Production of Calcium Oxide from Eggshell: Study on Calcination Temperature, Raw Weight and Contact Time

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Abstract

Egg waste is a huge hazardous waste in the world, which requires a high cost to treat. The major compound in eggshells is calcium carbonate that can be converted into calcium oxide by the calcination method. This study was conducted to examine what are the ideal conditions that may affect perfect calcium oxide production in terms of temperature, contact time, and weight. The eggshells were pre-treated to eliminate dirt and unnecessary biological substance, ground into powder form, and sieved with 500 μm size. In this study, the decomposition of eggshells to produce calcium oxide (CaO) were conducted at various conditions; (i) constant weight and contact time with different temperatures, (ii) constant temperature and weight with different contact times; and (iii) constant temperature and contact time with different weights. Primary characteristics of eggshell powder such as color changes, mass loss, bulk density, and moisture content, and identification of chemical bonds in a molecule were studied. The result showed significant differences in the color transition of the eggshell powder at parameter temperature and contact time. FTIR result reported that the grey color of calcined samples consists of calcium carbonate while solid white powder consists of metal oxide content. Calcined eggshell powder at a temperature of 900°C can be considered as a broken organic compound due to its high temperature whereas the eggshell powder at a contact time of 6 hours showed that there is calcium oxide corresponded to the wavelength. Thus, it can be concluded that the optimum calcination condition is at a temperature of 900°C with less than 3 hours contact time.

Keywords: *calcium carbonate, calcium oxide, eggshell, calcination*

1. Introduction

Poultry egg is an economical convenient food source due to its high protein contents and nutrients (Johnson & Ridlen, 2019). Rheault et al. (2019) found that both egg white and egg yolk consist of water, protein, carbohydrate, fat, and ash, but differ in percentages of the contents. Egg also consists of essential lipids, proteins, minerals, and low-calorie sources. Egg products are used as components in other foods for a great variety of industrial activities such as thickener, binder, leavening, glazing, and garnishing (Laca et al., 2014).

Eggshell waste is listed in the European Union regulation as hazardous waste and includes an elevated cost of disposal management (Carvalho et al., 2011; Laca et al., 2017; Quina et al., 2017; Ummartyotin & Manuspiya, 2018). Approximately 250,000 tons of eggshell wastes are produced annually worldwide (Verma et al., 2012). Hamidi (2017) reported that approximately 1,200 tons of eggshells were thrown away every single day from various industries. American companies pay up to millions of dollars annually to dispose of eggshells in landfills, and the capacity is reached by the fillings (Sonenklar, 1999). On top of that, many landfill operators do not wish to have eggshell wastes because rats and other vermin are attracted by the protein-rich membrane that adheres to the shell.

Most of the waste and by-products are usually disposed to a landfill without any pre-treatment as it was considered to be useless and has no commercial value (Nor-Hisham et al., 2017). What makes the problem of eggshell disposal even more puzzling is that the shells contain many valuable minerals. There have been many previous studies utilizing the eggshells in various applications such as agriculture (Omu et al., 2005; Tangboriboon et al., 2012; Ok et al., 2015; Arabhosseini & Faridi, 2018), a low-cost catalyst for biodiesel production (Yasar, 2019), construction materials (Beck et al., 2010; Karthick et al., 2014; Gabol et al., 2019; Razali et al., 2020), medical practices (Abdulrahman et al., 2014), and crafts (Hitchcock, 2012; Zoran 2018). Abdullah et al. (2018) studied the effects of eggshell powder (ESP) as supplementary material to produce recycled paper. Since the eggshell is more accessible, it can be classified as one of the sources of calcium for underprivileged rural people who have faced a risk of low calcium intake (Bartter et al., 2018). Eggshells' similar chemical characteristics to commercial calcium carbonate make it can be a potential source of bio-filter (Bashir & Manusamy, 2015).

Calcium oxide (CaO) is a member of the class of calcium oxide of calcium and oxygen in a 1:1 ratio. It occurs in the form of hard lumps, as an odorless, white, or grey-white solid that is a potent skin, eye, and mucous membrane irritant. A study by Verma et al. (2019) revealed that the composition of calcium oxide in conventional Portland cement is 62.91%. Besides being widely used in fertilizers and insecticide applications, calcium oxide is commonly used in the manufacturing of porcelain and glass, in the purification of sugar, in the preparation of whitening powder, calcium carbide, and cyanamide calcium, in water softeners, and mortar and cement. Also, it is used as a liming agent for treating acidic soils in agriculture.

Calcium carbonate ($CaCO_3$) can be converted to calcium oxide through the calcination process. Calcination is a process of extracting volatile compounds, heating solids to a high temperature, oxidizing a portion of the mass, or making them friable (Britannica, 2016). Calcination is often regarded as a purification process. There are two methods of calcination of calcium carbonate: combustion with carbon dioxide/oxygen gas and steam method (Lin et al., 2011). Producing a complete catalyst depends on the temperature used. The catalyst would be completely decomposed at a temperature range of 500ºC to 900ºC (Al-Fatesh & Fakeeha, 2010). In research by Nordin et al. (2015), a higher amount of calcium oxide by the formation of calcium oxide from seashells could be obtained at 800ºC based on Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), Energy Dispersive X-ray spectroscopy (EDX), and Thermal Gravimetric Analysis (TGA) tests. Mohamad et al. (2016) claimed that the calcination was highly dependent on the temperature, heating rate, and particle size. It was observed that the optimum operating condition for the decomposition of calcium carbonate samples is at 900ºC with a 20ºC per min heating rate and 0.3 mm particle size. Therefore, it is established that the temperature of the calcination of calcium carbonate is 800ºC and above.

Three parameters were used in this work to investigate the ideal conditions in the calcination process of converting calcium carbonate from chicken eggshells into calcium oxide. The parameters are weight, contact time, and temperatures. The physical and chemical properties of the calcium oxide studied were color changes, bulk density, moisture content, mass loss, and identification of chemical bonds in a molecule.

2. Objectives

The objective of this study is to investigate the parameters affecting the production of Calcium Oxide (CaO) from the eggshell. Three parameters were observed under the following protocols;

1) Constant weight and contact time with different temperatures
2) Constant temperature and weight with different contact times
3) Constant temperature and contact time with different weights

3. Materials and Methods

3.1 Raw Materials

There are 3 phases involved in preparing the sample namely (i) sample procurement; (ii) pre-treatment, and (iii) sample analysis as shown in Figure 1 (Doh et al., 2017). 1000g of chicken eggshells as a raw material of this study was collected from local food industries. The chicken eggshells were instantly washed with tap water after collected as a preparation for the pre-treatment process.

The second step is the pre-treatment process that includes cleansing, boiling, and drying to eliminate dirt and biological substances (Islam et al., 2013; Ali & Badawy, 2017). This boiling process at 100ºC is necessary to eliminate any pathogens present in the chicken eggshells and avoid decaying of the eggshell samples. Then, the clean chicken eggshell samples were placed in a drying oven at a temperature of 105ºC for 5 hours (Oulego et al., 2019). The chicken eggshell samples were ground using an electrical powder grinder (SY-25, 2000W) to form an eggshell powder (ESP). Then, the ESP was sieved with 500 µm size to eliminate granular sizes. All steps of the preparation were illustrated in Figure 1 below. The treated eggshell samples were then stored in a dark-colored container.

Pre-treated eggshells ESP

Figure 1 Preparation of chicken eggshells samples

3.2 Calcination of Chicken Eggshell

In this study, the calcination process was examined by three variable parameters. The parameters are temperature (T), contact time (C), and weight (W). Table 1 shows the design of the experiment for this study. Each test was done in three replications.

Table 1 Calcination Parameters

Sample	**Parameter / Condition**		
	Temperature (°C)	**Contact Time (hour)**	**Weight (g)**
T_{600}	600	3	20
T_{700}	700	3	20
T_{800}	800	3	20
T_{900}	900	3	20
C_3	800	3	20
C_4	800	4	20
C_5	800	5	20
C_6	800	6	20
W_{10}	800	3	10
W_{20}	800	3	20
W_{30}	800	3	30
W_{40}	800	3	40

3.3 Chemical and Physical Testing

3.3.1 Moisture Content

The temperature for moisture content was referred to a research by Oulego et al. (2019). One gram of pulverized chicken eggshells sample was placed in a drying oven at 105°C for 5 hours. The moisture content was determined by calculating different weights of chicken eggshells before and after the drying process using an analytical weight balanced. The weight loss was determined using Equation 1 below.

$$\text{Moisture} \frac{\text{W-D}}{\text{D}} \text{x100} \quad \text{Equation 1}$$

where;
W = Weight before drying
D = Weight after drying

3.3.2 Bulk Density

Bulk density can be measured by a direct or indirect method. According to Gatea et al. (2018), the core method is one of the direct methods that can be used to measure the bulk density. In this study, the bulk density for raw ESP was determined using Equation 2 below.

$$p \frac{\text{m}}{\text{v}} \text{x100} \quad \text{Equation 2}$$

where;
ρ = Bulk density
m = Mass of raw eggshell
v = Volume of container

The core method (volumetric cylinder method) was used by placing 5 ml of ESP into a 10 ml cylinder container. The weight of raw ESP was determined by using an analytical weight balance.

3.3.3 Compounds Identification - Functional Group Presence

Fourier-Transform Infrared Spectroscopy (FTIR) is used to identify the functional group present in the calcined eggshells of different parameters. The ESP samples have been mixed with Potassium Bromide (KBr) with a concentration range of 0.2% to 1% for the analytical process established by Beers Law.

4. Results and Discussion

4.1 Calcination Process

Calcination is the process of decomposing calcium carbonate by heating it to high temperatures. There are two methods of calcinating calcium carbonate; combustion with carbon dioxide/oxygen gas or steam method (Lin et al., 2011). The catalysts were completely decomposed at the temperature range of 500 ºC to 900 ºC (Al-Fatesh & Fakeeha, 2010). The optimum temperature for the calcination of the calcium carbonate process for the cockle shell is 900 ºC for 2 hours using a particle size of 0.3 mm under inert conditions (Mohamad et al., 2016). This statement is supported by Tangboriboon et al. (2012) that reported the suitable temperature and time for calcium carbonate from duck eggshell to fully decompose is at 900ºC and 1 hour. Loy et al. (2016) and Nordin et al. (2015) argued that the calcite was fully decomposed to form calcium oxide at 800ºC. Based on these studies, three conditions may affect the calcination process. In this study, calcium oxide was derived from ESP as shown in Equation 3 below.

$$CaCo_{3(s)} \rightarrow CaO_{(s)} + Co_{2(g)} \quad \text{Equation 3}$$

Primary characteristics of calcined ESP such as physical observation (color changes), moisture content, bulk density, and average mass loss were studied and recorded. An increase in the calcination temperature from 600℃ to 900℃ showed an increase in percentage average mass loss and decrease bulk density as shown in Table 2. The result shows that the calcination temperature affected the reduction in mass for ESP. Physical changes such as different colors between uncalcined and various calcinated samples had shown. The color of the uncalcined eggshell is light brown due to its original color while the perfect calcined eggshell should be solid white. Based on the result, it is concluded that at low temperatures dark or grey color is produced, and at high temperatures solid white color is obtained. The higher the calcination temperature, the more metal oxide was produced, which indicates that the white became white from the change in color of the ESP. Mohadi et al. (2016) stated a higher temperature will form a higher amount of metal oxide, and the sample color will become solid white. Mohamad et al. (2016) supported the statement by reporting the optimum temperature for decomposing calcium carbonate at 900ºC. High temperatures are required to calcine ESP to produce the perfect calcium oxide. The dark powder produced at a low temperature indicates that ESP has not decomposed completely.

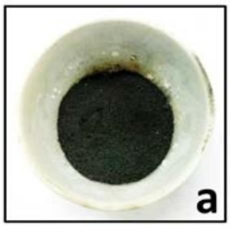
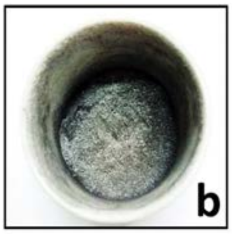
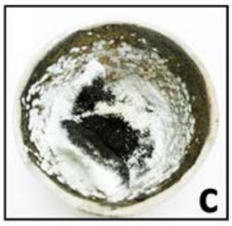
Samples calcined at different contact times showed a huge percentage of mass loss as compared with other manipulated variables. This could be explained whereby the longer the calcination duration, the greater the mass loss (Krähenbühl et al., 2016). The color transitions for different contact times were illustrated in Table 2. Even though the samples ESP C_3 to ESP C_6 calcined at constant temperature and weight, the contact time affected the calcination result. The formation of dark grey powder at a low contact time can be concluded as a lack of heat distribution. The grey powder at the inner part of the sample showed that the heat is not well distributed throughout the sample. Therefore, increasing the calcination period is one of the significant ways to increase the formation of metal oxide. Tangboriboon et al. (2012) stated that the higher the temperature and longer calcination time, the more complete calcium oxide obtained.

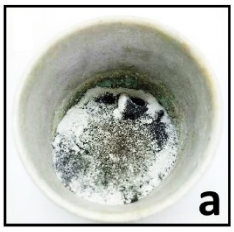
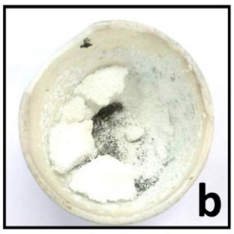
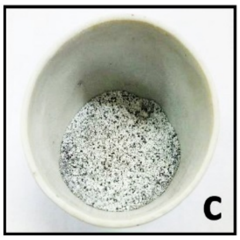
There is no color change in the samples ESP W_1, ESP W_2, ESP W_3, and ESP W_4 as shown in Table 2. The weight of the samples does not influence the decomposition of calcium carbonate. The dark powder of the sample indicates that the carbonation process has not been completed yet. The formation of the white powder also indicated the presence of metal oxide, whereby the greater amount of the white powder means the more metal oxide formed (Mohadi et al., 2016).

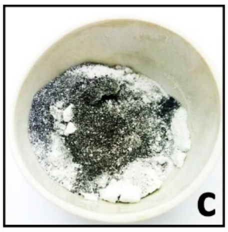
Moisture loss has occurred during the calcination process, which corresponds to the loss of water and/or gas. In this study, the moisture loss percentages of the calcined ESP in all conditions were too insignificant and considered negligible. Da Silva et al. (2019) reported a loss of volatile materials such as water and organic matter at a low temperate range of 30ºC to 400ºC. There is no change in acidity for raw ESP and dark powder. However, the result shows acidity for the solid white powder that changed to alkali. Therefore, if the amount of the dark powder is greater than the solid white powder, the sample can be considered as calcium carbonate. The result of the whole study is summarized in Table 3 below.

The bulk density of calcination of ESP with different parameters was tabulated in Table 2. The solid white powder at T_{900} and C_6 showed a softer and finer texture. Based on the result, the end particle size of the sample was influenced by the temperature and contact time. The statement is supported by Tangboriboon et al. (2012) which reported that the calcination at 900ºC for 1-hour decreases the particle size of the samples. The particle size of the samples will influence their density. According to Equation 2, the bulk density is inversely proportional to the volume. The bulk density decreased as the particle size was increased (Li et al., 2019).

Table 2 Calcination of ESP with different parameters

ESP Sample	Physical Observation	Mass Loss (%)	Moisture Content (%)	Bulk Density (g/ml)
Uncalcined		6.6	1.1	1.20
T_{600}		5.82	-	1.19
T_{700}		8.10	-	1.37
T_{800}		24.56	-	0.90
T_{900}		30.15	-	0.35

C_3		22.03	-	0.90
C_4		31.60	-	0.84
C_5		34.33	-	0.84
C_6		44.44	-	0.83
W_{10}		23.93	-	0.81

W_{20}		27.69	-	0.90
W_{30}		28.01	-	0.87
W_{40}		24.08	-	0.88

The summary of the result in Table 2 is summarized in Table 3. The calcination of the eggshell powder shows a significant difference in the mass loss for temperature and contact time parameters. The FTIR test result shows calcium oxide presents at a higher temperature and longer contact time, however, there is no presence of calcium oxide in dark grey powder.

Table 3 Characterization of Calcium Oxide

Dependent Variable	Mass Loss	Physical Appearance	Presence of Calcium Oxide
Weight	No significant difference	Dark grey powder	No
Temperature	There is a significant difference	Higher temperature leads to a whiter powder	Yes
Contact time		Increased contact time leads to a whiter powder	

4.2 Compounds Identification

The FTIR analysis can be divided into three broad regions where the first Region from 4000 to 3000 cm^{-1} revealed a hydrogen bonding, Region II from 3000 to 1500 cm^{-1} showed the presence of functional groups, and Region III revealed the information regarding the existence of bio-minerals. Figure 2 shows the FTIR analysis for raw ESP after pre-treatment.

The chemical characterization is conducted for the selected samples of ESP T_{600}, ESP T_{900}, ESP C_3, ESP C_4, ESP C_5, and ESP C_6. The FTIR analysis is conducted to identify the functional group present in the calcined eggshells of different parameters. Aiming at the optimum condition for the calcination of the eggshell, the FTIR spectra are analyzed for the difference in the calcination period and calcination temperature as the significant variables in this study.

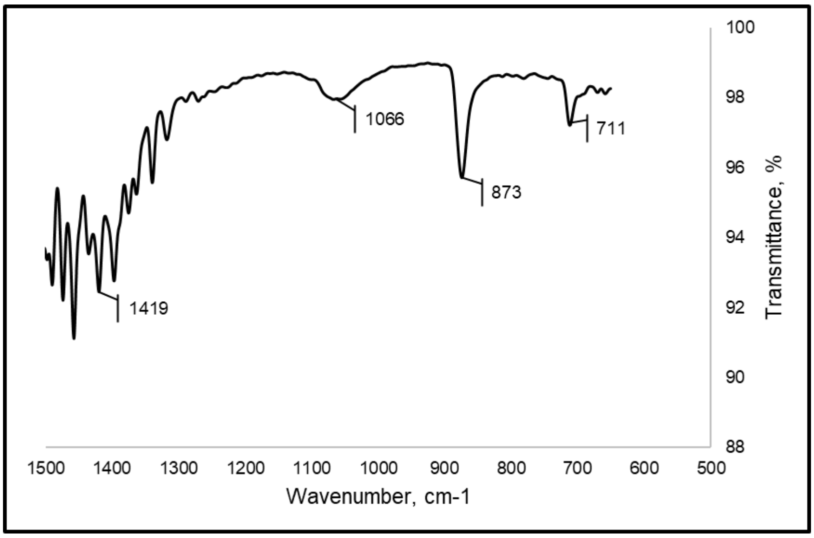
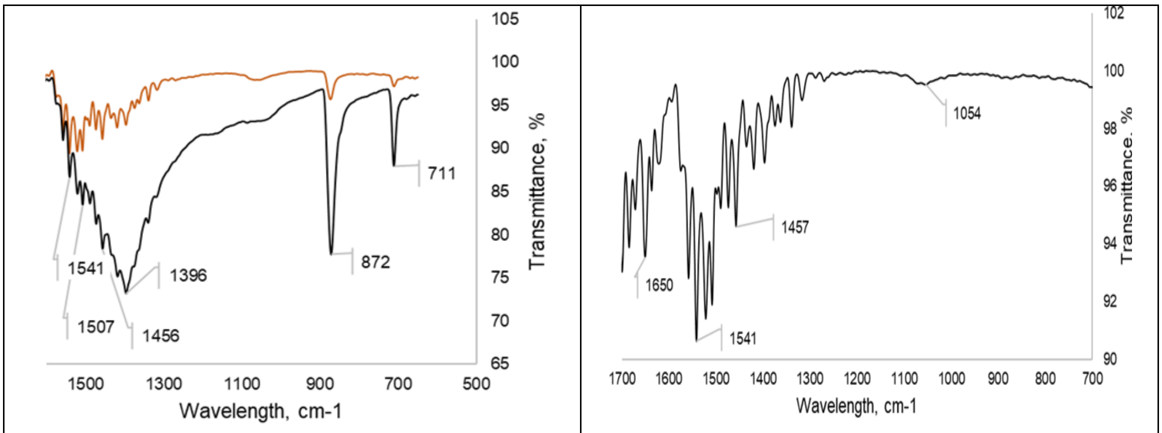
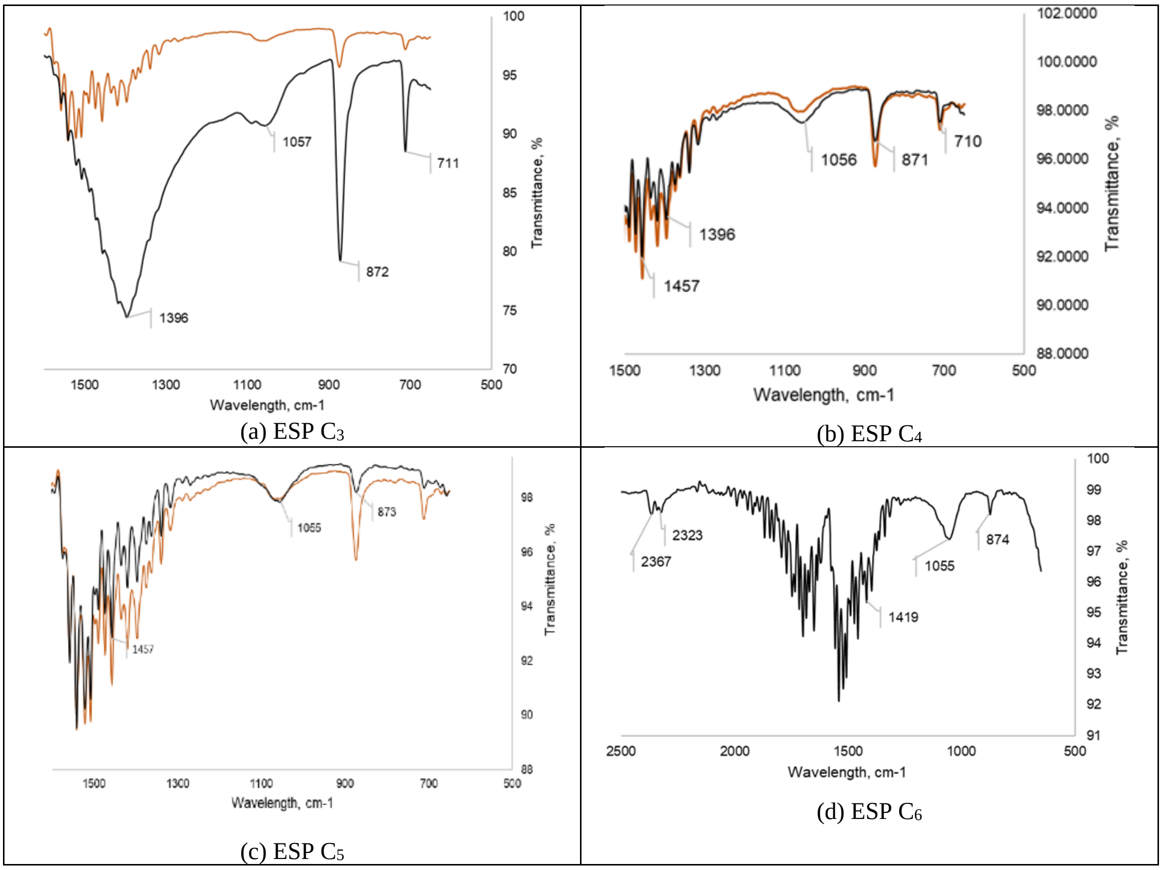


Figure 2 FTIR analysis for raw ESP



(a) ESP T_{600} (b) ESP T_{900}

Figure 3 The FTIR spectrum on calcined ESP at different temperatures



(a) ESP C_3 (b) ESP C_4 (c) ESP C_5 (d) ESP C_6

Figure 4 The FTIR spectrum on calcined ESP with different contact time

Figure 2 showed the FTIR spectrum for raw ESP. Sharp small peaks were detected at 711 cm^{-1} and 873 cm^{-1}, which is slightly higher in terms of wavenumber. The bands at 1419 cm^{-1}, 873 cm^{-1}, and 712 cm^{-1} indicated the presence of calcium carbonate in the tested samples. According to Carvalho (2011), the observable peaks between 713 and 875 cm^{-1} should be associated with the presence of calcium carbonate. Observation by Iram et al. (2019) reported that the bands concerned with calcium carbonate are 1430 cm^{-1}, 875 cm^{-1}, and 715 cm^{-1}, where the lowest band is considered as a weak band to indicate the presence of calcium carbonate. Another wide range is also detected at 1396 cm^{-1}, indicating the presence of -OH bending variation. Formation of -OH function is common during the calcination with the function of synthesizing calcium oxide (Zaman et al., 2018).

FTIR spectrum of ESP T_{600} and ESP T_{900} are presented in Figure 3. A sharp peak was observed at 711 cm^{-1}. The wavenumber increased as the transmittance increases intensely at a peak of 872 cm^{-1}. Both peaks are associated with the in-plane deformation and out-plane deformation modes, respectively, in the presence of calcium carbonate as described by Carvalho (2011). A small band detected at 1054 cm^{-1} is associated with the presence of amine and amide according to Cheng et al. (2016). The presence of the C-H bond was also detected at the peak at 1650 cm^{-1}. As described by Nordin et al. (2016), higher calcination temperature and time yield a higher percentage of calcium oxide content.

The FTIR analysis conducted at ESP C_3 and ESP C_4 showed the presence of calcium carbonate, which indicated that the inorganic compound has not yet fully transformed into its simplest form. The band detected at ~875 cm^{-1} showed a strong peak of calcium carbonate meanwhile another at ~715 cm^{-1} showed a weak presence of calcium carbonate (Iram et al., 2019). At ESP C_5, the band detected at 873 cm^{-1} is associated with the presence of calcium carbonate. As referred to Fajrah (2014), the wavenumbers of 1405 cm^{-1}, 873 cm^{-1}, and 712 cm^{-1} correspond to the Ca-CO group.

While an amine group is present at band 1055 cm^{-1}, Tsai et al. (2006) stated that the presence of functional groups of amines and amides was observable because of their chemical composition of fibrous proteins. Referred to Baitimirova et al. (2013), ESP C_6 showed that a medium-sized peak at 1055 cm^{-1} corresponds to stretching of both C-O in the C-OH bond. The detection of the peak at 1419 cm^{-1} showed the presence of OH bonding. The existence of hydroxide might be due to the exposure to the atmosphere during the calcination (Zaman et al., 2018). Other significant peaks were detected at 2323 cm^{-1} and 2367 cm^{-1}, both are associated with the presence of O=C=O bonds. The presence of O=C=O bonds indicated the carbonation of calcium oxide as shown in Equation 3 (Habte, 2020).

$$Ca(OH)_2 + CaCO_3 + H_2O \qquad \text{Equation 4}$$

5. Conclusion

Referring to the FTIR spectra in Figure 3, ESP C_6 has the properties of calcium oxide, in which the peak at 874 cm^{-1} is associated with the presence of Ca-O (Putra et al., 2017). Meanwhile, at ESP T_{900}, the peak at 1054 cm^{-1} is associated with the presence of amine and amide according to Tsai et al. (2016), however, the band is related to the C-O bond. As described by Tsai et al. (2016), higher calcination temperature and time yield a higher percentage of calcium oxide content. Tangboriboon et al. (2012) reported the complete calcination occurs at 900ºC after 1 hour whereas Lani et al. (2019) claimed that the optimum condition for the production of calcium oxide is at a temperature of 900ºC for 3.5 hours. In conclusion, the production of calcium oxide from eggshell powder depends on the calcination temperature and contact time, while weight yields no significantly different result. Therefore, the optimum conditions are the calcination temperature of 900°C, the calcination period of fewer than 3 hours, and 20 g of the sample, compared with other parameters.

Calcium oxide synthesized from eggshell powder will enhance waste management by managing and recycling them in the proper disposal despite the huge amount generated by food industries, and eventually, will reduce landfill waste. Other than that, it can also be used as a dielectric material, a dry agent for ammonia gas, an absorbent, a bleaching powder, used in the construction industry, and more. Furthermore, the use of natural minerals will be reduced, which, in turn, will preserve the environment

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