of solid extract: water was selected to obtain smaller MgO nanoparticles. The solid extract yielded magnesium hydroxide instead of MgO, which was evident from UV-visible spectroscopy analysis and thus, the calcination was performed at 350°C (4 h) for those samples, where the calcination temperature was selected from the thermogravimetric analysis results. Further, FTIR analysis confirmed the presence of MgO nanoparticles in both liquid and solid extracts synthesized samples along with alcoholic and carboxylic acid functional groups from phytochemicals that acts as reducing and stabilizing agent for the nanoparticle formation. Furthermore, TEM analysis confirmed that the spherical shaped MgO nanoparticles are formed from both extracts, where liquid extract yielded 25-35 nm sized particles and solid extract yielded 39-60 nm sized particles. Thus, the phytochemical extracts from Aloe barbadensis are proven to be beneficial in smaller sized MgO nanoparticles, which will be useful for various biomedical and pharmaceutical applications.

References

[1] Rukh, S., et al., Antibacterial activity of magnesium oxide nanostructures prepared by hydrothermal method. Asian Journal of Nanosciences and Materials, 2019. 2(4): p. 425-430.

[2] Sharmila, G., et al., Green fabrication, characterization of Pisonia alba leaf extract derived MgO nanoparticles and its biological applications. Nano-Structures & Nano-Objects, 2019. 20: p. 100380.

[3] Pudukudy, M., et al., One-pot sol-gel synthesis of MgO nanoparticles supported nickel and iron catalysts for undiluted methane decomposition into COx free hydrogen and nanocarbon.
Food Science & Technology, 2017. Sánchez Reynolds, T., modern uses of the leaf parenchyma gel. (3): p. 437 6 2016. Grindlay, D. and T. Reynolds, Clitoria ternatea—characterization and in vitro antioxidant studies. Sushma, N.J., et al., - p. 471 Mangalampalli, B., N. Dumala, and P. Grover, Toxicity assessment of magnesium oxide nano and microparticles on cancer and non-cancer cell lines. The Nucleus, 2019: p. 1-15. El-Sayyad, G.S., F.M. Mosallam, and A.I. El-Batal, One-pot green synthesis of magnesium oxide nanoparticles using Penicillium chrysogenum melanin pigment and gamma rays with antimicrobial activity against multidrug-resistant microbes. Advanced Powder Technology, 2018. 29(11): p. 2616-2625. Raliya, R., et al., Synthesis of MgO nanoparticles using Aspergillus tubingensis TFR-3. Journal of Bionanoscience, 2014. 8(1): p. 34-38. Sharma, G., R. Soni, and N.D. Jasuja, Phytoassisted synthesis of magnesium oxide nanoparticles with Swertia chirayaita. Journal of Taibah University for Science, 2017. 11(3): p. 471-477. Suresh, J., et al., Green synthesis and characterization of hexagonal shaped MgO nanoparticles using insulin plant (Costus pictus D. Don) leave extract and its antimicrobial as well as anticancer activity. Advanced Powder Technology, 2018. 29(7): p. 1685-1694. Sushma, N.J., et al., Facile approach to synthesize magnesium oxide nanoparticles by using Clitoria ternatea—characterization and in vitro antioxidant studies. Applied Nanoscience, 2016. 6(3): p. 437-444. Reynolds, T., Aloe: the genus Aloe. 2004: CRC press. Grindlay, D. and T. Reynolds, The Aloe vera phenomenon: a review of the properties and modern uses of the leaf parenchyma gel. Journal of ethnopharmacology, 1986. 16(2-3): p. 117-151. Sánchez-Machado, D.I., et al., Aloe vera: Ancient knowledge with new frontiers. Trends in Food Science & Technology, 2017. 61: p. 94-102. Kumar, A., et al., Prevalence and severity of periodontal diseases in type 2 diabetes mellitus of Bareilly region (India). Int J Med Sci Public Health, 2013. 2(1): p. 77-83. Ozsoy, N., E. Candoken, and N. Akev, Implications for degenerative disorders: Antioxidative activity, total phenols, flavonoids, ascorbic acid, β-carotene and β-tocopherol in Aloe vera. Oxidative medicine and cellular longevity, 2009. 2(2): p. 99-106. Tippayawat, P., et al., Green synthesis of silver nanoparticles in aloe vera plant extract prepared by a hydrothermal method and their synergistic antibacterial activity. PeerJ, 2016. 4: p. e2589. Radha, M.H. and N.P. Laxmiyriya, Evaluation of biological properties and clinical effectiveness of Aloe vera: A systematic review. Journal of traditional and complementary medicine, 2015. 5(1): p. 21-26. Pandey, A. and S. Singh, Aloe Vera: A Systematic Review of its Industrial and Ethno-Medical Efficacy. International Journal of Pharmaceutical Research & Allied Sciences, 2016. 5(1). Sangeetha, G., S. Rajeshwari, and R. Venkatesh, Green synthesis of zinc oxide nanoparticles by aloe barbadensis miller leaf extract: Structure and optical properties. Materials Research Bulletin, 2011. 46(12): p. 2560-2566. Kumar, P.P.N.V., et al., Green synthesis of copper oxide nanoparticles using Aloe vera leaf extract and its antibacterial activity against fish bacterial pathogens. BioNanoScience, 2015. 5(3): p. 135-139. Rao, K.G., et al., Green synthesis of TiO2 nanoparticles using Aloe vera extract. Int. J. Adv. Res. Phys. Sci, 2015. 2(1A): p. 28-34. Mehrabian, S., et al., A STUDY OF THE ANTIMUTAGENIC EFFECTS OF DIFFERENT
[25] Hu, K. and D.J. Mc Clemens, *Fabrication of biopolymer nanoparticles by antisolvent precipitation and electrostatic deposition: Zein-alginate core/shell nanoparticles*. Food Hydrocolloids, 2015. 44: p. 101-108.

[26] Jeevanandam, J., Y.S. Chan, and M.K. Danquah, *Calcination-Dependent Morphology Transformation of Sol-Gel- Synthesized MgO Nanoparticles*. Chemistry select, 2017. 2(32): p. 10393 - 10404.

[27] Vatsha, B., et al., *Effects of precipitation temperature on nanoparticle surface area and antibacterial behaviour of Mg (OH) 2 and MgO nanoparticles*. Journal of Biomaterials and Nanobiotechnology, 2013. 4(04): p. 365.

[28] Gopalakrishnan, D., D. Damien, and M.M. Shaijumon, *MoS2 quantum dot-interspersed exfoliated MoS2 nanosheets*. ACS nano, 2014. 8(5): p. 5297-5303.

[29] Sahu, P.K., P.K. Sahu, and D.D. Agarwal, *Role of basicity and the catalytic activity of KOH loaded MgO and hydrotalcite as catalysts for the efficient synthesis of 1-[2-benzothiazolylamino) arylmethyl]-2-naphthalenols*. RSC Advances, 2015. 5(85): p. 69143-69151.

[30] Christensen, L., et al., *Biosynthesis of silver nanoparticles using murraya koenigii (curry leaf): an investigation on the effect of broth concentration in reduction mechanism and particle size*. Adv Mat Lett, 2011. 2(6): p. 429-434.

[31] Ali, K., et al., *Aloe vera extract functionalized zinc oxide nanoparticles as nanoantibiotics against multi-drug resistant clinical bacterial isolates*. Journal of colloid and interface science, 2016. 472: p. 145-156.

[32] Ismail, A.A., et al., *Multilayered ordered mesoporous platinum/titania composite films: does the photocatalytic activity benefit from the film thickness?* Journal of Materials Chemistry, 2011. 21(21): p. 7802-7810.

[33] Ahmadi, M., M.R. Ghasemi, and H.H. Rafsanjani, *Study of different parameters in tio2 nanoparticles formation*. Journal of Materials Science and Engineering, 2011. 5(1): p. 87.

[34] Al-Qubaisi, M.S., et al., *Cytotoxicity of nickel zinc ferrite nanoparticles on cancer cells of epithelial origin*. International journal of nanomedicine, 2013. 8: p. 2497.

[35] Ozel, F., H. Kockar, and O. Karaagac, *Growth of iron oxide nanoparticles by hydrothermal process: effect of reaction parameters on the nanoparticle size*. Journal of Superconductivity and Novel Magnetism, 2015. 28(3): p. 823-829.

[36] Muangban, J. and P. Jaroenapibal, *Effects of precursor concentration on crystalline morphologies and particle sizes of electrosprayed WO3 nanofibers*. Ceramics International, 2014. 40(5): p. 6759-6764.

[37] Jeevanandam, J., Y. San Chan, and Y.H. Ku, *Aqueous Eucalyptus globulus leaf extract-mediated biosynthesis of MgO nanorods*. Applied Biological Chemistry, 2018. 61(2): p. 197-208.

[38] Jeevanandam, J., Y.S. Chan, and M.K. Danquah, *Effect of pH variations on morphological transformation of biosynthesized MgO nanoparticles*. Particulate Science and Technology, 2019.

[39] Mallick, L., S. Kumar, and A. Chowdhury, *Thermal decomposition of ammonium perchlorate—A TGA–FTIR–MS study: Part I*. Thermochimica acta, 2015. 610: p. 57-68.

[40] Jaison, J., S. Balakumar, and Y. Chan. *Sol–Gel synthesis and characterization of magnesium peroxide nanoparticles*. in IOP Conference Series: Materials Science and Engineering. 2015. IOP Publishing.

[41] Topnani, N., S. Kushwaha, and T. Athar, *Wet synthesis of copper oxide nanopowder*. International Journal of Green Nanotechnology: Materials Science & Engineering, 2010. 1(2): p. M67-M73.

[42] Kassama, L.S., A.J. Kuponiyi, and T. Kukhtareva, *Comparative effects of aloe vera (aloe barbadensis) water vs ethanol extracts on the physicochemical properties and stability of silver nanoparticles syntheses*. American International Journal of Contemporary Research, 2015. 5(2): p. 30-39.

[43] Jeevanandam, J., Y.S. Chan, and M.K. Danquah, *Biosynthesis and characterization of MgO nanoparticles using Aloe vera leaf gel and latex using Ames test*. ARAK MEDICAL UNIVERSITY JOURNAL (AMUJ), 2012. 15(2 (61)): p. 100-106.
nanoparticles from plant extracts via induced molecular nucleation. New Journal of Chemistry, 2017. 41: p. 2800-2814.

[44] Hii, S.Y., J. Jeevanandam, and Y.S. Chan, Plant mediated green synthesis and nanoencapsulation of MgO nanoparticle from Calotropis gigantea: Characterisation and kinetic release studies. Inorganic and Nano-Metal Chemistry, 2019. 48(12): p. 620-631.

[45] Jeevanandam, J., Y.S. Chan, and M.K. Danquah, Effect of Gelling Agent and Calcination Temperature in Sol–Gel Synthesized MgO Nanoparticles. Protection of Metals and Physical Chemistry of Surfaces, 2019. 55(2): p. 288-301.

[46] Wong, C.W., et al., Response Surface Methodology Optimization of Mono-dispersed MgO Nanoparticles Fabricated by Ultrasonic-Assisted Sol–Gel Method for Outstanding Antimicrobial and Antibiofilm Activities. Journal of Cluster Science, 2019: p. 1-23.