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# Effects of Calcination Temperature on the Physicochemical Behaviour of Catalysts Produced from Eggshell and Rice Husk


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***Authors' contributions***

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*


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## ABSTRACT


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 results of the study revealed notable changes in various properties such as color, mass, chemical



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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 from 600ºC to 700ºC led to enhanced crystallinity, with the highest average crystal size being 278.0374 nm, and chemical transformation in the eggshells. Conversely, the Rice husks exhibited decreased crystallinity (274.793 nm to 16.723 nm) but increased chemical transformation with higher calcination temperatures increasing from 600ºC to 800ºC. 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.*

## 1. 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 [1]. 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 [1]. 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 [2]. 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 ($SO_3$) [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 [4]. 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 [5,6]. 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 [7,8].

## 2. MATERIALS AND METHODS

### 2.1 Experimental Method

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

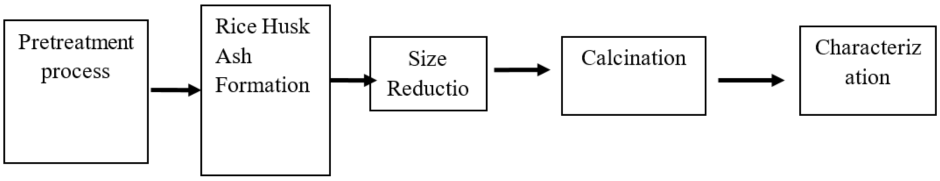


**Fig. 1. Flowchart of the synthesis of eggshell rice husk ash catalyst**

**Fig. 2. Roasted rice husk**

**Fig. 3. Washed and dried eggshell**

**Fig. 4. Calcined eggshells**

#### 2.1.1 Pretreatment and calcination of samples

**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 [9]. 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 [10]. 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 [11].

**Eggshell:** 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 [12]. 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.

### 2.2 Characterization of the Calcined Eggshell 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.

#### 2.2.1 X-ray diffraction analysis for mineral identification

The powdered samples were formed into pellets and sieved to a size of 0.074mm. 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 configured 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.

## 3. RESULTS AND DISCUSSION

The eggshells underwent decomposition through calcination in a furnace at various temperatures (600ºC, 650ºC, and 700ºC), resulting in a color change. Table 1 illustrates the observed color changes at different calcination temperatures. At 600ºC, the eggshells transformed into black powder, while a small amount of black-colored ash was produced at 650ºC. Finally, at 700ºC, 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 [4]. Also the calcined rice husks underwent decomposition during the calcination process in a furnace at varying temperatures (600ºC, 700ºC, and 800ºC). The samples exhibited an increasing change in color as the calcination temperature rose, as depicted in Table 1. At 600ºC, the rice husks resulted in a light ash powder, while a black-ash colored powder was obtained at 700ºC. Finally, at 800ºC, 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 [13].

### 3.1 Loss in Mass of the Raw Materials used

Table 2. 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 increases as calcination temperature increases and decomposition of $Ca_2SiO_4$ to $SiO_4$ When calcined Rice husk at different calcination temperatures from 600ºC to 700ºC to 800ºC. As showed in Table 2. 24.7% was loss when Eggshell was calcined at 600 ºC, at 650ºC there was 31.3% loss in mass of Eggshell and highest loss in mass of eggshell at 700ºC of about 35.7%. RH loss it's mass up to 75.2% by weight at 600ºC, and loss it's mass up to 84.6% at 700ºC while at 800 ºC there was mass loss up to 89.3% as shown in Table 2. 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.

### 3.2 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.

#### 3.2.1 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$^0$

**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.

**KA (eggshell calcined at 600 ºC):** Fig. 5 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 [14].

**KB (eggshell calcined at 650ºC):** Fig. 6 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 [14].

**KC (Eggshell Calcined at 700ºC):** Fig. 7 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 2Ө= 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 2Ө= 33º and 52º there is smaller amount of hydroxylapatite at 2Ө=38º [4].

**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.

**Table 1. 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 |

**Table 2. Loss in mass of the raw materials used**

| S/N | Material Used | Calcination temp ºC | 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% |

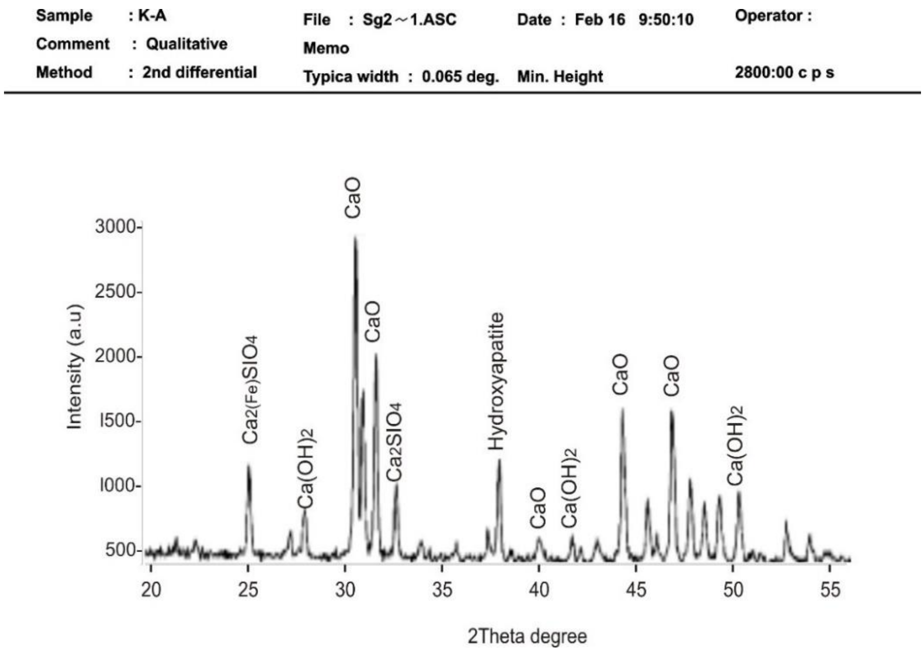


**Fig. 5. Eggshell Calcined at 600ºC (KA)**

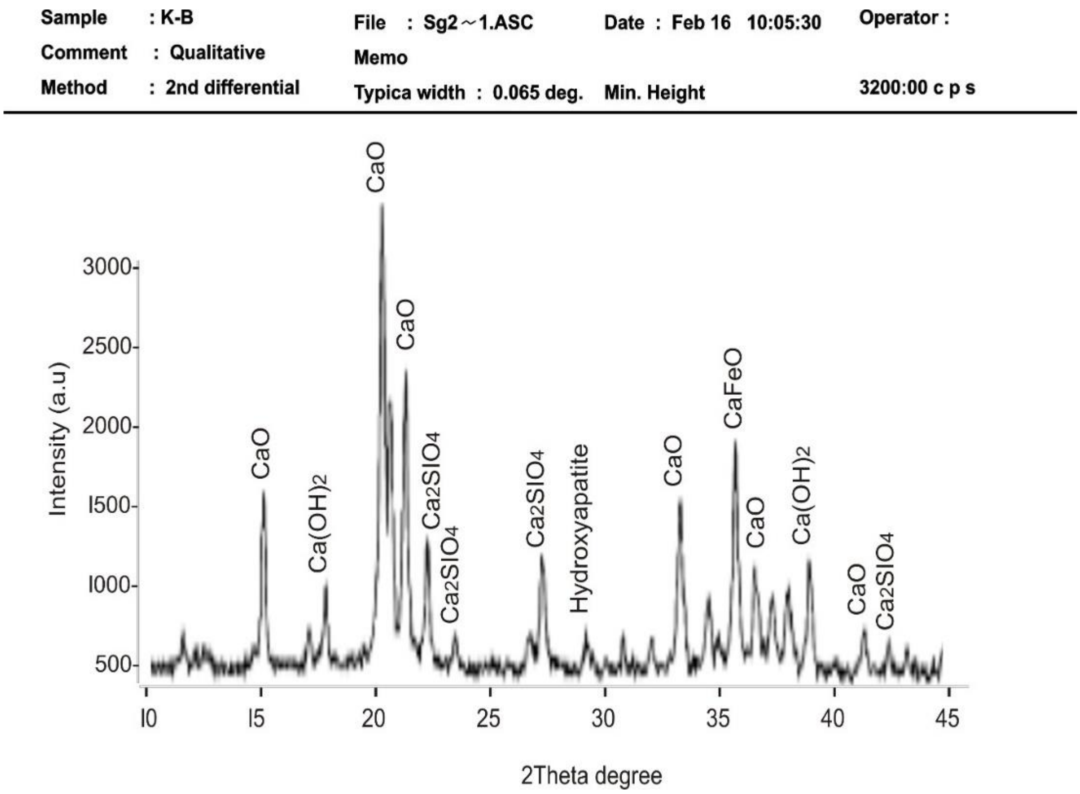


**Fig. 6. Eggshell Calcined at 650ºC (KB)**

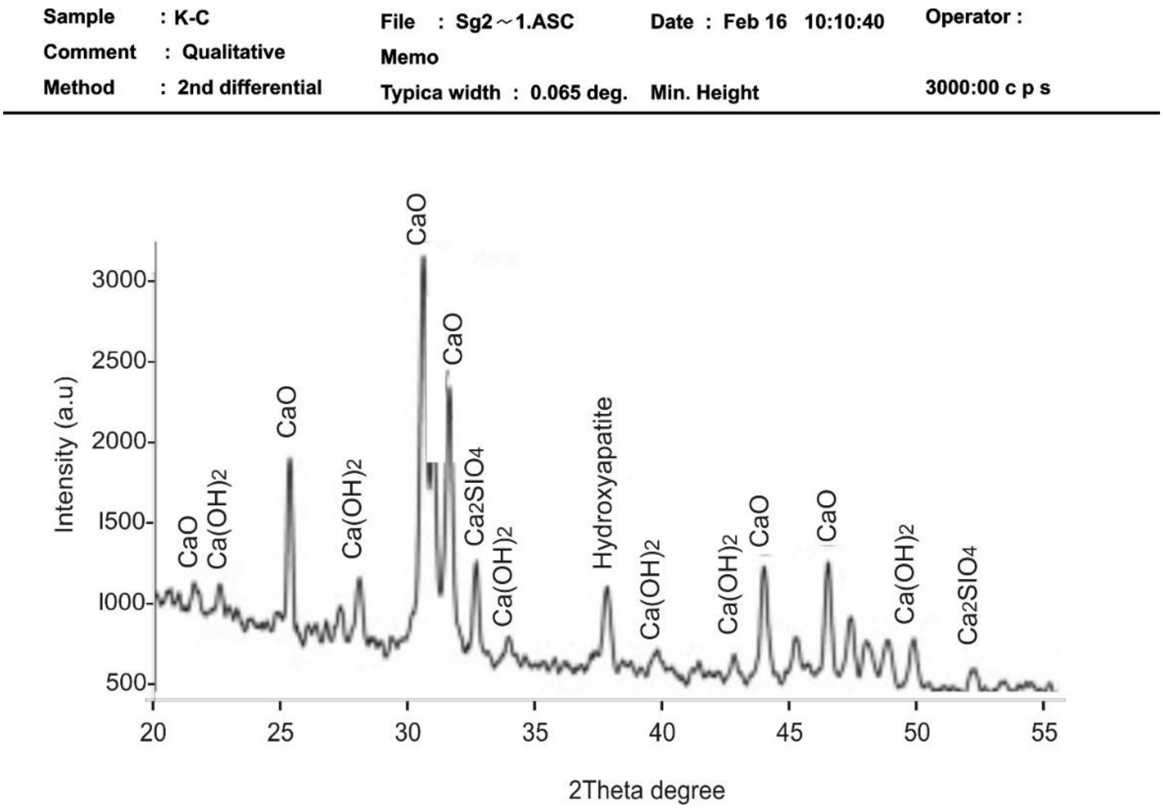


**Fig. 7. Eggshell Calcined at 700ºC (KC)**

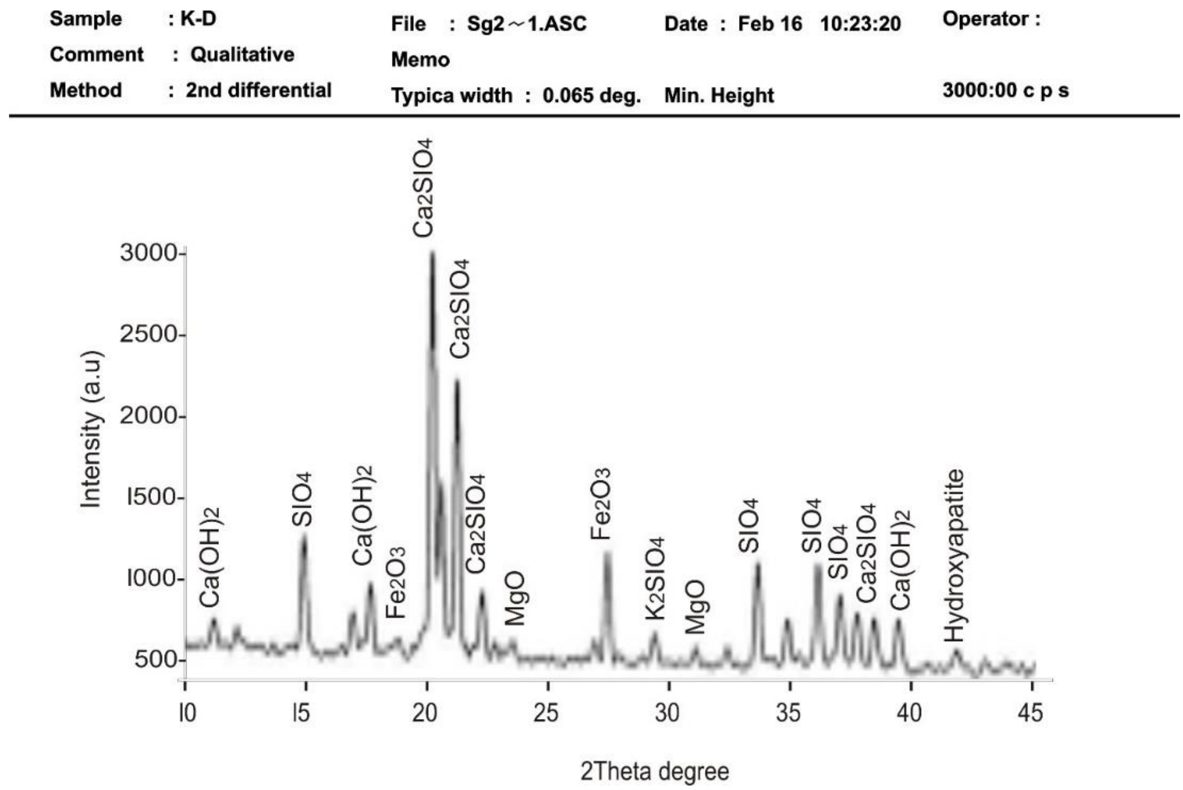


**Fig. 8. Rice Husk Calcined at 600ºC (KD)**

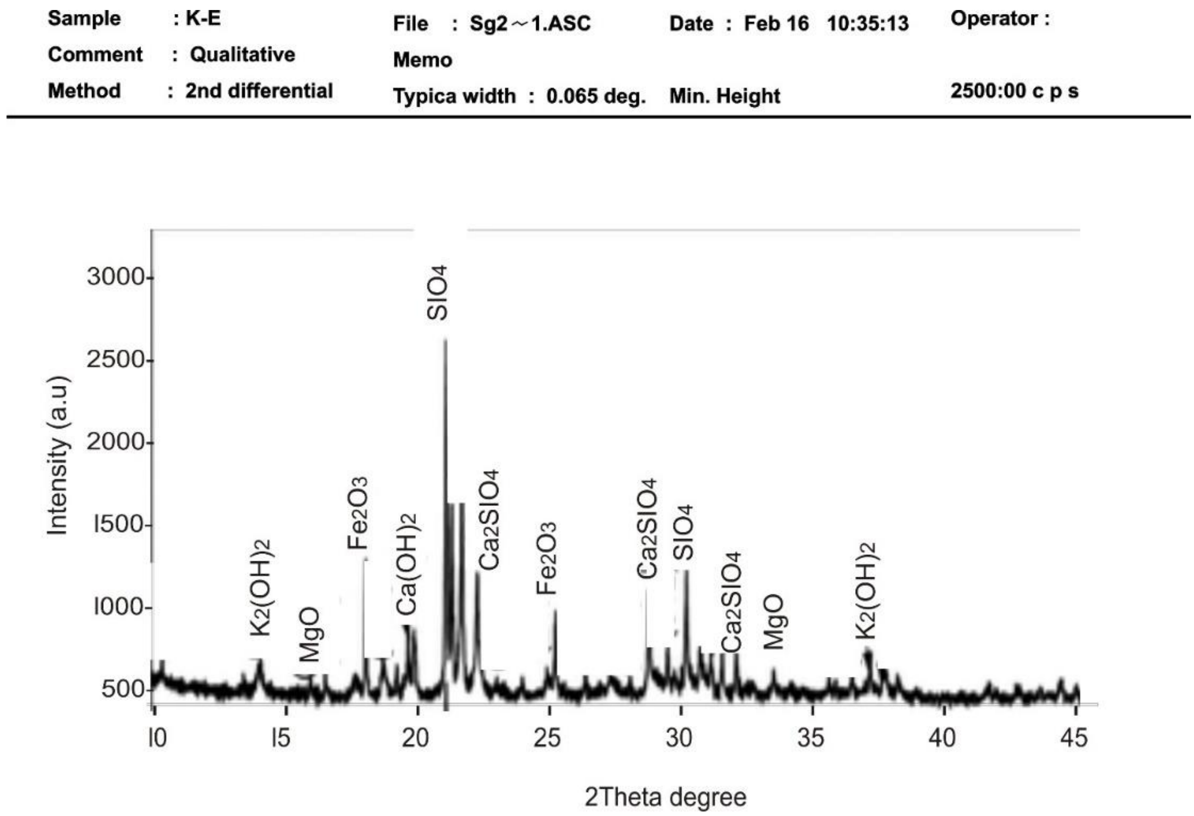


**Fig. 9. Rice Husk Calcined at 700ºC (KE)**

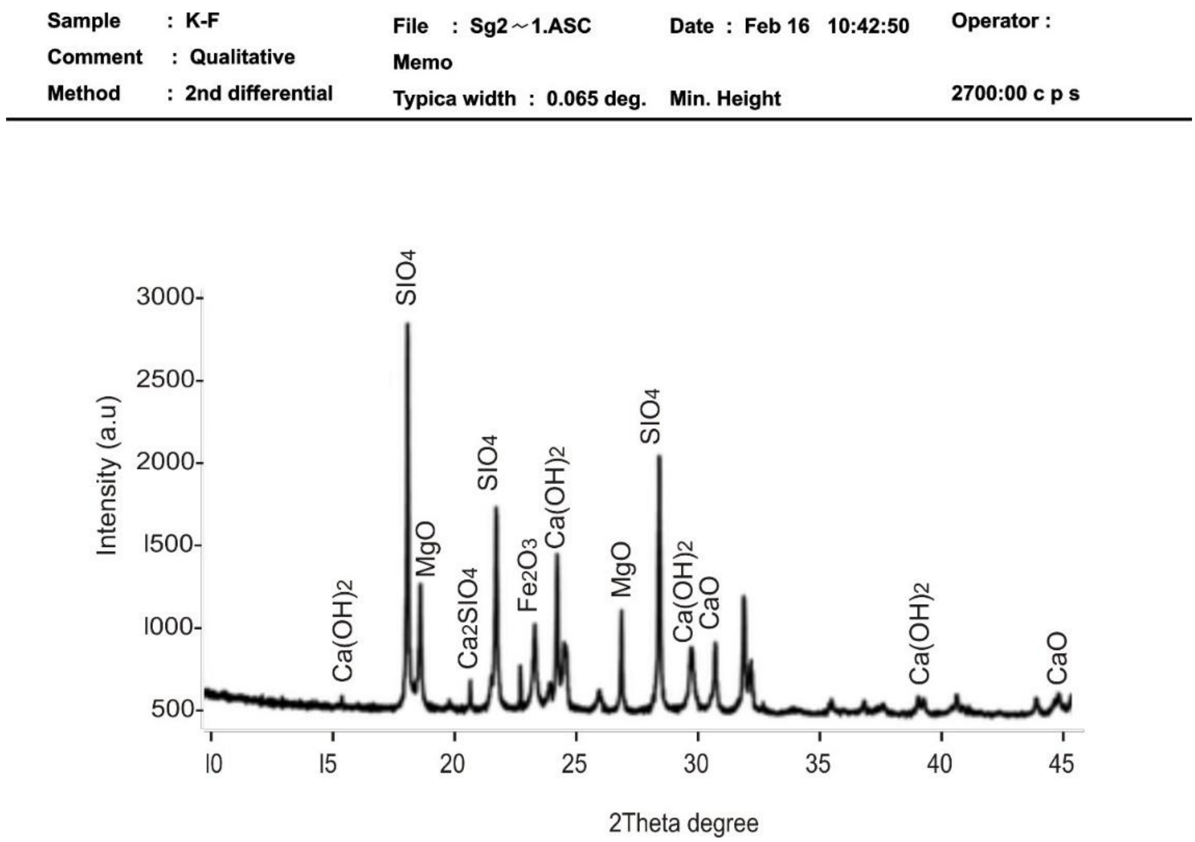


**Fig. 10. Rice Husk Calcined at 800ºC (KF)**

**Table 3. 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 |

**KD (rice husk calcined at 600ºC)**: The highest peak is at 2ϴ= 21º being the abundance component shows presence of $Ca_2SiO_4$ it exists at 2ϴ= 22º, 28º and 37º and $SiO_4$ is at 2ϴ= 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 $Fe_2O_3$.

**KE (rice husk calcined at 700ºC)**: 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 $Fe_2O_3$.

**KF (rice husk calcined at 800ºC)**: 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.

An examination of crystallite size changes with increasing temperature using the Scherrer equation reveals that the size of the crystallites in calcined Eggshell increases between 600ºC and 700ºC. 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 600ºC, has the highest crystallite size, while KF exhibits the smallest crystallite size among all the samples.

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

**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.

**KA (eggshell calcined at 600ºC) :** 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 3761.41$cm^{-1}$, 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.

**KB (eggshell calcined at 650ºC):** 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.

**KC (eggshell calcined at 700ºC):** 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 [15]. 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 [14].

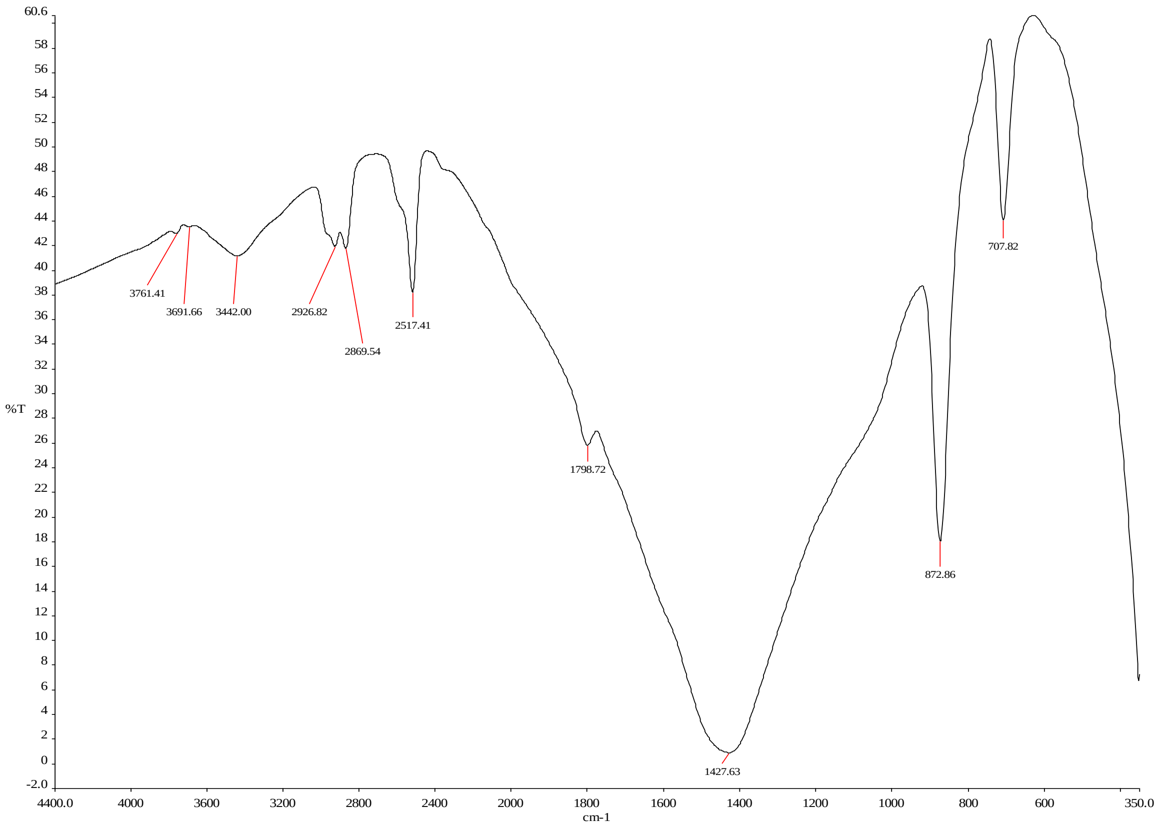


**Fig. 11. Eggshell Calcined at 600ºC (KA)**

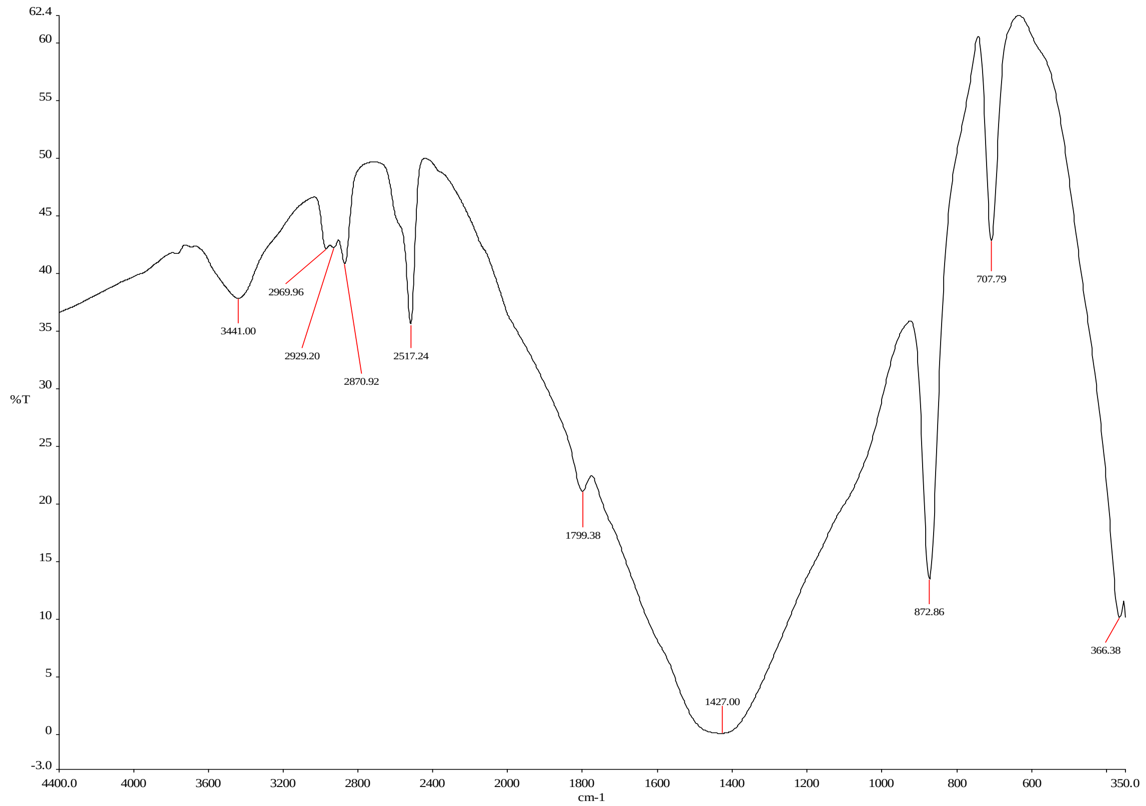


**Fig. 12. Eggshell Calcined at 650ºC (KB)**

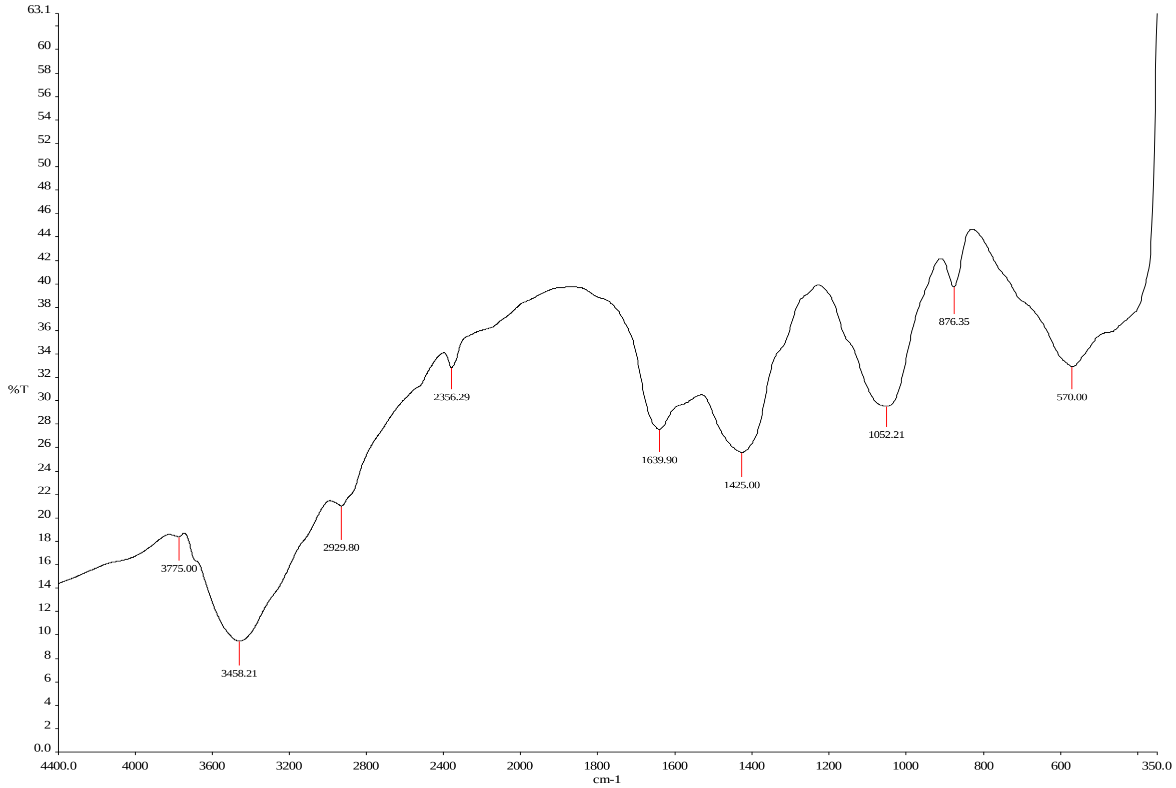


**Fig. 13. Eggshell Calcined at 700ºC (KC)**

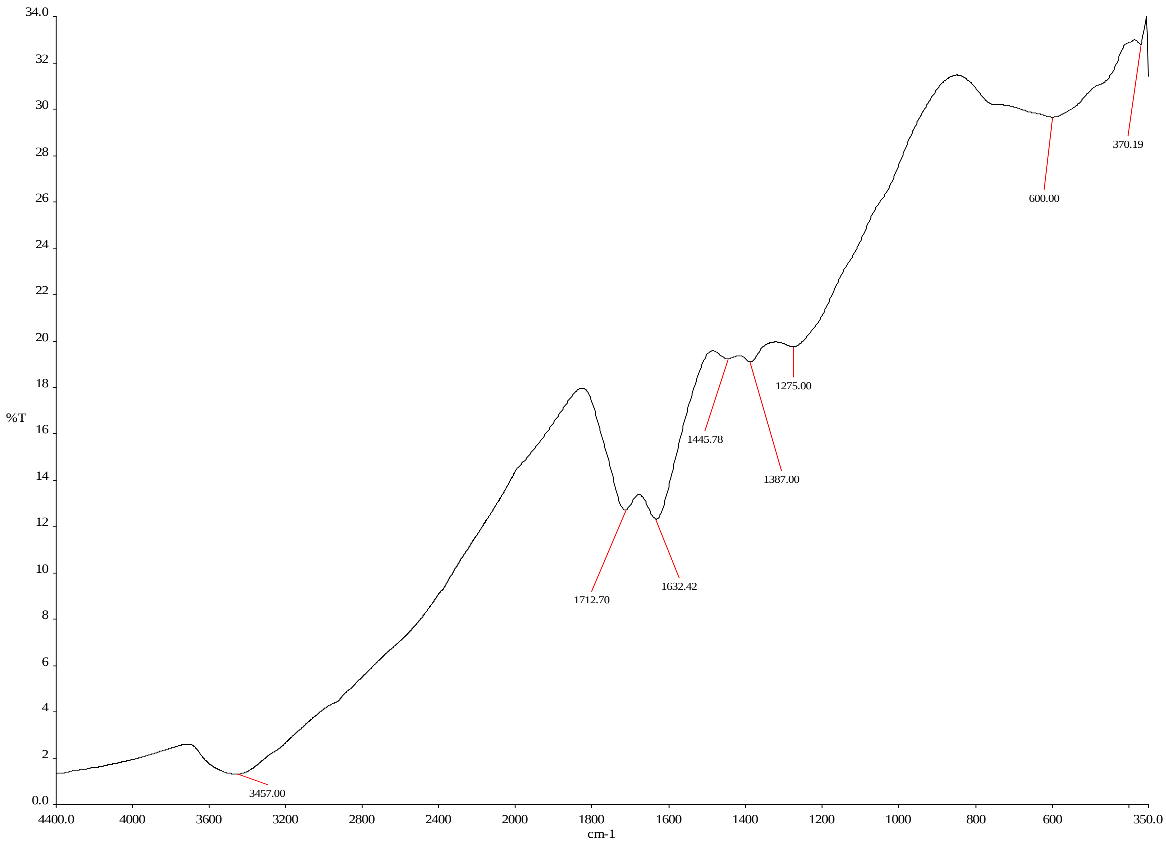


**Fig. 14. Rice Husk Calcined at 600ºC (KD)**

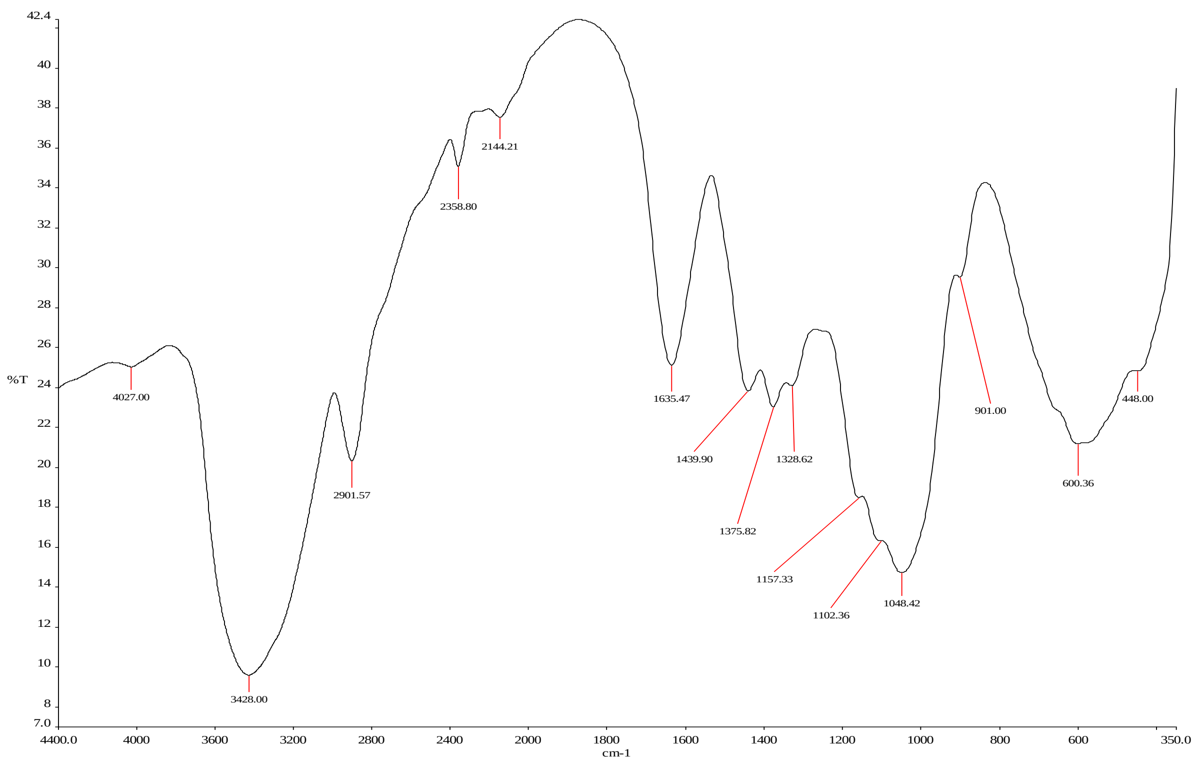


**Fig. 15. Rice Husk Calcined at 700ºC (KE)**

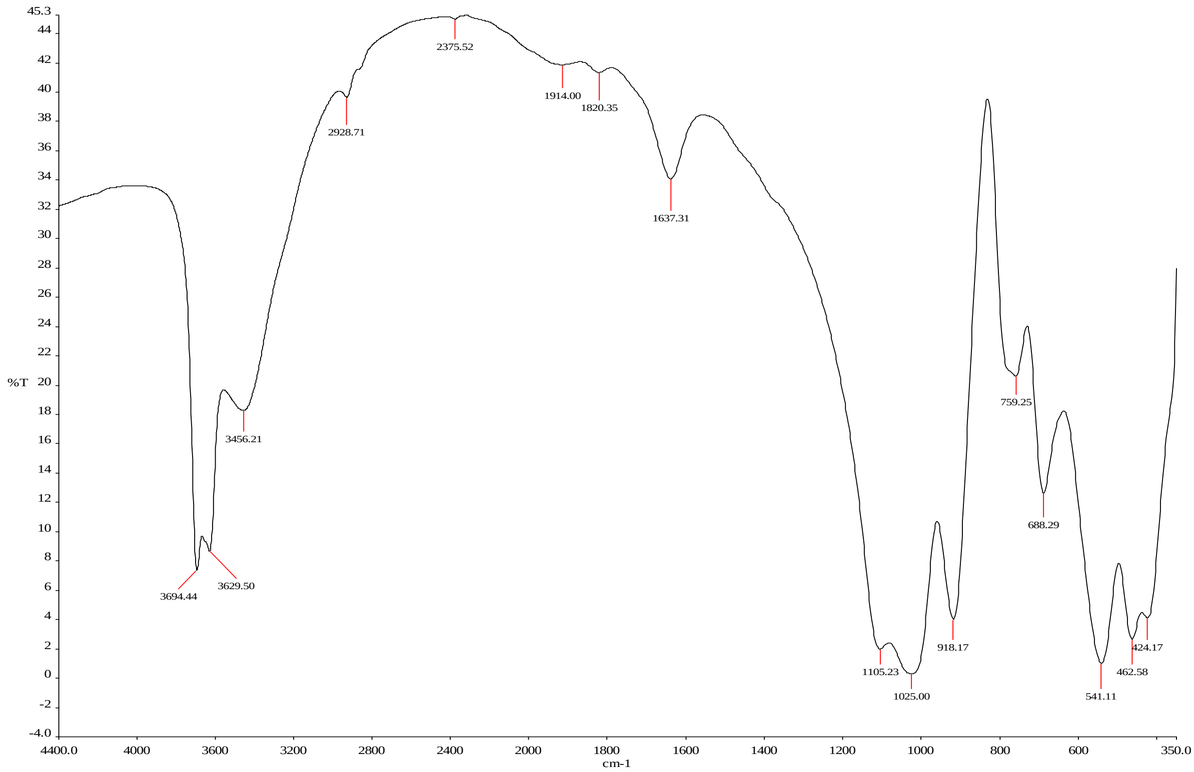


**Fig. 16. Rice Husk Calcined at 800ºC (KF)**

**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 16 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 [16,17].

**KD (rice husk calcined at 600ºC):** 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 because of bending vibration of the $CH_3$ group in cellulose as a residue and 1712.70 $cm^{-1}$ can be because of C=O bond in carbonyl group Hemicellulose.

**KE (rice husk calcined at 700ºC):** The spectrum is a complex one with more than 5 bands, the 4027.00 $cm^{-1}$ and 3428.00 $cm^{-1}$ are because 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 bond C-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 bond in another phase.

**KF (Eggshell Calcined at 800ºC)**: 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. 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 CaO and silicon (IV) oxide were observed at the highest calcination temperatures for eggshell and rice husk, respectively. Crystallite size increased for calcined Eggshell 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 700ºC) was identified as the best catalyst for eggshell, and KD (calcined at 600ºC) for rice husk, due to high crystallinity and low impurities.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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