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Co-Precipitation (Synthesis) of a Catalyst (Cao Nanoparticle) from a Household Waste from Gombi L. G. A, Adamawa State Nigeria

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Abstract - A usefull catalyst calcium oxide (CaO) nanoparticle was synthesized from a chicken eggshell using the means of Chemical Co-precipitation of the wet chemical synthesis method and this process basically be classified under the Bottom-Up process of production of nanopartiles. This shows the utilization and recycling of household wastes and the recycling process may also be performed on other household wastes Thermogravimetric analysis shows that the synthesized particle is thermally stable, 50% of the particle show perfect stability even at temperature of 950°C. FTIR analysis of the synthesized particle shows a very limited signals on the absorption band and this clearly indicates the total conversion of the main components of eggshell ($CaCO_3$) into CaO nanoparticle.

Keywords - Co-Precipitation, CaO Nanoparticle, Catalyst, Household West from Gombi L. G. A.

I. INTRODUCTION

A. *The Nature of Catalysis*

Catalysts are (chemical) individuals that, in the ideal case, are specific in their action, highly active, and have long useful lifetimes. The specific activity encompasses region-selectivity and stereo-selectivity, this means that, in a chemical reaction for synthesis or transformation, the catalyst selects specific binding sites (constitution) and at the same time allows only a particular spatial arrangement (conformation, configuration).

Control of merely the constitution may be sufficient for simple molecules such as, e.g., the oxidation of ethylene to ethylene oxide or acetaldehyde. On the other hand, the synthesis of typical life-science products involves an additional high steric requirement that is normally only fulfilled by catalysts with a tailored spatial structure (Carnerd *et al*., 2016).

It is just this distinction between the mere linking of atoms or groups and the precise spatial order (stereocontrol) that recommends catalysts as highly intelligent tools for the design of the material world. Catalysts are (chemical) individuals that accompany a catalytic process by having a major, even decisive, influence on its course without themselves undergoing any change thereby. Catalysts thus have a secure identity. Accordingly, catalysts are not without a profile; on the contrary, a special reaction profile in combination a steric profile is the essential condition in order that other substances can meet at the active center to join together or to undergo other transformations. The better the fit for the reaction partners involved, the more specific is the action of the catalyst. The lifetime of the catalyst, as the

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period in which it expresses its specific activity, depends on whether it emerges intact from each reaction unit (catalyst cycle), i.e., whether it has retained its original structure and reactivity characteristics. The expert describes the degree of activity of a catalyst as its *t*urn *o*ver *n*umber (TON) while the catalytic efficiency is described as the *t*urn *o*ver *f*requency (TOF). Today, a complex pharmaceutical precursor requires a TON in the range 103 to 105. This means that one-unit amount of catalyst (mol) produces the corresponding number of mols of product. In the case of TOF this productivity is referred to the unit of time (Carbral *et al.,* 2013).

In daily life we often hear that something is catalyzed or that someone must act as a catalyst. Even persistent catalysis is mentioned when a complex process must be kept in motion. It is always people, individuals with special talents, who bring others together and ensure that progress is made. And we know how important the results of such an activity can be. Although the major company mergers of today require analysts they do not make any progress without catalysts either at the start or during of the new company philosophy. The same is true for politics, nothing happens without catalysts whereas with catalysts the chemistry works (Wang *et al.,* 2015).

B. Protagonists of Change

Catalysis is ubiquitous. It is exemplified by life since the very beginnings of life. There is hardly any biological process that can proceed without its catalyst, if only to ensure reaction specificity, in other words to maintain the variety of life. The best known biocatalysts are the enzymes which, depending on their function, are employed by nature in metal-free or metal-containing forms. Most essential metals, for example, zinc, are components of enzymes and function therein as reaction centers, but they also influence the structures of the enzymes themselves. This shows once more just how important the spatial structural arrangement of a catalyst is for its activity. One interesting example from nature is the defense and survival strategy of the bombardier beetle. Under its stable chitin shell Brachynus explodans carries a mixture of *p*-hydroquinone and hydrogen peroxide. This mixture is metastable, i.e., the two components do not react with each other in the absence of an external stimulus. When the bombardier beetle feels itself to be endangered it adds two catalysts, an oxidase and a catalase, from its glandular system. Hydroquinone is then oxidized explosively while loud noise of the steam emitted frightens the natural enemy away (Waseda *et al.,* 2011).

Although aimed at a general understanding, the above description already gives an idea of the enormous potential of catalysis. Practically all chemical reactions, provided that they are indeed thermodynamically allowed, can be realized with the help of suitable catalysts. The wide variety of the material world still lies in front of us, not behind us. On the other hand, the search for specific, efficient catalysts becomes more and more difficult the higher our expectations with regard to the activity, the specificity, and the lifetime of a catalyst are. From an economic point of view, a catalyst may be expensive and have a short lifetime when it enables the production of a correspondingly high-value product. The ecological advantage is then largest when the use of a catalyst avoids the formation of by-products that are highly toxic or that have no use in other process cycles. Of course, from an ecological point of view, catalysts that achieve the chemical disposal of mass products are particularly sought after; these include numerous organic plastics, above all, polyethylene, polypropylene, and polyvinyl chloride (PVC) (Conerd *et al*., 2016).

C. Heterogeneous catalytic theory

In general, it is believed that the entire surface of the solid catalyst is not responsible for catalyzing any reaction. Only certain sites on the catalyst surface actually participate in the reaction and these sites are called active sites on the catalysts. These sites may be the unsaturated atoms resulting from surface irregularities or atoms with chemical properties that enable the interaction with the adsorbed reactant atoms or molecules. Activity of the catalyst is directly proportional to the number of these active sites available on the surface and is often expressed in terms of turnover frequency. Turnover frequency is defined as the number of molecules reacting per active site per second at the condition of experiments (Gelsoian *et al*., 2014).

D. Production of heterogeneous catalysts

The development of heterogeneously catalyzed reactions for the production of chemicals initiated the preparation of the required catalysts on a technical scale. Up to the end of World War II, solid catalysts were produced predominantly in process companies such as IGFarben and BASF in Germany, and Standard Oil Company and UOP in the USA. About ten years later some independent catalyst producing companies were founded in the USA, Western Europe, and Japan. At present many international companies are producing solid catalysts on multitoned scale; for example:

Synetix (ICI Catalysts and ICI Catalco) and Davison Chemicals and Grace (Elschenboich, 2006).

Approximately 24 – 28% of produced catalysts were sold to the chemical industry and 38 – 42% to petrochemical companies including refineries. 28 – 32%of solid catalysts were used in environmental protection, and 3 – 5% in the production of pharmaceuticals. The catalytic properties of solid catalysts are affected by the preparation method, production conditions, and quality of source materials (Helmut *et al.,* 2005). Therefore, it is necessary to control each production step and the physical or mechanical properties of all intermediates. To attain a better reproducibility of catalyst production, batch procedures were mainly replaced by continuous operations, such as precipitation, filtration, drying, calcination, and forming. Automation of various operations and computer control of different equipment were installed in catalyst production lines. Recently, SPC (statistical process control) and QA (quality assurance) were integrated into the catalyst production process. Catalysts applied in several industrial processes can be subdivided into the following categories: Unsupported (bulk) catalysts and Supported catalyst (Nelson *et al*., 2000).

E. Preparation of supported catalysts

The broad application of supported catalysts in industrial catalysis led to the development of numerous preparation methods applicable on a technical scale. Some of these methods are identical with those used in the production of unsupported catalysts, e.g., mechanical treatment, precipitation, thermal decomposition of metal – inorganic or metal – organic complexes and therefore they will be discussed here very briefly. Mechanical treatment, e.g., kneading of a catalyst precursor with a support is applied, e.g., in the production of kieselguhr-supported Ni (precursor $NiCO_3$) (Helmut *et al.,* 2005).

Also, MoO_3 supported on Al_2O_3 is sometimes produced by this process. However, the distribution of the active phase on the support is in some cases not sufficient.

Better results are obtained by the combination of mechanical and thermal treatment which, results in spreading of, e.g., MoO_3 on Al_2O_3 or of V_2O_5 on Al_2O_3 or TiO_2 (Max, 2010). Impregnation by pore filling of a carrier with an active phase is a frequently used production method for supported catalysts. The object of this method is to fill pores of the support with a solution of the catalyst precursor, e.g., a metal salt of sufficient concentration to achieve the desired loading. If higher loadings with active phases are required, it is mostly necessary to repeat the impregnation after drying or calcination of the intermediate. Examples of catalysts prepared by the pore filling method are Ni or Co on Al_2O_3 – MoO_3, MoO_3 on aluminosilicates including zeolites, Ni or Ag on a-alumina, noble metals on active carbon, etc.

Adsorption is a very good method to achieve uniform deposition of small amounts of active component on a support. Powders or particles exposed to metal salt solutions adsorb equilibrium quantities of salt ions, in accordance with adsorption isotherms. Adsorption may be either cationic or anionic, depending on the properties of the carrier surface. For example, alumina (depending on the adsorption conditions, mainly on the pH of the solution) adsorbs both cations and anions. Silica weakly adsorbs cations, while magnesia strongly adsorbs anions (Max, 2010).

Adsorption of $PdCl_2$ from aqueous solution on different aluminas is very fast, and a high equilibrium concentration (2ww%) can be obtained. The Pd deposition takes place mainly in an outer shell (egg shell profile) of shaped particles. With $H_2[PtC_{l4}]$ only 1 wt% Pt loading on alumina is possible owing to from the flat adsorption isotherm. The addition of oxalic, tartaric, and citric acid to the metal salt solution changes the profiles of active component on the carrier. In general, with increasing acid strength the metal ions are forced deeper into the support particles (Keith *et al*., 2002).

F. Reduction, Activation, and Protection

Reduction, activation, or protection, is in several cases the last step in catalyst production. These operations are performed by the catalyst producer or in the plant of the client. For example, in the production of Ni catalysts for the liquid-phase hydrogenation of fats and oils, the reduction of NiO deposited on kieselguhr is carried out exclusively by the catalyst producer (Max, 2010). The reduction of powders (50 – 500 m particle size) is performed on an industrial scale in fluid-bed reactors. The reduced material is pyrophoric and must be protected with a hard fat such as tallow, to make its handling easy and safe. The finished catalyst is supplied in the form of flakes or pastilles (Grasselli, 2014).

The reduction of metal oxides such as NiO, CuO, CoO, or Fe_2O_3 is carried out with H_2 at elevated temperature (470 K) and has two steps. In the first step metal nuclei are formed.

In the second, nuclei accumulate to form metal crystallites. The rates of both processes depend on temperature and on the nature of the substrate. Reduction at lower temperatures (<570 K) provides a narrow distribution of small metal crystallites. Reduction at higher temperatures (> 670 K) gives a broader distribution and larger metal crystallites. Reduction of some oxides, such as those of Cu and Fe, is exothermic and needs to be carried out carefully with H_2 diluted with N_2. Water, the reduction product, has negative effects on the rate and on the extent of reduction. In industrial practice, where H_2 is recycled, the removal of water by freezing out (below 270 K) and by adsorption on molecular sieve is essential (Getsoian *et al*., 2014).

To achieve optimal activity, partial reduction of oxidic catalysts is common. For example, Ni catalysts for fat and oil hydrogenation contain about 50 – 60 wt% of metallic Ni, 45 – 35 wt% NiO, and about 5 wt% Ni silicate (Cuenya, 2010). When the reduction of shaped oxidic catalysts is conducted by the catalyst producer, then the active material is protected either with a high-boiling liquid such as higher aliphatic alcohols or C_{14} –C_{18} paraffin or it is passivated. In this procedure, chemisorbed hydrogen is removed in a gas stream composed of N_2 and about 0.1 – 1.0 vol% of O_2 at ambient temperature. After this treatment, catalyst can be handled in air without any precautions. The activation of hydrotreating catalysts composed of Ni- or Co-promoted MoO_3 –Al_2O_3 is carried out with H_2 containing 10 Vol% of H_2S (Max, 2010). In the past, this activation was performed exclusively in the hydridesulfurization plants. However, presulfiding at catalyst producers is becoming more common. Mainly electrical or gas-heated shaft reactors are used for the reduction of extrudates, spheres, or pellets in plants of catalyst producers. Catalyst forming the size and shape of catalyst particles depend on the nature of the reaction and on the type of applied reactor (Max, 2010).

Reactions in the liquid phase require small particles or even powders (50 – 200 m). Such materials are made by grinding of a dried or calcined precursor, e.g., filter cake, using granulators equipped with sieves to give uniform particle size. Catalysts for fluidized bed reactors (0.05 –0.25 mm) are usually made by spray drying or by cooling molten material droplets (V_2O_5) in an air stream.

Spheres consisting of Al_2O_3, SiO_2, aluminosilicate with 3 – 9 mm diameter are used preferentially as a support for catalysts in moving-bed or ebullating-bed reactors. They are produced by the so-called oil-drop method. Spheres prepared in this way possess sufficient abrasion resistance (Chizallet *et al.,* 2006).

Another method for producing spheres is based on agglomeration of powder bymoistening on a rotating disk (spherudizer). As the spheres reach the desired diameter they are removed automatically and transported to the dryer and calciner. Such spheres are suitable for fixed bed reactors. Other methods for forming spherical particles include tumbling short, freshly extruded cylinders in a rotating drum. In the briquetting technique, the plastic mixture of catalyst powder with a binder is fed between two rotating rolls provided with hollowed-out hemispheres.

Extrusion of pastes containing catalyst powder, binders and lubricants is a frequently used industrial shaping method. Depending on the properties of the paste, press or screw extruders are applied. Press extruders are principally suitable for viscous pastes (Cuenya, 2010).

Screw extruders are preferred for thixotropic masses. In both cases, pastes are forced through a die, and the extruded material is cut with a special device to a desired length and falls onto a moving belt that transports it through a drier or calciner. Poly(vinyl alcohol), powdered stearine, and Al stearate are used as lubricants. If the mass being extruded contains alumina, then peptizing agents such as nitric acid is added mainly to improve the mechanical strength. Another type of binder is calcium aluminate cement, which sets up by treatment with steam. The extruded material can have different shapes, such as cylinders (noodles), hollow cylinders (macaroni), or ribbed cylinders. The sizes depend on the shape and are in the range of 1.5 – 15 mm diameter (Max, 2010).

Added organic lubricants and pore-forming agents can be removed by calcination in a stream of air. Special extrusion techniques and equipment are necessary to produce honeycombs. Extruding is less expensive than pelletizing, but extrudates have less resistance to abrasion than pellets. Extrudates are suitable for different types of fixed-bed reactors operating in the gas or trickle phase. Pelletizing is a very common method for catalyst forming. It is based on compression of a certain volume of powder in a die between two moving punchers, one of which also serves to eject the formed pellet. Depending on the size and the shape of the prepared pellets, the material being pelletized must be crushed and forced through a corresponding sieve. Furthermore, lubricants such as graphite, Al stearate, pol(vinyl alcohol), kaolin, and bentonite are added before the material enters the tabletting machine (Cuenya, 2010). The fluidity of the material is required to assure homogeneous filling of the die. As in the case of extrudates, organic lubricants can be removed by calcination of the

pellets. Industrial pelleting machines are equipped with around thirty dies and produce about 10 liter of pellets per hour or more, depending on their shape and size. Pressures in the range 10 –100 MPa in the pelleting machine are common (Thomas *et al*., 2014).

G. Characterization of Solid Catalysts

Catalytic activity and selectivity critically depend on the morphology and texture, surface chemical composition, phase composition, and structure of solid catalysts. Therefore, many physical and chemical methods are used in catalysis research to characterize solid catalysts and to search for correlations between structure and performance of catalysts. These methods include classical procedures as well as techniques developed more recently for the study of the chemistry and physics of surfaces (Chizallet *et al.,*2006).

H. Physical properties

1. Surface area and porosity

The specific surface area of a catalyst or support (m^2/g) is determined by measuring the volume of gas, usually N_2, needed to provide a monomolecular layer according to the Brunauer – Emmett – Teller (BET) method. In this approach, the determination of the monolayer capacity is based on the physisorption of the test gas. The volume adsorbed at a given equilibrium pressure can be measured by static methods, namely, volumetric or gravimetric measurements. Flow or dynamic techniques are also applied. The total surface area of a porous material is given by the sum of the internal and external surface areas. Pores are classified as microspores (pore width < 2 nm), mesoporous (pore width 2 – 50 nm), and macrospores (pore width > 50 nm) according to IUPAC definitions (Thomas *et al.,* 2014).

2. Structure and morphology

X-ray powder diffraction (Structure Analysis by Diffraction – Diffraction by Polycrystalline Specimens) (XRD) is a routine technique for the identification of phases present in a catalyst. It is based on the comparison of the observed set of reflections of the catalyst sample with those of pure reference phases, or with a database (Powder Diffraction File (PDF) distributed by ICDD, the International Centre for Diffraction Data). XRD studies can now be carried out in situ on the working catalyst, and the use of synchrotron radiation permits dynamic experiments in real time. Time-resolved studies on a timescale of seconds are now becoming possible. Quantification of phase compositions can also be performed. More sophisticated analysis of the diffraction patterns of crystalline materials provides detailed information on their atomic structure. The Rietveld method is used for structure refinements. Perhaps more importantly for catalytic materials, the local atomic arrangement of amorphous catalysts is based on the Debye equation, which gives the intensity scattered by a collection of randomly distributed atoms (Max, 2010). The Fourier transform of the Debye equation gives the radial distribution function (RDF) of electrons, from which the number of atoms (electrons) located in the volume between two spheres of radius r around a central atom, i.e., the radial density of atoms, can be obtained. This approach has been applied for the structural analysis of amorphous or poorly crystalline catalyst materials and of small metal particles (Li *et al*., 2010).

X-ray Absorption Spectroscopy (XAS), is the method of choice where the applicability of XRD for structure analyses ceases to be possible. Because of their high photon flux, synchrotron facilities are the preferred sources for XAS experiments. The physical principle of XAS is the ejection of a photoelectron from a core level of an atom by absorption of an X-ray photon. The position of the absorption edge gives the binding energy of the electron in the particular core level and is thus characteristic of the respective element and its chemical state (Sau *et al*.,2010).

When electrons penetrate through matter in an electron microscope, contrast is formed by differential absorption (amplitude contrast) or by diffraction phenomena (phase contrast). Electron micrographs of catalyst materials can provide for identification of phases, images of surfaces and their morphologies, and elemental compositions and distributions. Image interpretations are often not straightforward and need expert analysis (Mondloch *et al.,* 2012).

I. Chemical properties

A. Surface chemical composition

The atomic composition of a catalyst surface plays a decisive role for the catalytic properties. Electron and ion spectroscopies are surface-sensitive analytical tools which provide information on the atomic composition within the topmost atomic layers. The information depth, i.e., the number of atomic layers contributing to the measured signal, depends on the method. Concentration profiles can be obtained by sputter etching of the surface by ion bombardment. The application of these particle spectroscopies requires ultrahigh-vacuum (UHV) conditions (Niu *et al.,* 2011).

The basis for the identification of atoms on surfaces of solid materials by electron spectroscopies, such as Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS) are the electronic binding energies. With ion spectroscopies, such as low-energy ion scattering (LEIS) and Rutherford backscattering (RDS), surface atoms are identified by their nuclear masses. Ion bombardment of surfaces is accompanied by sputtering processes (surface etching) which lead to the removal of secondary ionic and neutral particles. These are analyzed by mass spectroscopic techniques, such as secondary ion mass spectroscopy (SIMS), and secondary neutral mass spectroscopy (SNMS) (Mondloch *et al.,*2012).

II. MATERIALS AND METHODS

A. *Materials*

Eggshell, Nicolet IR 100 FTIR Spectrometer, Deionized water, stirrer, measuring cylinder, beakers, weighing balance, heating mantle, oven etc. are the materials to be used. Some of the reagents to be used includes; distilled water, methanol, sodium sulphate etc., was obtained from the laboratory, Adamawa state University Mubi, Adamawa state, American University Yola, Nigeria.

B. *Sample location*

Egg shells ware collected from the household west and the local tea vendors in Gombi town, central senatorial zone of Adamawa State of Nigeria, it was properly washed three times with tap water to remove the unwanted materials adhered on the surface and properly rinsed twice with distilled water, it was then dried under shade at room temperature.

C. *Sample preservation*

The sample was collected in a container and preserved according to the standard method of American Public Health (APHA).

D. *Quality assurance*

All reagents used were of pure analytical grades. Contamination was thoroughly checked by running a blank of all determinations. Glass apparatus used were soaked in a clean soap solution and in H_2SO_4/HNO_3 mixture and then rinsed with distilled water.

E. *CaO Catalyst preparation*

Highly active CaO was prepared by the calcinations-hydration-dehydration treatment of the eggshell. The dried eggshell was reduced to the smallest size and calcined in a muffle funnance under static condition at 900^0C for 5 hours, this is believed to transformed the calcium species ie $CaCO_3$and other substances in the eggshell in to CaO and can be denoted as eggshell – CaO – 900 (Niju *et al*.,2014)

F. *Synthesis of CaO nanoparticles catalyst*

A simple and cost effective synthetic route was adopted to synthesize inorganic metal oxide nanoparticle using thermal decomposition/chemical co-precipitation. In this process drop by drop addition of an aqueous sodium hydroxide (NaOH) in 20ml solution of a calcined chicken eggshell which resulted in the supersaturated aqueous solution of CaO, under vigorous stirring at 60°C on a magnetic stirrer for 60 minutes. This form a colloidal solution of calcium hydroxide (Ca(OH). The obtained white colloidal solution of calcium hydroxide ($Ca(OH)_2$) was washed in an alcoholic suspension to remove sodium and enhanced its stability due to a lower degree of agglomeration. The resulting suspension was passed across 125 mm Whitman filter paper and dried at 60°C in an oven for 4 hours. These nanoparticles were then stored in a desiccator box for use. The ionic equation of the reaction can be predicted to be as followed

$$6Ca^{2+} + 12OH \rightarrow 6Ca(OH)_2 \rightarrow 6CaO + 6H_2O.$$

G. *Procedure for TG FT-IR determination*

In the TG-FTIR experiment, Sample was placed in a ceramic crucible in a thermogravimetric analyzer (PerkinElmer, STA8000). The sample (33.350 mg) was heated from 30 ^{o}C to 1000 °C. The heating procedure goes as follows;

First, it was heated from 30^{o}C to 600^{o}C at once 20^{o}C/min. and was halt constantly at that 600^{o}C for 10min. it was again heated from 600^{o}C to 700^{o}C at interval of 20^{o}C/min. and halt at 700^{o}C for 20min. It was again heated from 700^{o}C to 800^{o}C at 20^{o}C/min. and halt for 10min at that temperature i.e., 800^{o}C. And was lastly heated from 800^{o}C to 900^{o}C at 20^{o}C/min and was halt for 10min. High- purity nitrogen was used as a carrier gas during the pyrolysis tests.

The evolved gas was carried by the carrier gas to the sample cell of FTIR spectrometer (Hyphenated Technology Compendium; PerkinElmer, Frontier) and then transferred to the injection port of a TG-FTIR system (PerkinElmer, STA8000). The FTIR sample cell temperature was kept at 900°C, and the resolution was set at 1 or 4 cm^{-1}. The scanning range was 450–4000 cm^{-1}. Time Base software was used to record the spectrum based on TG sample temperature. The gases evolved at selected temperatures

were analyzed. The gas sample loop volume was 1 mL, and the injection time was 10minits. Gas analysis was performed using FTIR. All transfer lines in the evolved gas analysis system (EGA PerkinElmer, TL9000) were maintained at 20 °C interval to avoid condensation of volatile gases with boiling temperatures less than 280°C.

The transfer and source temperatures were 20°C. Mass spectra were recorded in the mass range m/z = 35−500. Mass of the chicken eggshell keep depreciating from 33.4 mg to 17.50mg. Analyte was identified based on the mass spectra shown as can be seen in the result;

H. Thermogravimetric Analysis (TG-FTIR) result

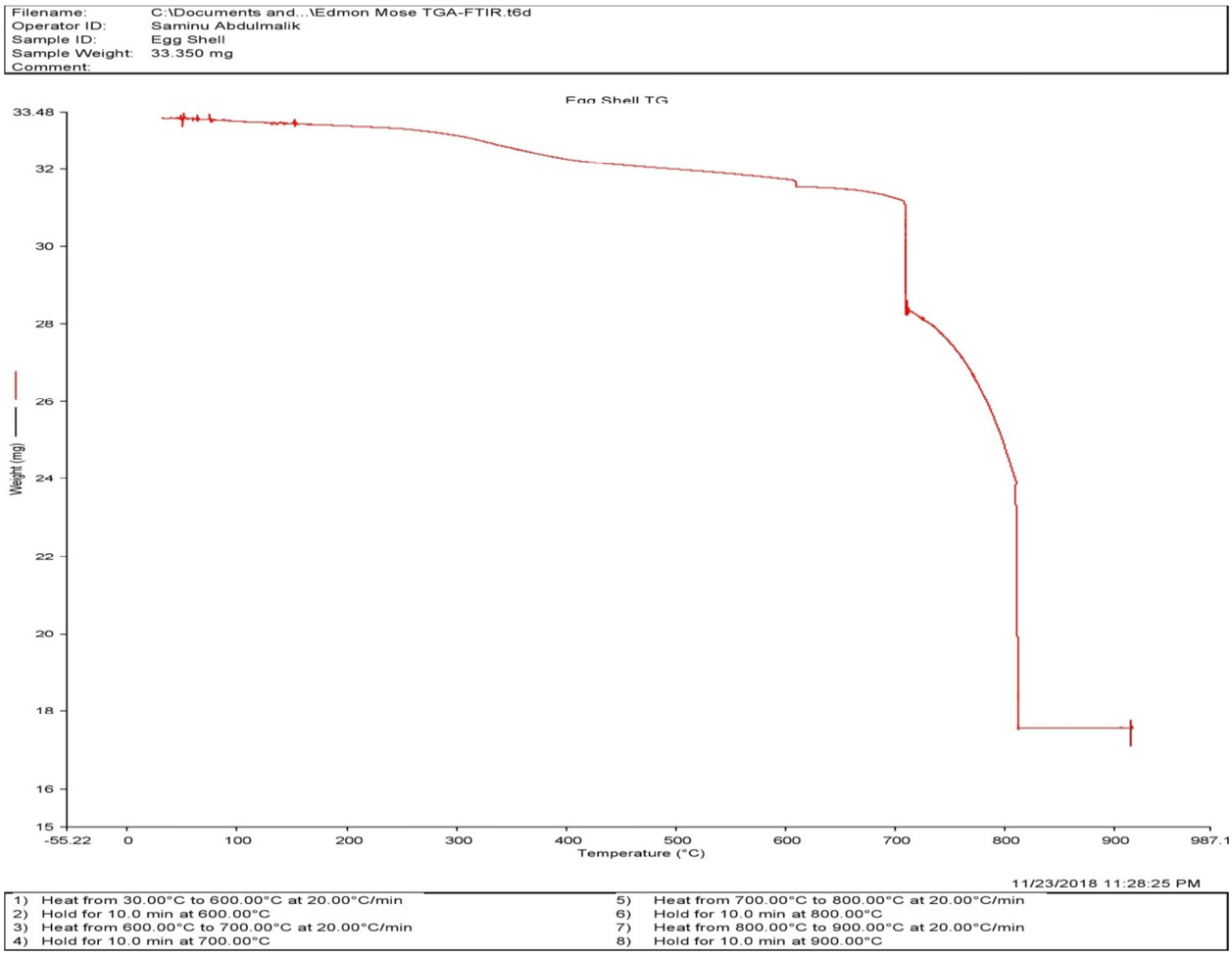

Figure 1. Thermal Analysis of CaO nanoparticles

Figure 1.: shows the thermal transitions during the reaction process were described by TG-FTIR. 33.48mg of the sample was used. The figure indicates that the degradation in nanoparticles occurred in four phases, when the temperature is varied from 0°C to 900°C. Initially, mass loss starts at about 250°C to 32.00 mg due to the hydration of water contents. The second mass loss slightly occurred at 600°C to 31,00mg attributing to the exhaustion of ethanol used in chemical reactions. The third mass loss occurred at 700^{O}C to 26.00 mg. The rapid mass change was observed in the fourth phase at 800°C to 17.00mg, and the last mass loss is observed at 900^{o}C to 16.30 mg. This variation is due to the phase change from CaCO3 and other elements in to CaO phase which is also confirmed by X-ray diffraction. The calcination continued to ensure complete conversion into CaO. The decomposition of the carbonate was accompanied by evolution of CO_2, according to the following endothermic reaction; $CaCO_{3(s)} \longleftrightarrow CaO_{(s)} + CO_{2(g)}$

I. Fourier Transform Infrared Spectroscopy Analysis for the control (uncalcinned chicken eggshell)

The absorption between $870cm^1$ – $675cm^1$ is a strong band bending C – H of a phenyl ring constituent. At absorption of $873cm^1$, it shows a weak band of C – O bond

related to carbonation of Calcium oxide nanoparticle. At absorption band of 553cm[1], it identifies the vibration of the Ca – O bond. Generally, the sharp absorption around 720cm[1] to 924cm[1] indicates that the CaCO as the basic component in an eggshells are much available.

The strong band at 3641.68 cm^{-1} corresponds to the free OH bonds stretching only in a dilute solvent from the remaining hydroxide. The bands at 1412.06 cm^{-1} corresponds to vibrational group for Amides, C-C(=O). This clearly shows C-N and skeletal stretching. Generally, the absence of a sharp absorption around 724 cm[1] and 924 cm[1] indicates that the Calcium Carbonate ($CaCO_3$) as the basic component of the eggshell are no longer present as it is already converted to CaO. This is in line with the research reported by Risfidian *et al.,* (2016).

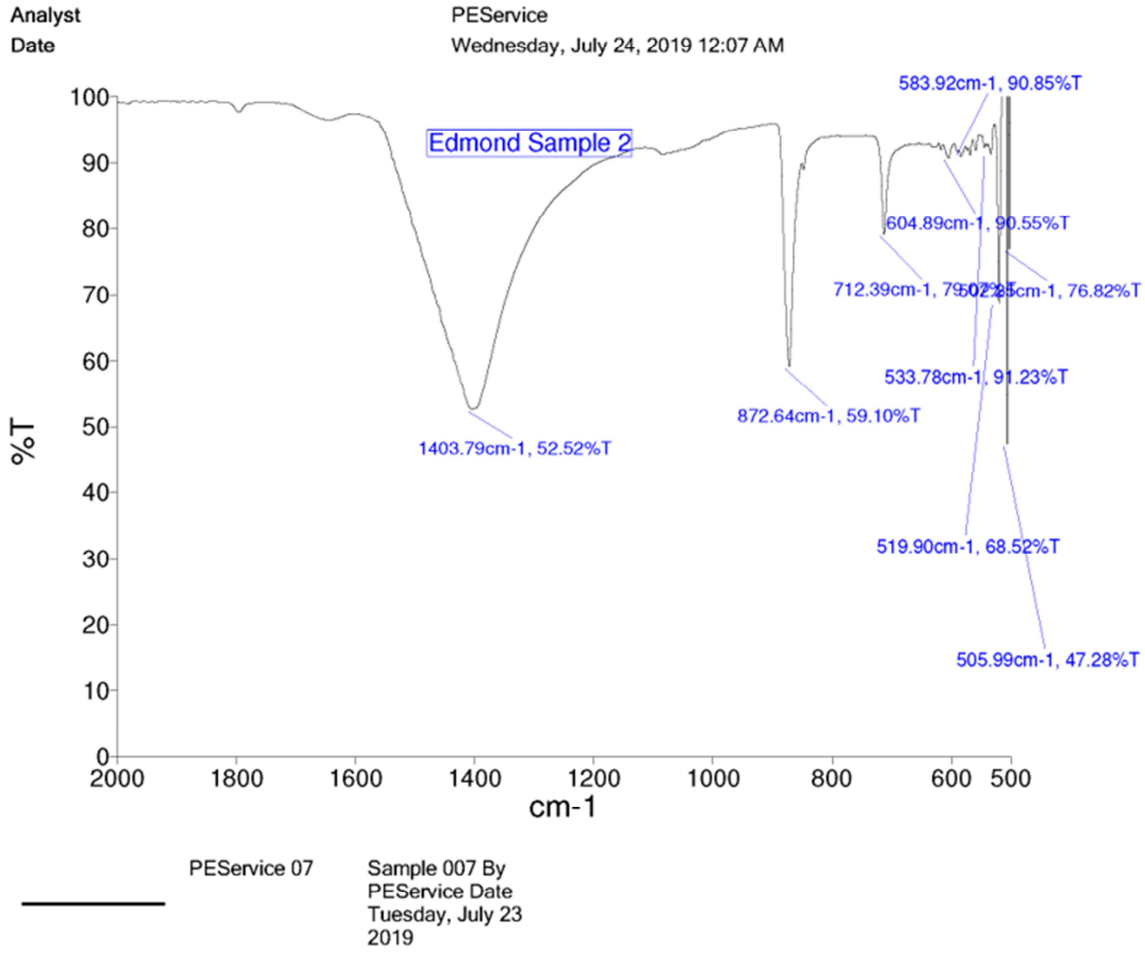

Figure 2. FTIT result for the uncalcinned chicken eggshell (control).

Table 1. Observed infrared band positions and their assignments

Frequency	Vibrational group	Remark
3641.68	Primary aliphatic alcohol	Free OH stretching, only in dilute solution

1412.06	Amides, C-C(=O)-NH_2	C-N and skeletal stretching
873.05	Aliphatic, R-CR_2-NH_2	NH_2 swag

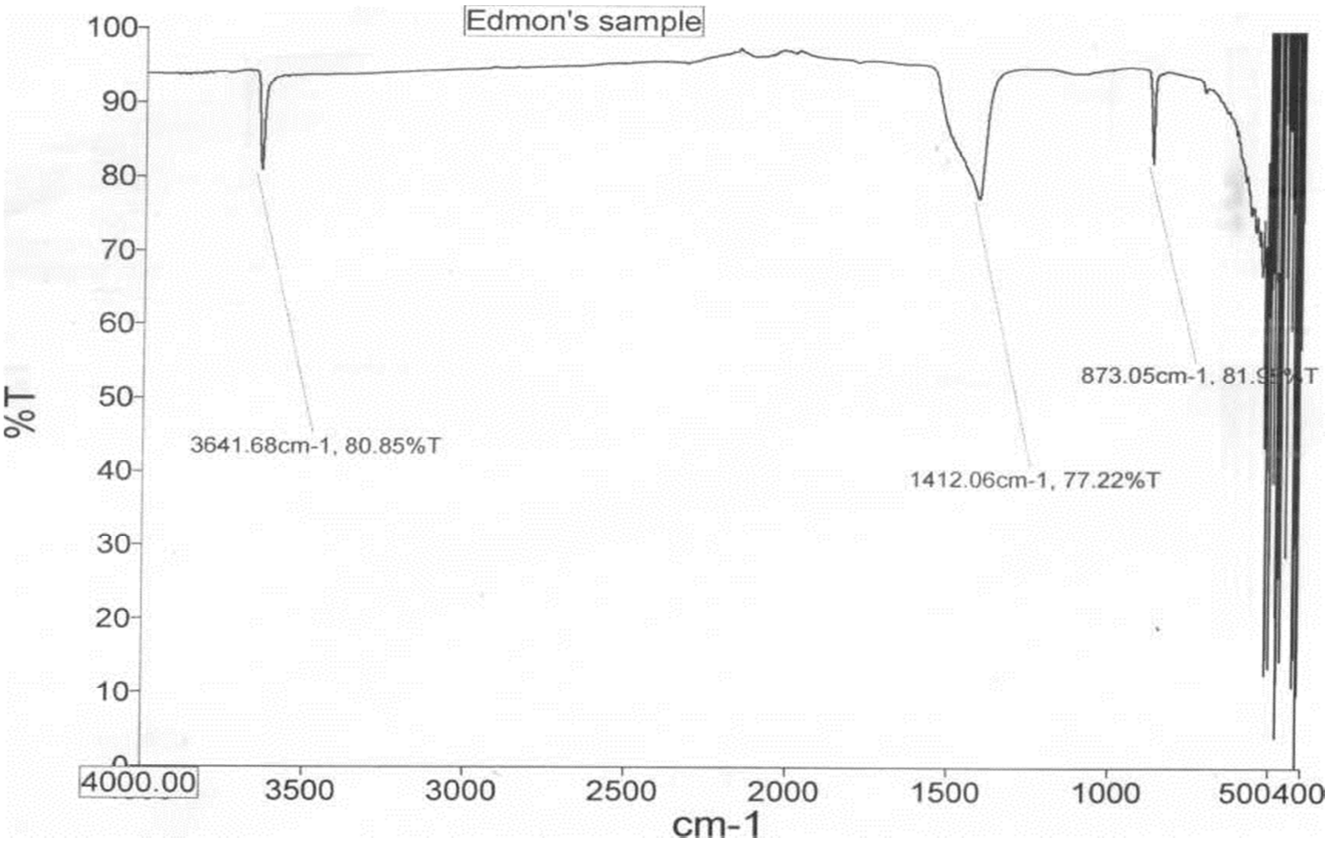

Figure 3 FTIR analysis of calcium oxide nanoparticle produced from chicken eggshell.

III. Conclusion

CaO nanoparticle was synthesized from a chicken eggshell by the means of Chemical O-precipitation of the wet chemical synthesis method and this process basically be classified under the Bottom-Up process of production of nanoparticles. This shows the utilization and recycling of household wastes and the recycling process may also be performed on other household wastes Thermogravimetric analysis shows that the synthesized particle is thermally stable, 50% of the particle show perfect stability even at temperature of 950oC. FTIR analysis of the synthesized particle shows very limited signals on the absorption band and this clearly indicates the total conversion of the main components of eggshell (CaCO3) into CaO nanoparticle.

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