Original Research Article

INVESTIGATION OF THE EFFECTSCALCINATION TEMPERATURE ON THEPHYSIOCHEMICAL PROPERTIES OF HETEROGENOUS CATALYSTS PRODUCED FROM EGGSHELL AND RICE HUSK

ABSRACT

The production, security, sustainability, and affordability of energy are crucial factors in various industries and physical and chemical processes. To address environmental concerns and promote energy production, solid waste materials can serve as catalysts. This research specifically investigates the influence of different calcination temperatures on the properties of two solid wastes, namely Eggshell and Rice husk, as heterogeneous catalysts in Biodiesel production.The raw materials, Eggshell and Rice husk, were locally sourced from Bosso, Minna Niger State. Eggshell was subjected to calcination temperatures of 600 ºC, 650 ºC, and 700 ºC in a muffle furnace, labeled as KA, KB, and KC, respectively. Rice husk, on the other hand, underwent calcination temperatures of 600 ºC, 700 ºC, and 800 ºC in a muffle furnace, labeled as KD, KE, and KF, respectively.The results of the study revealed notable changes in various properties such as color, mass, chemical transformation, crystallinity, and functional groups as the calcination temperatures increased for both raw materials. X-ray diffractometer (XRD) analysis was used to characterize the calcined Eggshells and Rice husks. It was observed that increasing the calcination temperature led to enhanced crystallinity and chemical transformation in the eggshells. Conversely, the Rice husks exhibited decreased crystallinity but increased chemical transformation with higher calcination temperatures. The presence of a small amount of K_2O in the calcined Rice husk was attributed to leaching with HCl after roasting and quenching before calcination. Furthermore, the Fourier Transform Infrared Spectroscopy (FT-IR) analysis indicated the formation of desirable functional peaks with increasing calcination temperatures in both materials. Additionally, the presence of $CO3^{-2}$ was observed in KA and KB due to the incomplete decomposition of $CaCO_3$. Notably, the highest formation of CaO occurred at 700ºC for Eggshell, while the Rice husk calcined at 800ºC exhibited the highest formation of silicon (IV) oxide and lower impurity content (lignin, cellulose, and hemicellulose). Overall, this research provides valuable insights into the effects of calcination temperatures on the properties of Eggshell and Rice husk as catalysts for Biodiesel production. The findings highlight the potential of utilizing solid waste materials as catalysts for energy production while also considering the optimization of calcination temperatures to enhance their performance.

Keywords: *Characterization, physicochemical, chemical transformation, calcination temperature.*

INTRODUCTION

Energy is widely recognized as a fundamental factor in economic growth and essential for various aspects of human life, including sustainable industrial development, transportation, and electricity generation. However, the depletion of fossil fuel reserves, coupled with environmental concerns and high costs associated with fossil fuel combustion, has highlighted the need for alternative energy sources. One promising alternative is biofuels, such as biodiesel (Olutoye et al., 2016). To address potential fuel crises, high costs, and environmental challenges linked to fossil fuels, the search for environmentally friendly energy substitutes is essential. One potential source of such substitutes is biofuels, with biodiesel being a particularly promising option (Olutoye et al., 2016). Among the abundant solid waste materials generated daily, eggshells have emerged as a potential resource with economic value. Instead of incurring costly disposal methods, eggshells can be economically transformed into valuable products. Eggshell waste can serve as a solid base catalyst in biodiesel production, leading to reduced pollutants, lower production costs, and environmentally friendly processes. Eggshells contain bioactive chemicals, primarily calcium carbonate ($CaCO_3$), magnesium carbonate ($MgCO_3$), and calcium phosphate ($Ca_3(PO_4)_2$). They also contain trace amounts of other elements like sodium (Na^+), potassium (K^+), zinc (Zn^{2+}), manganese (Mn^{2+}), iron (Fe^{2+}/Fe^{3+}), and copper (Cu^{2+}). Furthermore, eggshells can be used as absorbents for heavy metals in wastewater treatment and as biomaterials for bone tissue replacement (Faridi and Arabhosseini, 2018). Rice husk, another abundant waste product, has significant potential as a biomass source for various applications. It contains chemical compositions such as silicon oxide (SiO_2), aluminum oxide (Al_2O_3), ferric oxide (Fe_2O_3), calcium oxide (CaO), magnesium oxide (MgO), sodium oxide (Na_2O), potassium oxide (K_2O), manganese oxide (MnO_2), and sulfur trioxide (SO_3). Extracting silica from rice husk and producing value-added silicon-based compounds is one of the potential uses. Currently, a substantial amount of rice husk is burned for energy generation, leading to the production of rice husk ash. However, only a small portion of rice husk is utilized for silica extraction. Transforming rice husk into a heterogeneous catalyst for biofuel production has gained attention in research. Catalysts play a vital role in various reactions, making research on their application essential. In the context of biodiesel production, base catalysts have shown superior performance compared to acid catalysts, particularly in alkaline catalysis. Heterogeneous catalysis, which involves solid surfaces facilitating chemical interactions between molecules, offers advantages over homogeneous catalysis in terms of regeneration, activity, selectivity, and ease of separation from reactants. Metal oxide catalysts like CaO, SiO_2, and MgO are commonly employed in reactions (Mohadi et al., 2016).The use of solid-based catalysts, particularly in renewable energy industries, has gained attention as a means to reduce reliance on fossil fuels and meet increasing energy demands. Heterogeneous catalysts offer advantages such as easy separation, catalyst recycling, reduced energy requirements, and lower water consumption, contributing to a reduction in biodiesel production costs and addressing corrosion and toxicity challenges (Faruque et al., 2020; Mat.R et al., 2012). The need for environmentally friendly and renewable energy sources is driven by rising petroleum prices, population growth, and environmental concerns. Biodiesel, derived from mono-alkyl esters of long-chain fatty acids found in vegetable oils or animal fats, offers a viable solution. It aligns with renewable energy objectives and can be used as a drop-in biofuel in existing diesel engines and infrastructure (Chakraborty et al., 2010; Gerhard, 2010).

2.0 MATERIALS AND METHODS

2.1 MATERIALS/REAGENTS

Tables 1 and 2 show the materials/reagents and equipment used in the experiment respectively.

Table 1. List of materials/reagent used for the experiment and their sources.

Materials/Reagents	Source	Remarks
Rice Husk	Bosso Rice Milling Factory	Brown
Chicken Eggshell	Merchant Shop Bosso	Light brown

Distilled water	SIM Best Chemical Shop.	Colourless, odourless and tasteless
Hydrochloric Acid (HCl)	SIM BEST Scientific and Chemical	Colourless

2.2 EQUIPMENT

Table 2. The list of equipment used for the experiment

Equipment	**Manufacturer**	**Sources**
Digital weighing balance	Ohaus cooperation	Chem.Engr.Lab. FUTMinna
Muffle Furnace	E. Track England	SIM Best Chemical Lab.
Grinder	Pyrex England	SIM Best Chemical
Sieve(500mm)	Pyrex England	SIM Best Chemical Lab,
Mortar and Pestle	Nigeria Made	Bosso Milling Factory
XRD Machine	Rigaku Tokyo, Japan	University of Ibadan Lab.
FT-IR Machine	Nicolet iS10	University of Ibadan Lab.
Drying Oven	Pyrex England	SIM Best Chemical Lab
Ceramiccrucible	Nigeria Made	Step-B lab FUTMINNA
Hot plate.	Nigeria made	SIM Best Lab Chemical Lab
Heating Pot	Nigeria made	SIM Best Lab Chemical Lab

2.3 Experimental Method

This shall involve Pretreatment of Samples, Size Reduction, Rice Husk Ash (RHA) formation, Calcination of the Egg Shell and Rice Husk (RH) and Characterization of the calcined Materials.

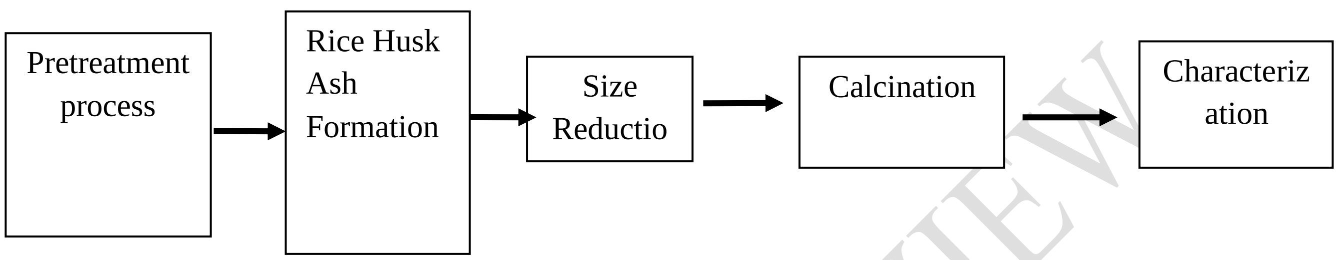

Fig 1: Flowchart of the Synthesis of Eggshell Rice Husk Ash Catalyst

2.2.1 PRETREATMENT AND CALCINATION OF SAMPLES

2.3.1.1 Rice Husk

Rice Husk was obtained from a rice milling factory and underwent a cleaning process using distilled water to eliminate soil particles and impurities. It was then naturally dried in sunlight and stored in an airtight container. Subsequently, 450g of the cleaned rice husk (RH) was roasted in a heating pot on a heat plate. The resulting roasted rice husk was then quenched in room temperature distilled water and separated from the water through filtration.The roasted and quenched rice husk was subjected to leaching. This involved immersing 300g of the roasted and quenched rice husk in 900ml of diluted hydrochloric acid (HCl) for a duration of 3 hours. The mixture was filtered using filter paper and washed three times with distilled water. Finally, the residue was dried at 100 °C for several hours in a controlled air environment and labeled as LRQRH (Leached-Quenched-Roasted Rice Husk).The LRQRH sample was divided into three equal parts, each weighing 80g and labeled as KD, KE, and KF. These divided LRQRH samples were then subjected to calcination in a muffle furnace. The samples were heated to temperatures of 600 °C, 700 °C, and 800 °C, respectively. Once the desired temperatures were reached, the samples were left inside the furnace to complete the calcination process. Afterward, they were cooled down and stored for characterization.

fig2: roasted rice husk

3.3.1.2 Egg Shell

The sample was obtained from a merchant located in Bosso Minna, Niger State, Nigeria. It was washed thoroughly to eliminate any impurities and subsequently dried using sunlight. The dried sample was ground into a powder using an electric grinder and sieved to remove some of the white membrane present. The ground samples were then divided into three equal portions, each weighing 250g. These portions were labeled as KA, KB, and KC, respectively. They were loaded into ceramic crucibles and subjected to calcination in a furnace at temperatures of 600 °C, 650 °C, and 700 °C, respectively. Each crucible was left inside the furnace for a duration of 3 hours after reaching the desired temperature. Following the calcination process, the samples were cooled down, and subsequently stored for further characterization

fig3washed and dried egg shell

fig4: muffle furnace **fig5: calcined egg shells**

2.4 Characterization of the calcined Egg Shell and Rice Husk

The elemental and physical properties of the prepared samples were assessed through analysis methods including Fourier Transform-Infrared Ray (FT-IR) and X-ray Diffraction (XRD) analysis.

2.4.1 X-ray diffraction Analysis for Mineral Identification

The powdered samples were formed into pellets and sieved to a size of 0.074mm. These pellets were then placed onto an aluminum alloy grid measuring 35mm x 50mm, which was positioned on a flat glass plate. A paper cover was placed over the samples. While wearing protective hand gloves, gentle pressure was applied by hand to compact the samples.Each sample was subjected to analysis using the Rigaku D/Max-lllC X-ray diffractometer, developed by Rigaku Int. Corp. in Tokyo, Japan. The instrument was configd to generate diffractions at a scanning rate of 2 0/min within the 2 to 500 range, at room temperature, using CuKa radiation set at 40kV and 20mA. The obtained diffraction data, including d values and relative intensity, were compared to the standard data of minerals from the mineral powder diffraction file, ICDD. This database encompasses standard data for over 3000 minerals. Similar diffraction data indicates the presence of the same minerals as those found in the calcined samples when compared to the standard minerals.

fig 6: Rigaku D/Max-lllC X-ray diffractometer

2.4.2 Fourier Transform -Infrared Ray Analysis for Mineral Identification

It was necessary to sign on the notebook, followed by turning on the computer and signing in. The next step involves turning on the power of the instrument and waiting for the initialization process to be successful. If the small panel of the instrument labeled "Perkin Elmer Spectrum 100 Series" shows a successful initialization, the procedure can continue. Otherwise, it is advised to seek assistance from Jianhua.To ensure cleanliness, the sample holder was cleaned using acetone and Kim wipes, taking care not to splash the acetone onto the instrument. Following this, the "spectrum" software was launched from the desktop, using the User Name "Analyst" and Password "analyst". The "Spectrum 100" option should be selected. Upon accessing the "Instrument setup" button, a dialog box titled "Scan and Instrument Setup" will appear. It was necessary to input the sample name, scan range (limited to 650-4500 cm-1), and the desired scan number. To collect background information of the sample holder, the "background" button should be clicked. The next step involved placing the sample on the sample holder. If the sample is liquid, pressing "Apply" and then "Start" will initiate the spectrum collection. In the case of a solid sample, it was advised to first click the "monitor" button on the "Scan and Instrument Setup" dialog. Subsequently, the pressure arm was lowered, and the total pressure applied to the sample was monitored through the "monitor" dialog. Setting the "Force Gauge" to approximately 80, the process can be completed by clicking "Finish". Finally, pressing "Apply" and "Start" will initiate the spectrum collection. For data processing, selecting "View" from the menu and then choosing "Label Peaks" will enable peak labeling. To label a specific peak, selecting "View" > "Cursor" > "Vertical Continuous" activated the cursor, which can be moved to the desired peak. By selecting "View" > "Label Cursor", the peak can be labeled. Saving data in ASCII format can be done by selecting "File" > "Save as" and choosing "ASCII (*.ASC)" as the file type. After completing the necessary tasks, it was important to turn off the software, log off the computer, and switch off the instrument. Additionally, the sample holder was thoroughly cleaned before leaving the FTIR area.

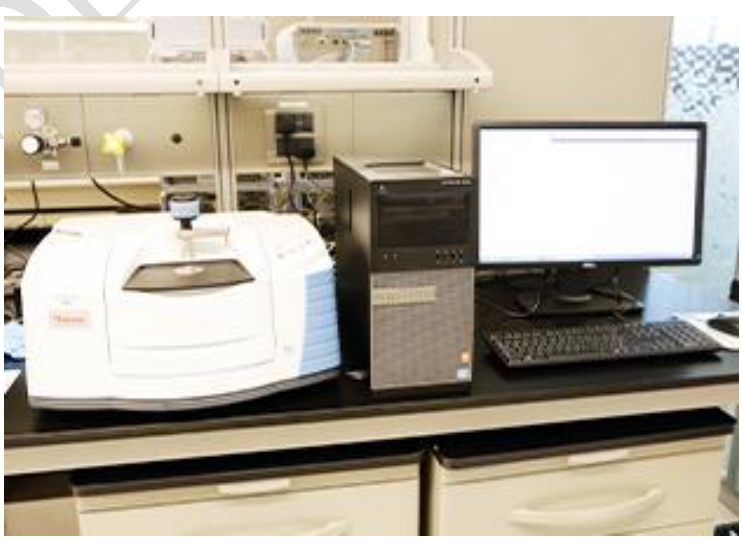

fig 7: FTIR Scientific Equipment

3.0 RESULT AND DISCUSSION

3.1 Change in physical properties of the materials used (colour change)

Table 3. Change in physical properties of the materials used (colour change)

Material Used		Color Change after calcination
Calcined waste eggshell (whitish-brown)		
1.	600 ºC	black
2.	650 ºC	black-gray
3.	700 ºC	gray
Calcined Rice husk (brown)		
1.	600 ºC	light-ash
2.	700 ºC	black-ash
3.	800 ºC	Ash

The eggshells underwent decomposition through calcination in a furnace at various temperatures (600 ºC, 650 ºC, and 700 ºC), resulting in a color change. Table 3 illustrates the observed color changes at different calcination temperatures. At 600 ºC, the eggshells transformed into black powder, while a small amount of black-colored ash was produced at 650 ºC. Finally, at 700 ºC, a significant amount of black powder with a higher ash content was obtained. The results indicate that as the calcination temperature increases, there is a corresponding increase in the formation of metal oxide, as evidenced by the color change in the chicken eggshell powder (Mohadi et al., 2016). Also the calcined rice husks underwent decomposition during the calcination process in a furnace at varying temperatures (600 ºC, 700 ºC, and 800 ºC). The samples exhibited an increasing change in color as the calcination temperature rose, as depicted in table 3. At 600 ºC, the rice husks resulted in a light ash powder, while a black-ash colored powder was obtained at 700 ºC. Finally, at 800 ºC, an ash-colored powder was obtained. The results indicate that as the calcination temperature increases, there is an increased formation of metal oxide, as evidenced by the color change in the rice husk powder (Sekifuji et al., 2021).

3.2 Loss in mass of the raw materials used

Table 4. Loss in mass of the raw materials used

S/N	Material Used	Calcination temp ^{O}C		Mass loss after calcination			
				Initial mass(g)	Final mass(g)	Mass loss(g)	% loss
1.	**Eggshell**	1)	600 ºC	250	188.25	61.75	24.7%
		2)	650 ºC	250	171.75	78.25	31.3%
		3)	700 ºC	250	160.75	89.25	35.7%
2.	**Rice husk**						
		1)	600 ºC	80	19.84	60.16	75.2%
		2)	700 ºC	80	12.32	67.68	84.6%
		3)	800 ºC	80	8.56	71.44	89.3%

Table 4. shows that there was loss in masses of calcined eggshells rice husks during each calcination due to thermal decomposition of $CaCO_3$ to CaO and $Ca(OH)_2$, which is as a result of loss in CO_2 and other impurities, and loss of some volatile materials in Rice husk increasesas calcination temperature increases and decomposition of Ca_2SiO_4 to SiO_4When calcined Rice husk at different calcination temperatures from 600 ºC to 700 ºC to 800 ºC.As showed in table 4. 24.7% was loss when Eggshell was calcined at 600 ºC, at 650 ºC there was 31.3% loss in mass of Eggshell and highest loss in mass of eggshell at 700 ºC of about 35.7%.RH loss it's mass up to 75.2% by weight at 600 ºC, and loss it's mass up to 84.6% at 700 ºC while at 800 ºC there was mass loss up to 89.3% as shown in Table 4. due to presence of physically absorbed moisture and water content in the RH, the removal of organic group by oxidation reaction and the decomposition of hemicellulose and cellulose followed by total decomposition of lignin.

3.3 Characterization of the Calcined Eggshells and Rice husks

The samples produced were characterized to know the chemical transformation, crystallinity and functional groups presence X-ray diffractometer and FT-IR Spectrometer respectively.

3.3.1 X-ray diffraction

Samples were analyzed using Cu- K source equipped with an Inel CPS 120 hemispherical detector (RigakuD/Max-IIIC Co., Japan). at scanning rate of 2 ^{0}/min in the 10^0 to 55^0

3.3.1.1 calcined eggshell

X-ray diffraction is a non-destructive analytical technique which reveals information about crystallographic structure. This technique is widely used in the characterization of shell. The XRD patterns for calcined eggshells.

3.3.1.1.1 KA (eggshell calcined at 600 ºC)

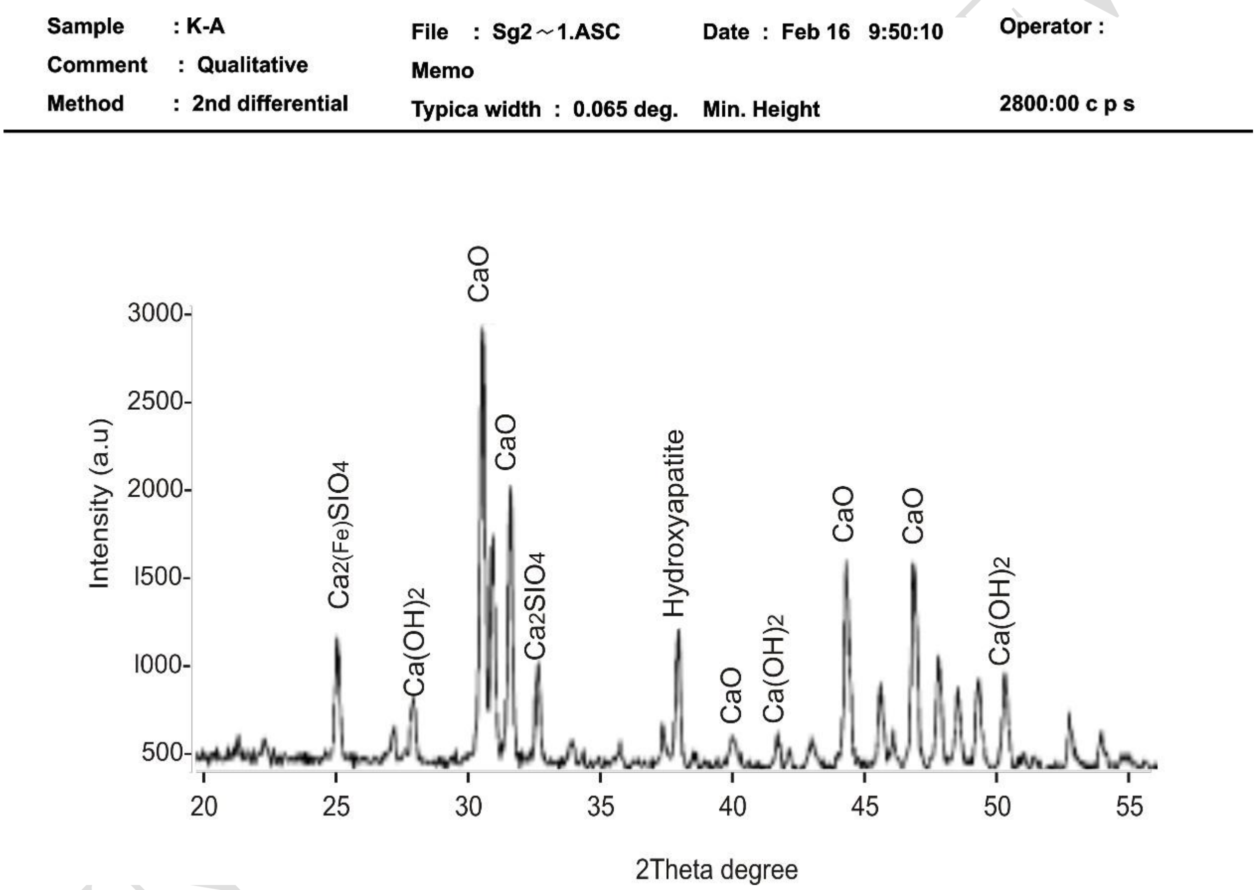

fig 8. Eggshell Calcined at 600 ºC (KA)

Fig8 showed the highest peak is at 2ϴ= 30º shows presence of CaO as crystalline form,there are more CaO at 2ϴ = 31º, 40º, 44º and 47º at difference phases. There is presence of $Ca(OH)_2$ at 2ϴ= 28º, 42º and 50º as a result of moisture content of the calcined sample. $Ca_2(Fe)SiO_4$ exists at 2ϴ= 25º shows incomplete decomposition of the eggshell while hydroxyapatite occurs at 2ϴ= 37.5º (Pornchai *et al*., 2016).

3.3.1.1.2 KB (eggshell calcined at 650ºC)

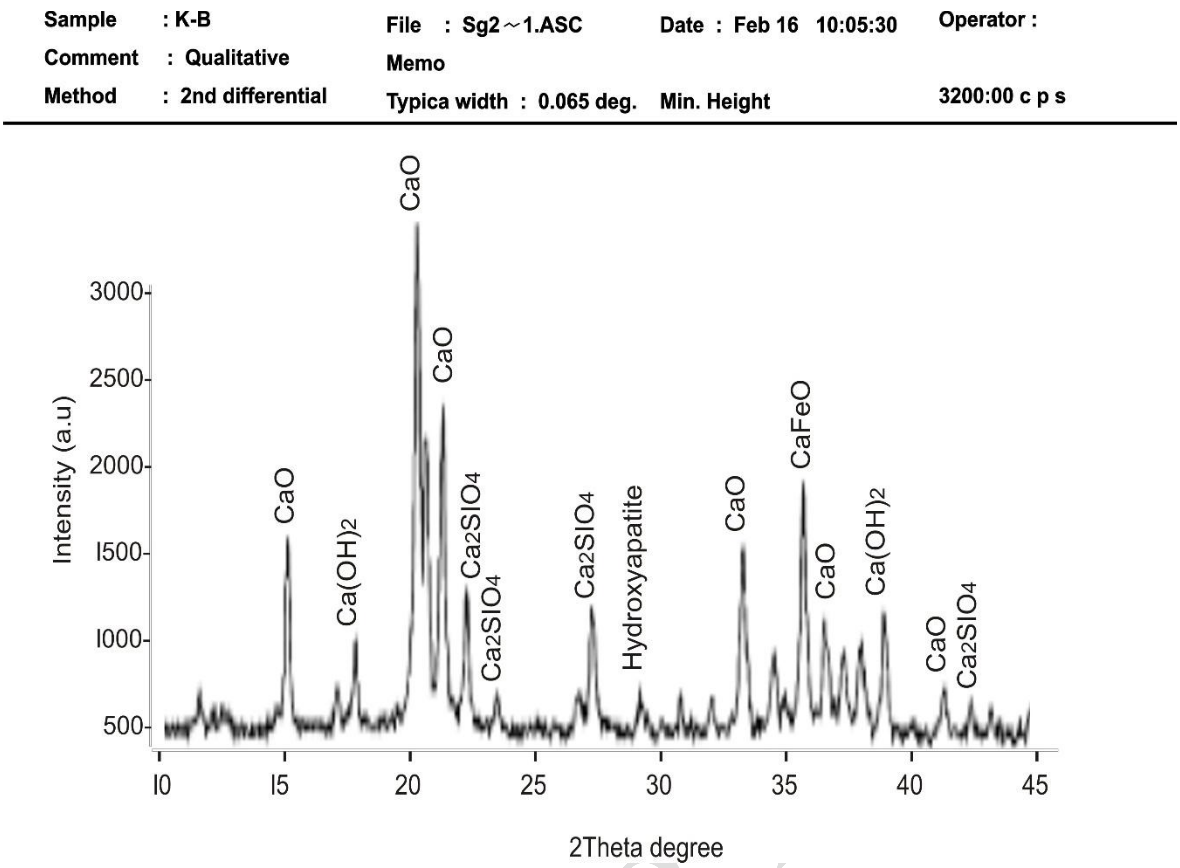

Fig9 Eggshell Calcined at 650 ºC (KB)

Fig9 showed the highest peak is at 2ϴ= 22º shows presence of CaO being most abundance in a crystalline form, there are more formation of as compared to KA, other formation of CaO at 2ϴ= 15º, 22º, 34º, 37º and 42º at difference phases.

There is presence of $Ca(OH)_2$ at 2ϴ= 17º, 44º and 39º where the amount is small as in KA. $Ca_2(Fe)SiO_4$ has decomposed to Ca_2SiO_4 at 2ϴ= 22º, 24º, 27º and 42º.

there is small amount of hydroxylapatite at 2ϴ=30º (Pornchai *et al*., 2016).

3.3.1.1.2 KC (Eggshell Calcined at 700ºC)

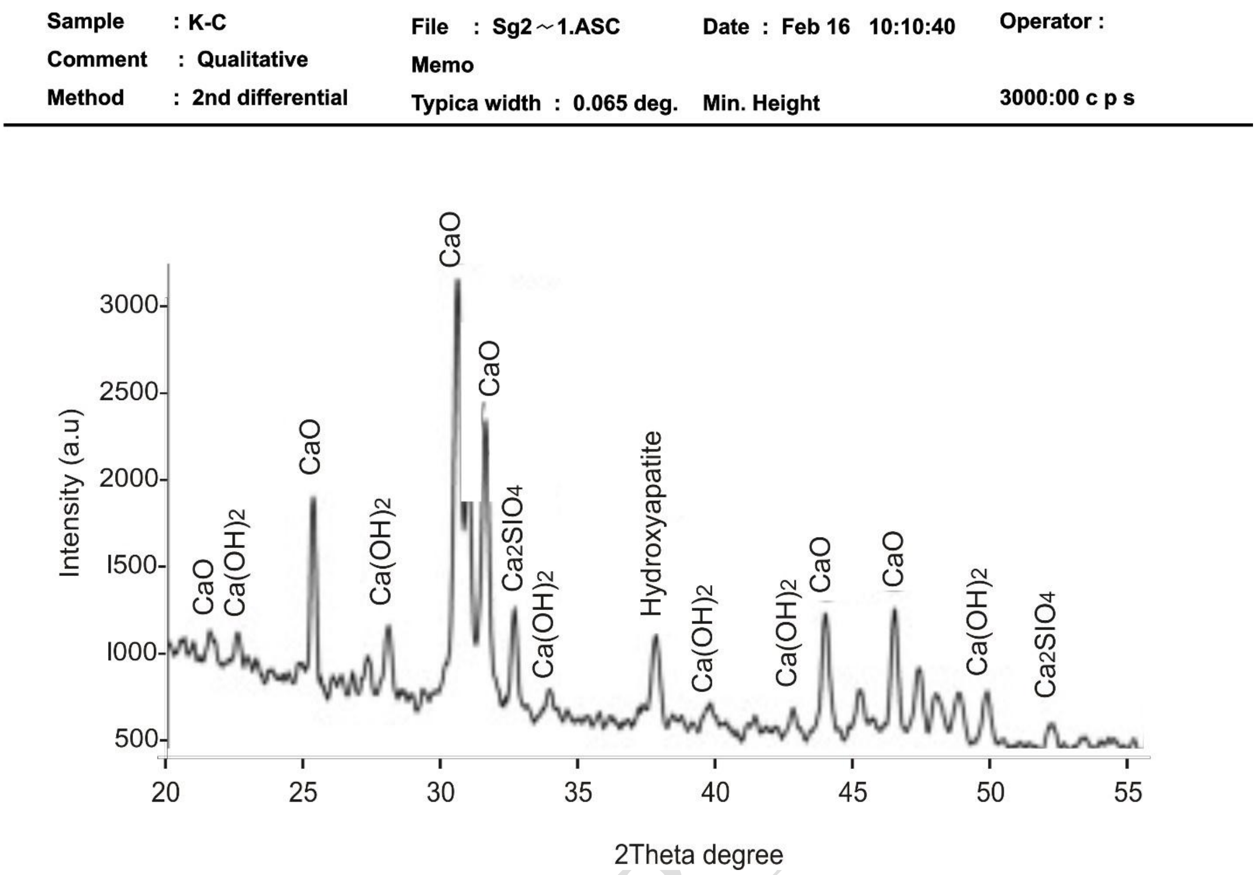

Fig10 Eggshell Calcined at 700 ºC (KC)

Fig10 showed the highest peak is at 2ϴ= 32º with strong crystalline nature as sharp the band shows presence of CaO being most abundance, there are more formation of CaO as compared to KA and KC, other formation of CaO at 2ϴ= 26º, 32º in small quantity and at 2ϴ= 44º, 47º and 42º at amorphous-like indicated that the sample is getting from crystalline to amorphous. There is presence of $Ca(OH)_2$ at 2ϴ= 28º, 34º, 40º and 50º in small amount as a result of decreased in moisture content as temperature increased where.. $Ca_2(Fe)SiO_4$ has decomposed to Ca_2SiO_4 at 2ϴ= 33º and 52º there is smaller amount of hydroxylapatite at 2ϴ=38º (Mohadi *et al.*, 2016).

3.3.1.2 calcined rice husk

X-ray diffraction is a method of analysis that provides insights into the crystal structure without causing damage. It is extensively employed in the examination of shells to understand their crystallographic arrangement. The XRD patterns for processed rice husks can be obtained using this technique.

3.3.1.2.1 KD (rice husk calcined at 600 ºC)

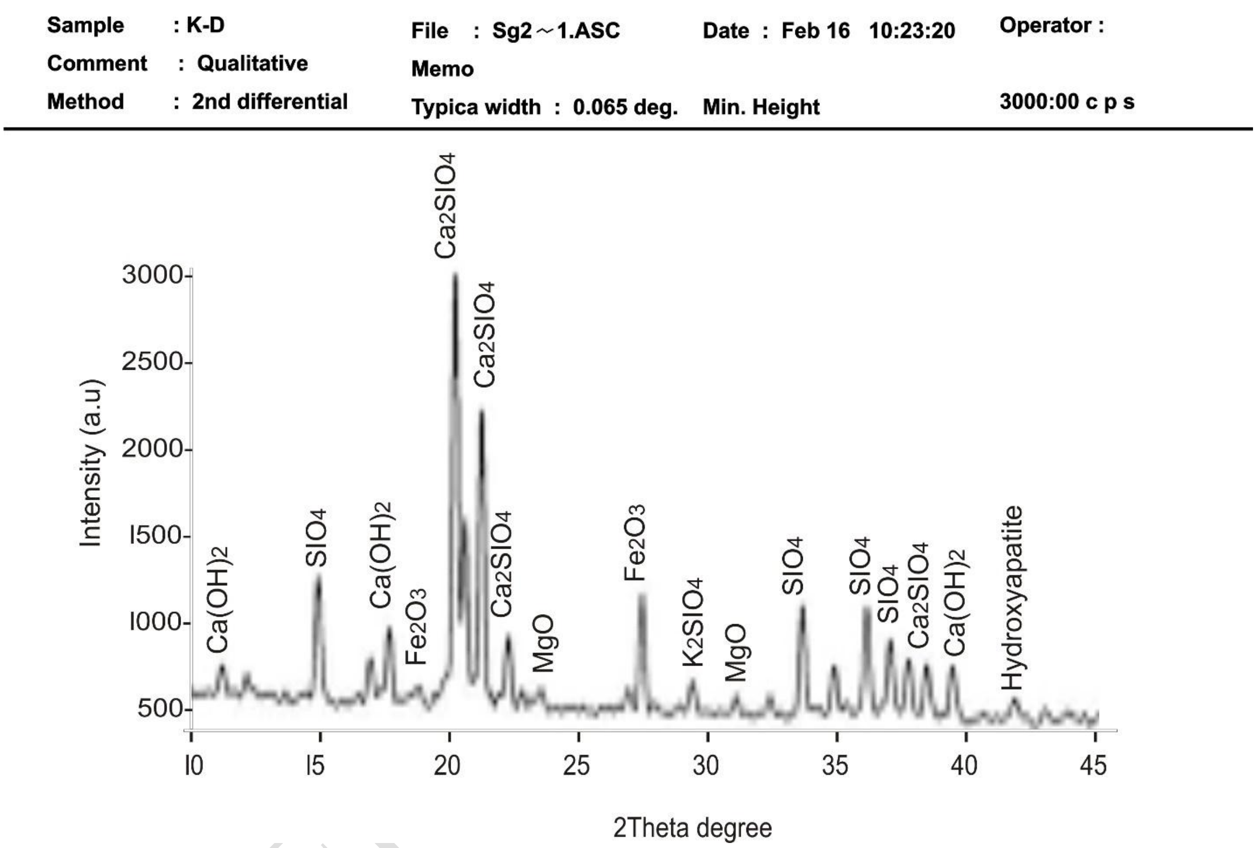

Fig11. Rice Husk Calcined at 600 ºC (KD)

The highest peak is at 2Ө= 21º being the abundance component shows presence of Ca_2SiO_4 it exists at 2Ө= 22º, 28º and 37º and SiO_4 is at 2Ө= 15º, 34º, 36º, 37º in small amount.

$Ca(OH)_2$ occurred in small amount and there is a trace of hydroxylapatite at 42º also trace of MgO and Fe_2O_3.

4.3.1.2.2 KE (rice husk calcined at 700 ºC)

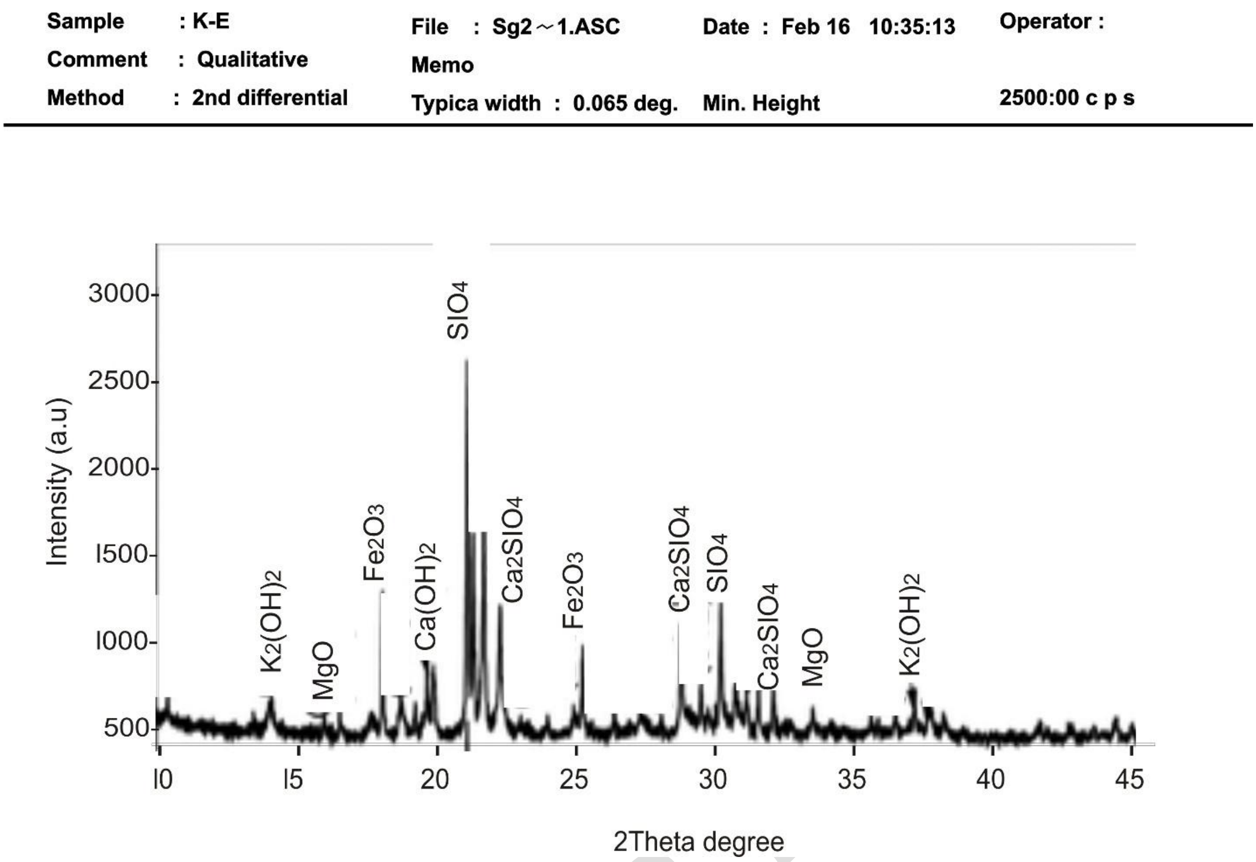

Fig12. Rice Husk Calcined at 700 ºC (KE)

The highest peak is at 2ϴ= 22º and 31º being the abundance component shows presence of SiO_4 in crystalline state. At 2ϴ= 22º, 38º and 37º there is Ca_2SiO_4 formation there is absence of hydroxylapatite , also trace of MgO and Fe_2O_3.

4.3.1.2.3 KF (rice husk calcined at 800ºC)

Sample : K-F	**File : Sg2～1.ASC**	**Date : Feb 16 10:42:50**	**Operator :**
Comment : Qualitative	**Memo**		
Method : 2nd differential	**Typica width : 0.065 deg.**	**Min. Height**	**2700:00 c p s**

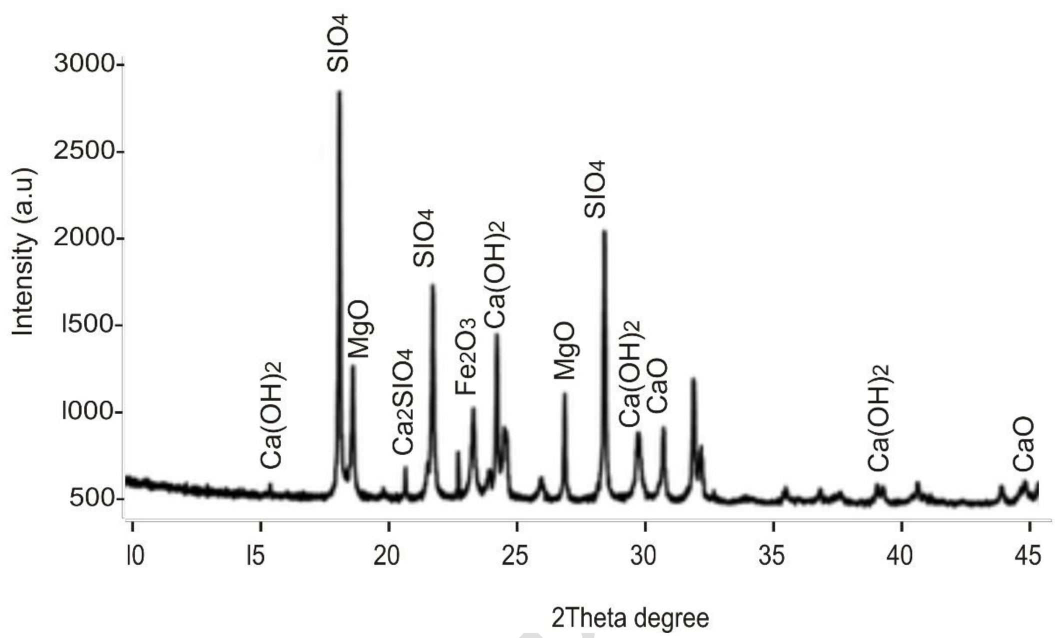

Fig 13. Rice Husk Calcined at 800 ºC (KF)

The highest peak is at 2ϴ= 18º, 22º and 28º showed SiO_4 present in highest composition, Ca_2SiO_4 exists at 2ϴ= 21º in small amount due to more decomposition of the sample at this temperature.$Ca(OH)_2$ occurred in small amount and there is a trace of hydroxylapatite at 24º also trace of MgO, CaO and Fe_2O_3 were observed.

Table 5. Crystallite size (nm) using Scherrer equation

Sample	Average Crystallite size (nm)
KA	177.3967207
KB	274.7932751
KC	278.0374549
KD	274.7932751
KE	238.7086417
KF	16.72281279

An examination of crystallite size changes with increasing temperature using the Scherrer equation reveals that the size of the crystallites in calcined Egg Shell increases between 600 ºC and 700 ºC. Among the samples, KC exhibits the largest crystallite size. On the other hand, the crystallite size of calcined Rice Husk decreases as the calcination temperature rises. Among them, KD, calcined at 600 ºC, has the highest crystallite size, while KF exhibits the smallest crystallite size among all the samples.

3.3.2 FT-IR RESULTS

The samples were characterized to know functional groups present using Perkin Elmer Spectrum 100 Series, Nicolet iS10 FT-IR Spectrometer**.**

3.3.2.1 Eggshell

Eggshell was characterized by FTIR. This technique could be used to identify the major functional groups consisting Calcined eggshell. From the wave number of the molecular vibration modes, a good explanation of the chemical structure could be obtained

3.3.2.1.1 KA (eggshell calcined at 600 ºC)

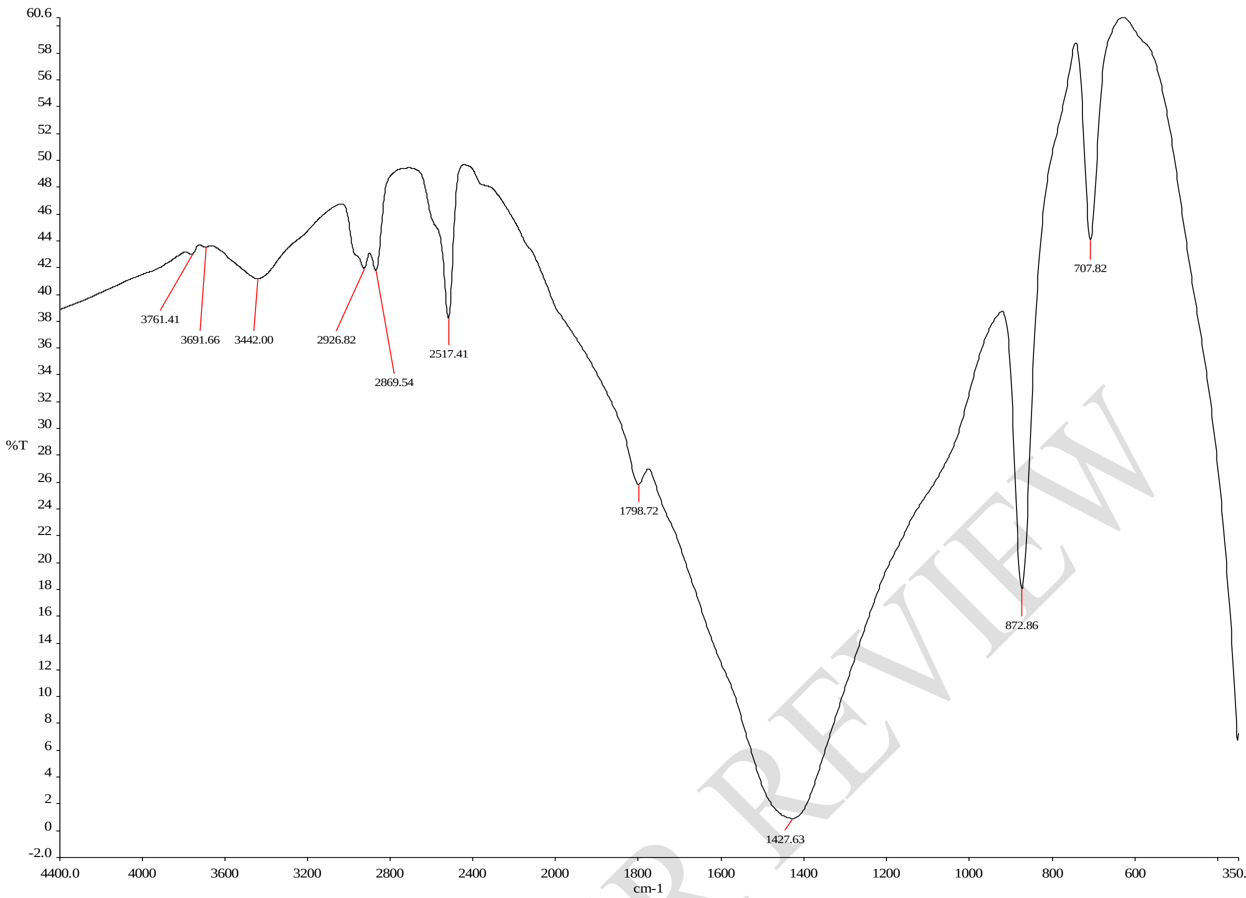

Fig14. Eggshell Calcined at 600 ºC (KA)

The highest band is at 707.82 cm^{-1} indicates presence of symmetric stretching vibrations of CO_3^{-2} bond due to presence of $CaCO_3$, -OH group presents at 3761.41cm^{-1}, 3691.66 cm^{-1} and 3442.00 cm^{-1} due to presence of hydroxyapatite, Ca–OH, –OH–Si, and Ca–OH–Si units in the octahedral layer.There was a broad band at 1460 cm^{-1} due to bending vibrations of the Si-O bond and sharp band at 872.86 cm^{-1} due to presence of CaO bond.

3.3.2.1.2 KB (eggshell calcined at 650 ºC)

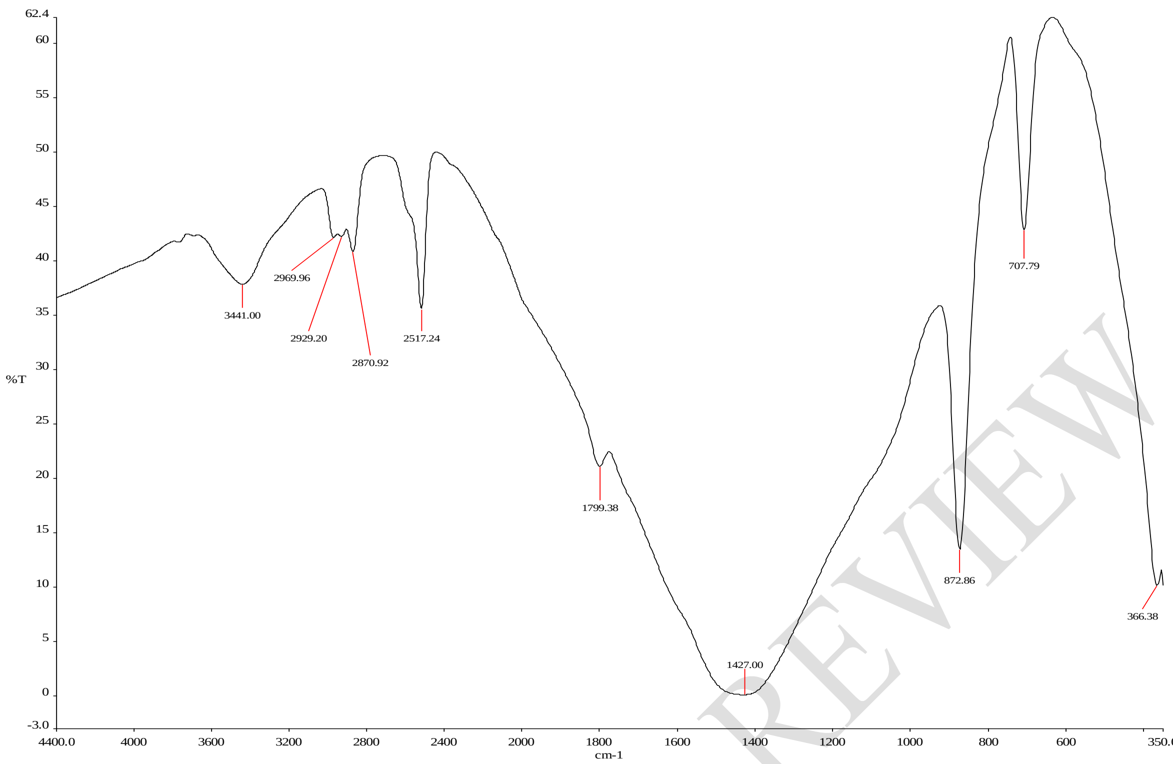

Fig15. Eggshell Calcined at 650 ºC (KB)

The highest band is at 707.79 cm^{-1} indicates presence of symmetric stretching vibrations of CO_3^{-2} bond due to presence of $CaCO_3$, -OH group presents at 3441.00 cm^{-1} due to presence of $Ca(OH)_2$ and small hydroxylapatite as compared to KA.There was a broad band at 1427 cm^{-1} due to bending vibrations of the Si-O-Si group and sharp band at 872.86 cm^{-1} due to presence of Ca-O bond.

3.3.2.1.3 KC (eggshell calcined at 700 ºC)

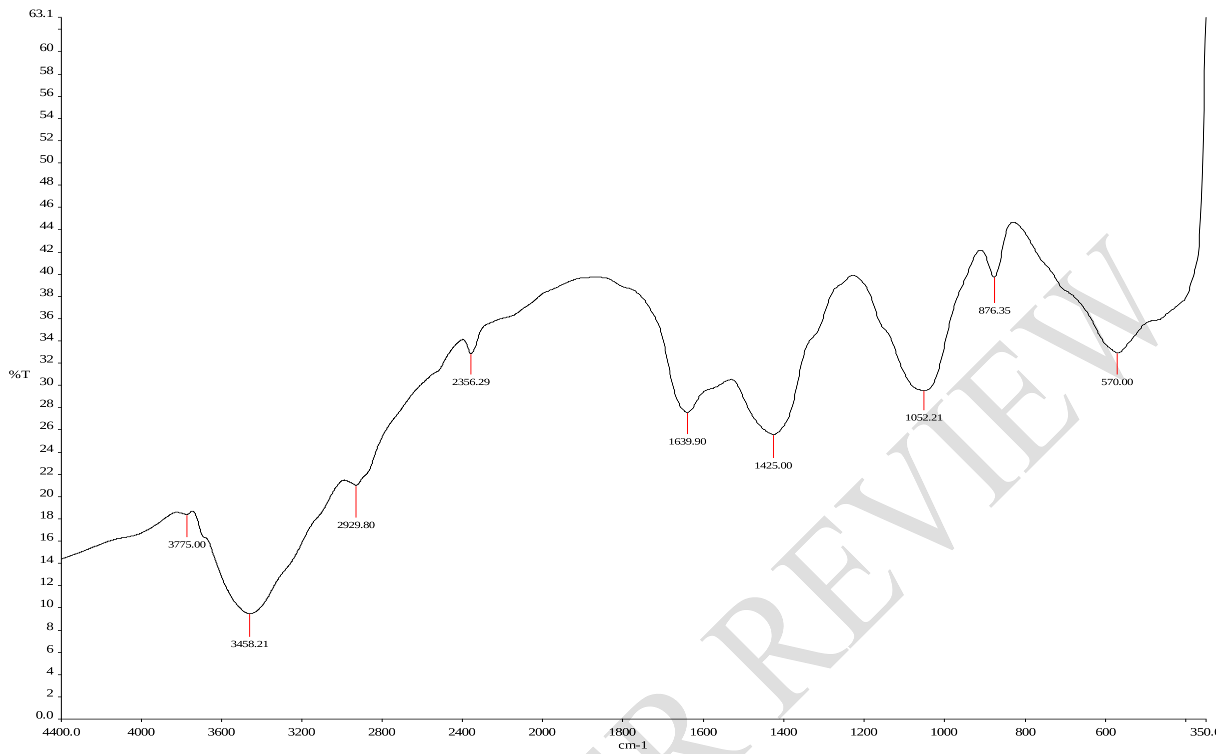

Fig16. Eggshell Calcined at 700 ºC (KC)

The characteristic IR band of eggshell appeared at 3775.00 cm^{-1} and 3458.21 cm^{-1} represent the fundamental stretching vibrations of different –OH groups present in Ca–OH, –OH–Si, and Ca–OH–Si units in the octahedral layer. The strong peak appears at 876.35 cm^{-1} is related to the stretching vibrations of Ca–O, while the bands at 525 and 466 cm^{-1} are due to Ca–O–Si and Ca–O–Ca bending vibrations, respectively. This shows highest formation of CaO (Pornchai *et al*., 2016).

3.3.2.2 Rice husk

KD, KE and KF consist of mixture of CaO and silicate mineral. SiO_2 are present as dominant mineral in all patterns. Fig.17 shows that the percent of SiO2 increase significantly after purification process while the percent of CaO in purified eggshell is increased. K-E and K-F shows similar pattern for the calcined eggshell with SiO2 as the main mineral.

3.3.2.1.4 KD (rice husk calcined at 600 ºC)

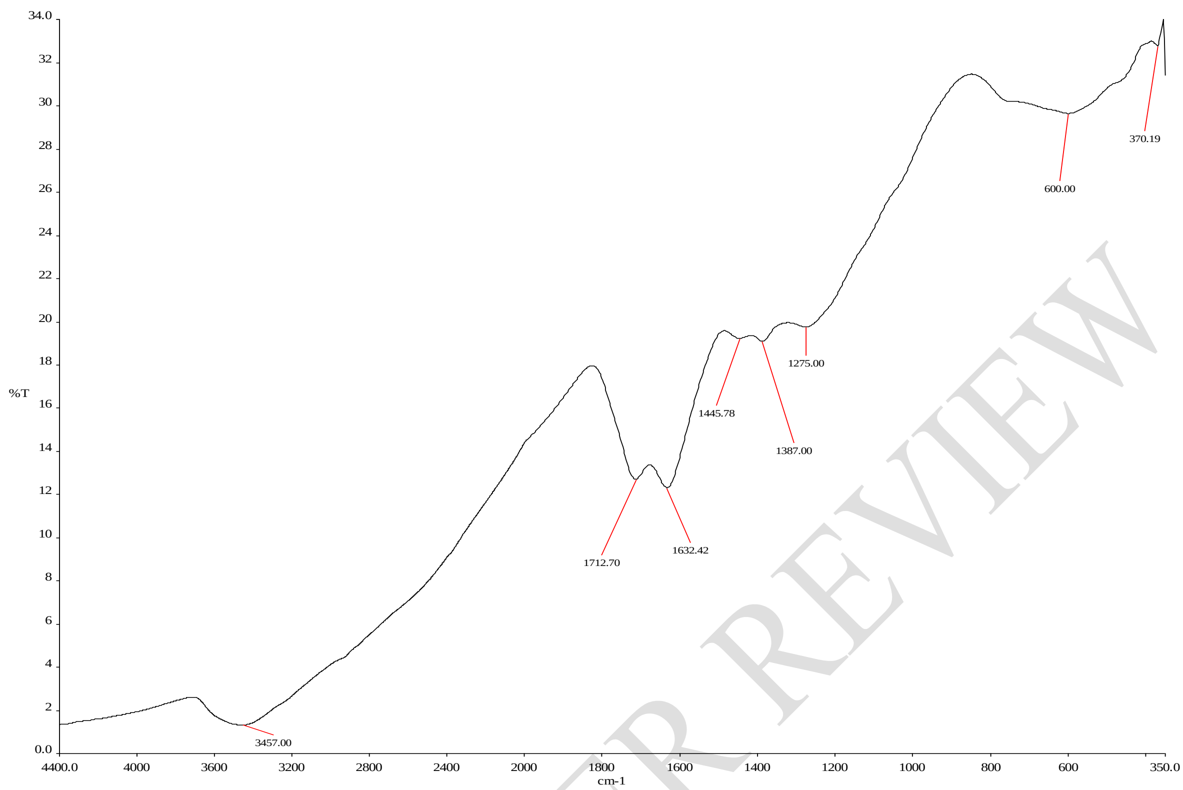

Fig17. Rice Husk Calcined at 600 ºC (KD)

There is -OH group at 3457.00 cm^{-1} indicated presence of $Ca(OH)_2$ in small amount. The highest peak at 370.19 $cm^{-1}$and 600 cm^{-1} are due to presence of bending vibration of Si-O-Si bond and band 1387.00 cm^{-1} is as a result of bending vibration of the CH_3 group in cellulose as a residue and 1712.70 cm^{-1} can be as a result of C=O bond in carbonyl group Hemicellulose.

3.3.2.1.5 KE (rice husk calcined at 700 ºC)

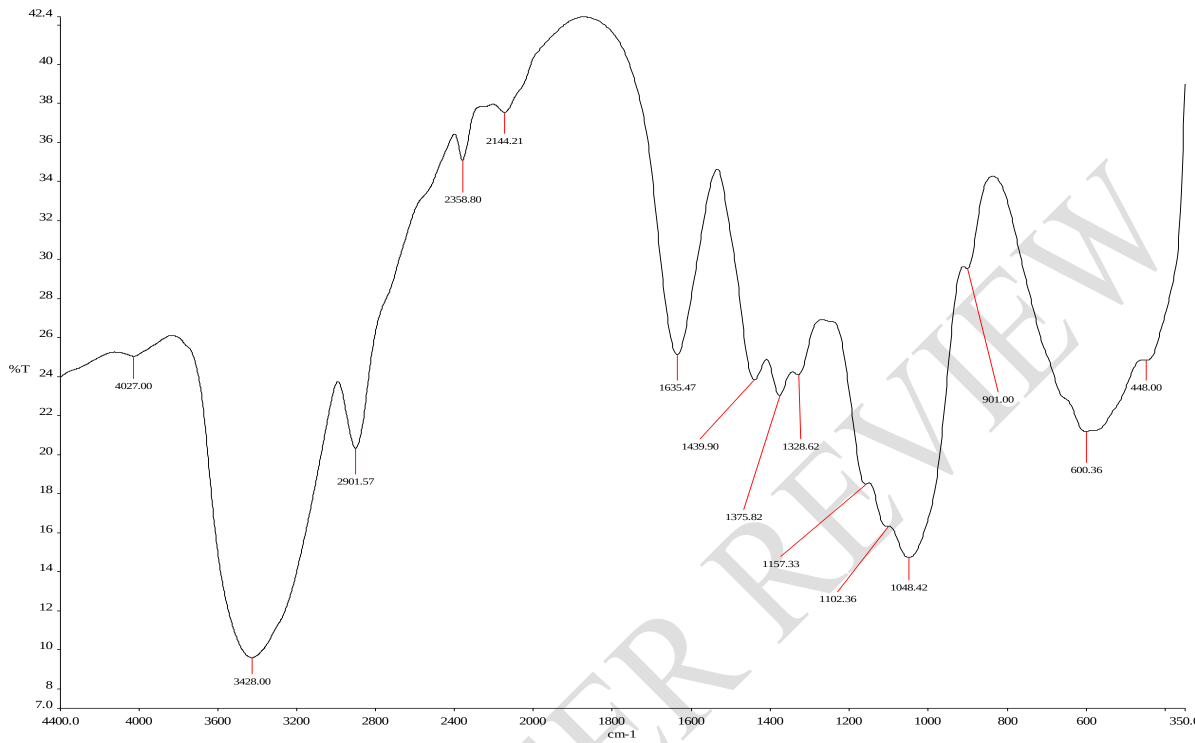

Fig18. Rice Husk Calcined at 700 ºC (KE)

The spectrum is a complex one with more than 5 bands, the 4027.00 cm^{-1} and 3428.00 cm^{-1} are as a result of -OH group in Ca–OH and Ca–OH–Si. bands at 600.36 cm^{-1} and 448.00 cm^{-1} are due to bending vibration of Si-O-Si bondC-H bond in lignin could be at the 901.00 cm^{-1} as shown in small amount, at 1048.42 and 1102.36 could be accounted for presence of Si-O-Si bondin another phase.

3.3.2.1.6 KF (Eggshell Calcined at 800 ºC)

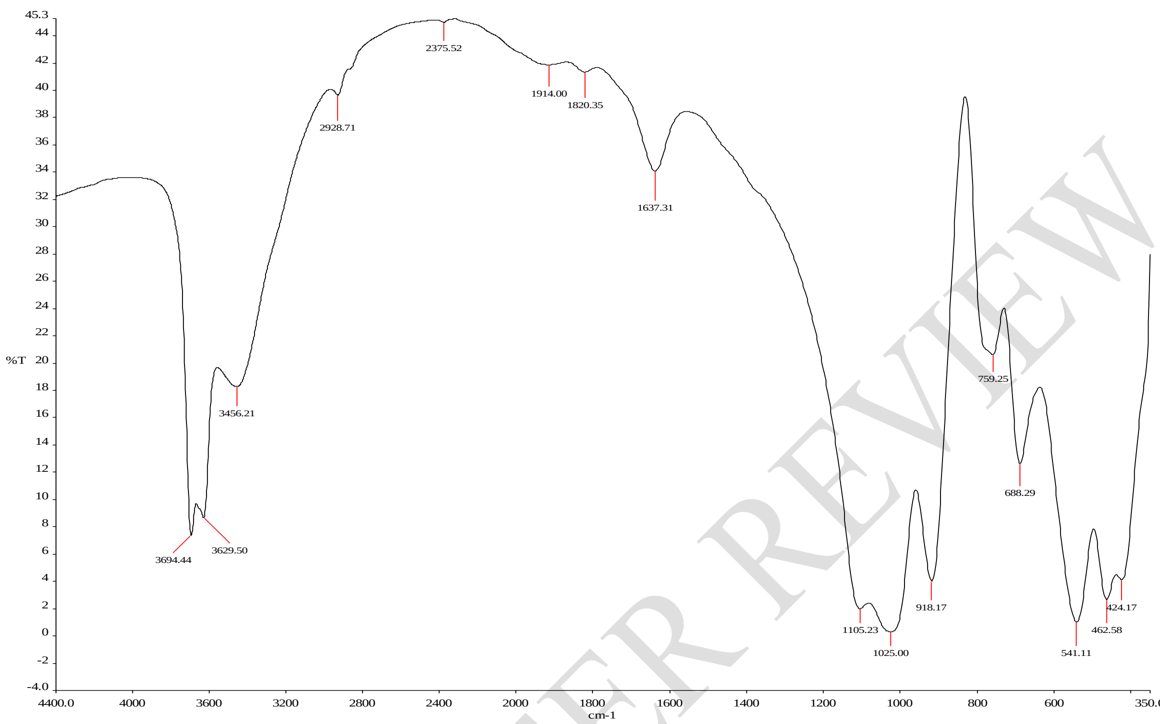

Fig19. Rice Husk Calcined at 800 ºC (KF)

The -OH group vibration is shown at 3694.44 cm^{-1} and 3456.21 cm^{-1} and the band at 424.17 cm^{-1}, 462.58 cm^{-1} and 541.11 cm^{-1} are all due to stretching vibration of Si-O-Si bond.Lignin compound having C-H bending vibration formed at 688.29 cm^{-1}

4.0 CONCLUSION

The study successfully investigated the effects of calcination on the physiochemical properties of solid wastes, namely Eggshell and Rice husk, used as heterogeneous catalysts. The results showed significant changes in properties such as color, weight, crystallinity, chemical transformation, and functional groups with increasing calcination temperature. XRD analysis revealed the presence of potassium compounds in roasted rice husk due to leaching effects. The highest amounts of Ca0 and silicon (IV) oxide were observed at the highest calcination temperatures for eggshell and rice husk, respectively. Crystallite size increased for calcined Egg Shell between 600 ºC and 700 ºC, with KC having the largest size, while the size decreased for calcined Rice Husk with increasing temperature. FT-IR analysis indicated the presence of CO_3^{-2} in certain samples, suggesting higher decomposition and impurities. Based on the findings, KC (calcined at 700 ºC) was identified as the best catalyst for eggshell, and KD (calcined at 600 ºC) for rice husk, due to high crystallinity and low impurities.

REFERENCES

Chakraborty, R., Bepari, S., & Banerjee, A. (2010). Transesterification of soybean oil catalyzed by fly ash and egg shell derived solid catalysts. *Chemical Engineering Journal*, *165*(3), 798–805. https://doi.org/10.1016/j.cej.2010.10.019.

Faridi, H. and Arabhosseini, A. (2018) Application of Eggshell Wastes as Valuable and Utilizable Products: A Review. Research in Agriculture Engineering, 64, 104-114.

Faruque, M. O., Razzak, S. A., & Hossain, M. M. (2020). *Application of Heterogeneous Catalysts for Biodiesel Production from Microalgal Oil — A Review*.

Gerhard Knothe, (2010). The History of Vegetable Oil Based Diesel Fuels. The Biodiesel Handbook, Chapter 2, ISBN 978-1-893997-79-0.

Mat, R., Samsudin, R. A., Mohamed, M., & Johari, A. (2012). Solid catalysts and their application in biodiesel production. *Bulletin of Chemical Reaction Engineering and Catalysis*, *7*(2), 142–149. https://doi.org/10.9767/bcrec.7.2.3047.142-149

Mohadi, R., Anggraini, K., Riyanti, F., & Lesbani, A. (2016). Preparation Calcium Oxide From Chicken Eggshells. *Sriwijaya Journal of Environment*, *1*(2), 32–35. https://doi.org/10.22135/sje.2016.1.2.32-35

Mohadi, R., Anggraini, K., Riyanti, F., & Lesbani, A. (2016). Preparation Calcium Oxide From Chicken Eggshells. *Sriwijaya Journal of Environment*, *1*(2), 32–35. https://doi.org/10.22135/sje.2016.1.2.32-35

Olutoye, M.A., Adeniyi, O. D., & Yusuff A. S. (2016). Synthesis of Biodiesel from Palm Kernel Oil using Mixed Clay-Eggshell Heterogeneous Catalysts. (2016). *Iranica Journal of Energy and Environment*, *7*(3) https://doi.org/10.5829/idosi.ijee.2016.07.03.14

Pornchai, T., Imkum, A., & Apipong, P. (2016). *Effect of Calcination Time on Physical and Chemical Properties of CaO- catalyst Derived from Industrial-eggshell Wastes*. *March*, 2–6.

Sekifuji, R., Chieu, L. Van, Tateda, M., & Takimoto, H. (2021). *Solubility and physical composition of rice husk ash silica as a function of calcination temperature and duration*. 19–27. https://doi.org/10.30486/IJROWA.2021.1899156.1069

Zou, L., & Yang, H. (2019). Heterogeneous catalysts from biomass wastes for biodiesel production. Catalysts, 9(11), 916.