

# Characterisation of Cassava Peel-Derived Silica at Varied Combustion Temperatures: Identifying the Optimum for Filler Applications

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DOI: <https://doi.org/10.30880/rpmme.2025.06.02.025>

## Article Info

Received: 24 April 2025

Accepted: 12 August 2025

Available online: 10 December 2025

## Keywords

Cassava peel, Silica extraction,  
Agricultural waste, Controlled  
combustion, Acid leaching

## Abstract

This study looks at the possibility of using cassava peel, which is a common waste product from agriculture and industry, as a long-term source of high-purity silica by controlled burning (400–800 °C) and acid leaching. FTIR, SEM, and TGA were used to look at the structural and thermal properties of the silica that was taken out. The results showed that silica made at 600–700 °C had the best properties, such as high purity, an amorphous structure, and better thermal stability. The cassava peel-derived silica had the same physical and chemical properties as commercial silica, showing that it could be used as a cheap, environmentally friendly filler for polymers. This also shows how useful agricultural waste can be in developing sustainable materials engineering.

## 1. Introduction

Cassava peel, an abundant agricultural waste, as a renewable and eco-friendly source of silica for industrial applications [1]. Cassava peel, often discarded despite its high organic content, poses environmental concerns due to its slow decomposition and the potential for pollution [2]. However, when subjected to controlled thermal and chemical processing, cassava peel ash can yield high-purity silica, offering a sustainable alternative to conventional silica derived from mining [3]. Silica extracted from agricultural waste like cassava peel has shown promising characteristics such as thermal stability, mechanical strength, and compatibility with biodegradable polymers [4]. These properties make it an attractive reinforcing filler in the production of composite materials. One of the key factors influencing the quality of extracted silica is the combustion temperature used during calcination. This temperature determines the silica's crystallinity where amorphous silica is generally more effective for filler applications and its surface morphology, which affects its interaction with polymer matrices [5].

Although other biomass sources such as rice husk and sugarcane bagasse have been extensively studied, cassava peel remains relatively underexplored [6]. This research seeks to address that gap by investigating the effect of different combustion temperatures on the physicochemical properties of silica derived from cassava peel ash. Through systematic analysis, the study aims to determine the optimal thermal conditions that yield silica with desirable characteristics for industrial use.

## 2.0 Methodology

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## 2.1 Material Preparation

The main raw material was cassava peels, which are high in silica and easy to find as farm waste. We got the peels from Azhar Manufacturing Sdn. Bhd. in Rengit, Batu Pahat. The peels were washed well with clean water to get rid of dirt and other impurities on the surface. This was done to make sure they were clean and avoid contamination. Then, they were left out in the open air to dry in the sun for one to two days until they were brittle and completely dry, which is an important step for lowering the moisture content before burning [7]. After the peels were dry, they were put away in tight plastic bags to keep them from absorbing more water and getting contaminated from the outside. An analytical balance was used to weigh 100 grams of the dried cassava leaves for each experiment. This was done to make sure that the sample size was the same for all temperature treatments in the combustion process.

## 2.2 Combustion Process

A Protherm muffle furnace was used to carefully burn the dried cassava peels, turning them into ash. This ash is then used as the starting material for silica extraction. 5 different temperatures were used to heat each batch of 100 g for 2 hours which is the parameter is 400°C, 500°C, 600°C, 700°C, and 800°C. The crucible had to be clean. This range of temperatures was chosen so that we could see how changing the heat level affects the chemical and physical changes that happen in the biomass as it turns into silica-rich ash [8]. So that there was no thermal shock or contamination, the oven was left to cool down naturally to room temperature after it was heated. The ash that was made at each temperature level was carefully collected using clean tools and kept in sealed cases to keep it in good shape for further sieving, leaching, and analysis. The figure 2.1 shows the peel visual after combustion process.

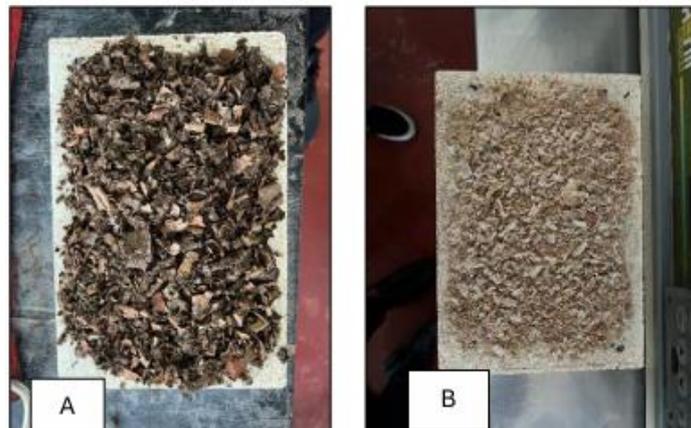


Figure 2.1: A) Peel before combustion and B) Peel after combustion

## 2.3 Ash Collection

Ashes were carefully collected with clean stainless steel tools to keep them from getting dirty after the calcination process was over and the furnace had dropped to room temperature. After that, a 140-mesh sieve (106  $\mu\text{m}$ ) was used to get rid of any big pieces and make sure the bits were all the same size so they could be treated chemically. The ash that had been sieved was then weighed at the Polymer Laboratory UTHM using a Mettler Toled analytical balance. The goal is to keep track of the output at each temperature of combustion. After that, each sample was given a label with its combustion temperature and kept in clean, airtight cases to keep its quality and stop it from absorbing water or becoming contaminated before the acid leaching process.

## 2.4 Silica Extraction Process

Following the sieving process, the ash was subjected to an acid leaching treatment to eliminate residual impurities, particularly metallic elements that may interfere with the structural and chemical integrity of the silica [9]. The method employed involved using 1 M hydrochloric acid (HCl), a commonly used reagent for removing basic metal oxides such as iron (Fe), aluminium (Al), and calcium (Ca) which are often present in biomass ash. For each sample, exactly 11 grams of ash were measured and placed into a clean 250 mL glass beaker, followed by the addition of 110 mL of HCl solution, maintaining a consistent solid-to-liquid ratio of 1:10. The beaker was then

positioned on a hot plate with a magnetic stirrer and heated to a temperature between 60°C and 80°C. Continuous stirring was applied throughout the one-hour treatment period to ensure maximum contact between the acid and the ash particles, facilitating the dissolution of undesirable compounds. As the reaction progressed, a change in the solution's colour and clarity was observed, indicating the leaching of impurities into the liquid phase. After one hour, the mixture was allowed to cool slightly before being filtered using Whatman filter paper to separate the solid silica-rich residue from the acidic filtrate. The retained solid was then washed multiple times with distilled water to ensure that all traces of HCl were removed. Washing was continued until the pH of the rinse water reached a neutral level (around pH 7), confirming that the sample was free from residual acid. This neutralised, purified residue represented the preliminary form of bio-silica, now ready for drying and further characterisation in the following steps

## 2.5 Drying Process

After the acid leaching and thorough rinsing process, the neutralised ash was prepared for drying to obtain purified silica in powder form. The filtered residue was transferred to a clean glass dish or ceramic crucible and placed in a drying oven set at 100 °C. The drying process was carried out for 12 hours to ensure complete removal of moisture. This step is essential to stabilise the silica and prevent further reactions or contamination during storage and testing. Once dried, the silica was allowed to cool to room temperature before being stored in airtight containers to preserve its quality for subsequent characterisation and analysis. .

## 2.6 Characterization & Analysis

A series of advanced characterisation techniques were conducted to evaluate their structural, elemental, and thermal properties. These analyses aimed to determine the effect of combustion temperature on the quality and suitability of cassava peel-derived silica for use as a filler material in composite applications. Firstly, Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify the functional groups present in the silica. FTIR spectra were collected in the range of 4000–400 cm<sup>-1</sup> to detect characteristic vibrations such as Si-O-Si symmetric and asymmetric stretching, as well as Si-OH bonds, which serve as indicators of silica purity and structure. Secondly, the Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray (EDX) analysis was used to examine the surface morphology and elemental composition of the silica samples. SEM provided detailed images at high magnifications, revealing surface features such as particle shape, porosity, and agglomeration. Meanwhile, EDX offered quantitative data on the elemental content, specifically measuring the proportions of silicon (Si), oxygen (O), and any residual elements like aluminium (Al) or iron (Fe). A higher Si/O ratio and reduced presence of impurities would indicate a successful extraction. Finally, Thermogravimetric Analysis (TGA) was carried out to assess the thermal stability and decomposition behaviour of the silica samples under increasing temperature. Each sample was gradually heated in a nitrogen environment while monitoring the weight change over time. The TGA curves helped determine the temperature range at which moisture and volatile components were released and confirmed the thermal resistance of the resulting silica. Collectively, these three characterisation methods provided a comprehensive evaluation of the physicochemical qualities of the silica extracted at each combustion temperature, helping to identify the most optimal condition for filler applications.

$$\text{Silica Yield (\%)} = \left( \frac{\text{Weight of Silica}}{\text{Weight of Ash}} \right) \times 100$$

## 3.0 Result and Discussion

### 3.1 Silica Yield Analysis

The silica yield analysis shows that combustion temperature plays a major role in determining how much usable silica can be extracted from cassava peel. At lower temperatures like 400°C, the combustion was incomplete, resulting in higher ash weight and lower silica yield (25.2%). This is because not all organic materials were burned off, making it harder to isolate silica. As the temperature increased to 500°C and 600°C, the ash weight decreased and the silica yield improved to 43.8% and 45.9% respectively. These temperatures allowed better decomposition of the biomass and more effective release of silica. The highest yield was recorded at 700°C, reaching 54.3%, with the lowest ash amount and a dark brown colour indicating clean combustion and high silica purity. However, at 800°C, although the silica weight remained high, the yield dropped to 24.1%, likely due to over-combustion or crystallisation, which reduces extractable silica. This pattern confirms that 600°C to 700°C is the most effective range for obtaining high-yield silica from cassava peel, with 700°C being the optimal temperature.

### 3.2 Fourier Transform Infrared Spectroscopy Analysis

The FTIR (Fourier Transform Infrared Spectroscopy) analysis was conducted to identify the functional groups present in the silica samples obtained at different combustion temperatures. Across all samples, characteristic peaks associated with silica were observed, particularly in the regions around 1100  $\text{cm}^{-1}$ , 800  $\text{cm}^{-1}$ , and 470  $\text{cm}^{-1}$ . These peaks correspond to the Si-O-Si asymmetric stretching, Si-O symmetric stretching, and Si-O bending vibrations, respectively, confirming the presence of silica structures. The results silica at different temperature FTIR examination are shown in Figure 3.1 through 3.5

Table 3.1: FTIR spectral data for functional groups

Peak No	Wave number $\text{cm}^{-1}$	Functional Group
1	3358-3362	O-H stretching
2	2996	C-H bending
3	1006-1043	Si-O-Si asymmetric stretching
4	800-898	Si-O symmetric stretching

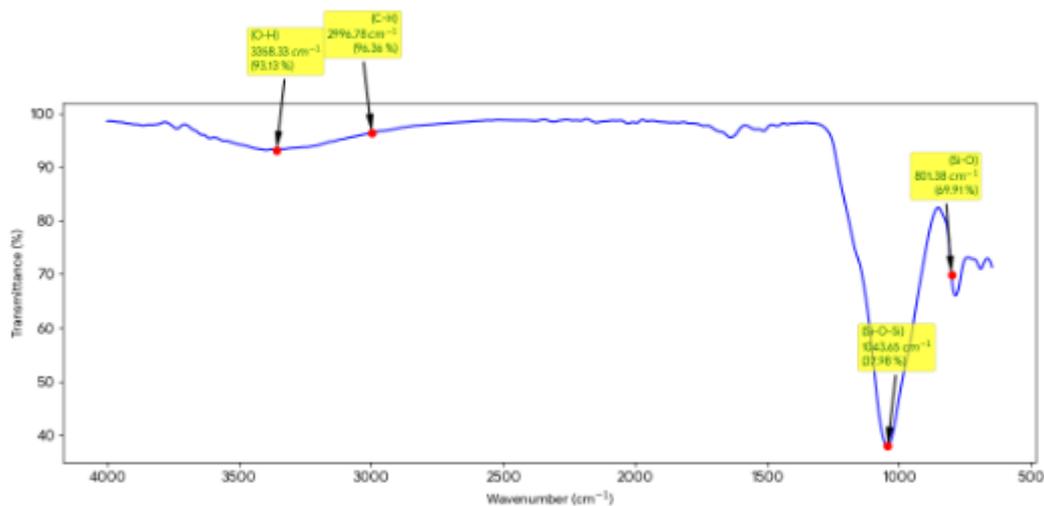


Figure 3.1: FTIR analysis on sample 400°C

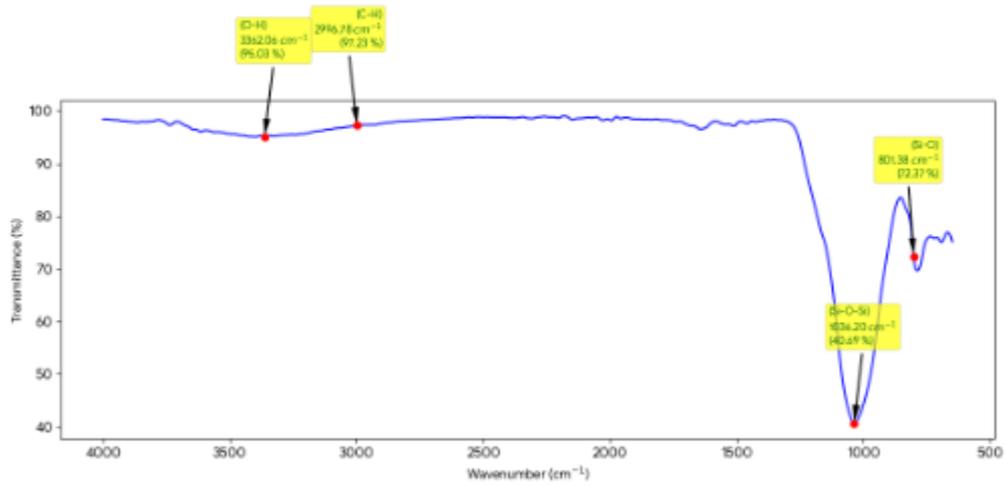


Figure 3.2: FTIR analysis on sample 500°C

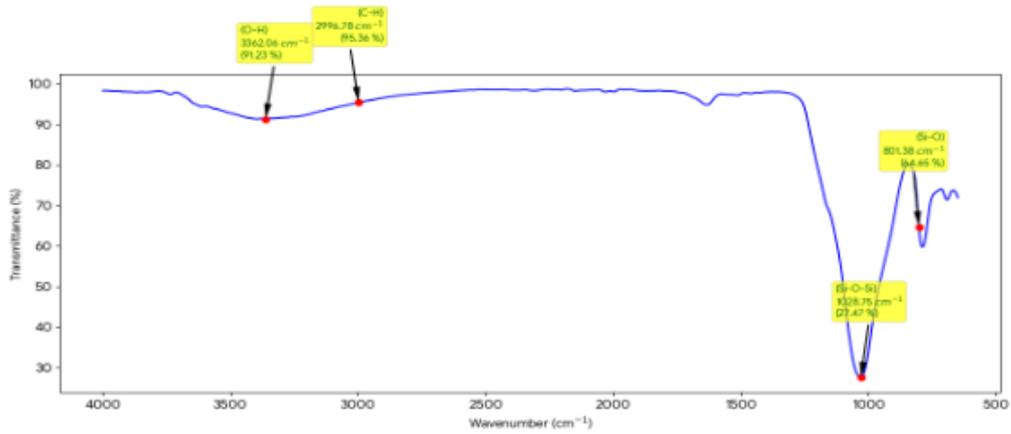


Figure 3.3: FTIR analysis on sample 600°C

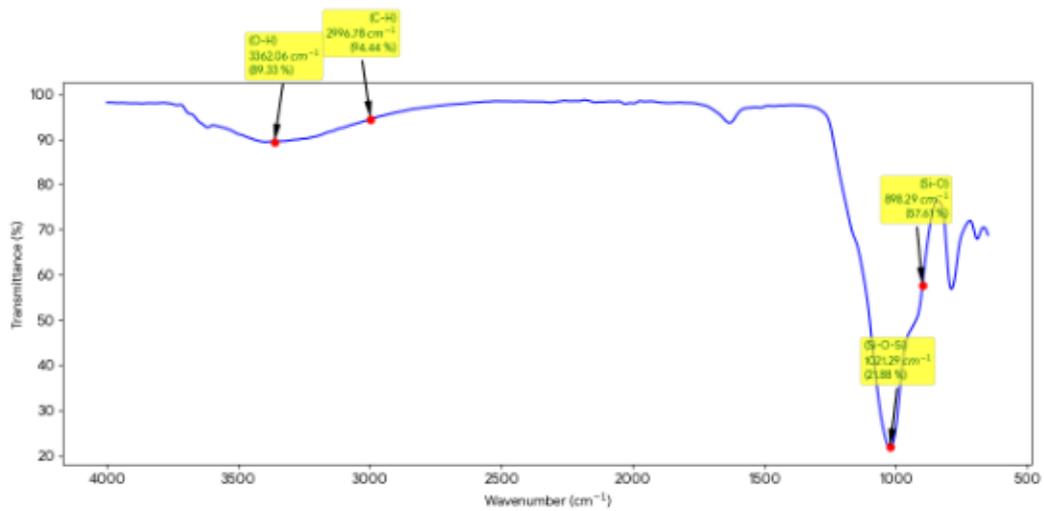


Figure 3.4: FTIR analysis on sample 700°C

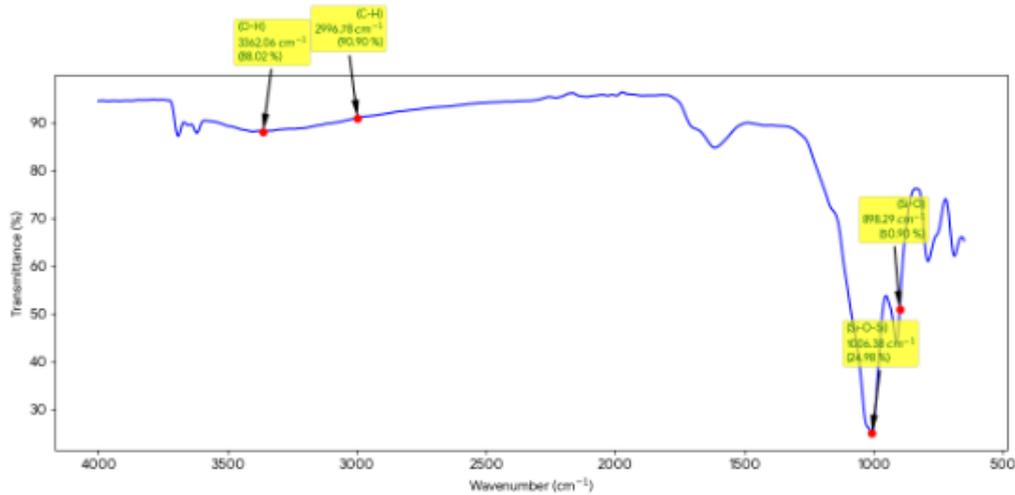


Figure 3.5: FTIR analysis on sample 800°C

The FTIR spectrum of silica samples extracted at different combustion temperatures reveal key changes in chemical structure. At 400°C and 500°C, broad peaks at  $\sim 3360\text{ cm}^{-1}$  and  $\sim 2996\text{ cm}^{-1}$  indicate residual moisture (O-H) and organic matter (C-H), while peaks near  $1043\text{--}1036\text{ cm}^{-1}$  and  $801\text{ cm}^{-1}$  confirm initial silica formation. At 600°C, sharper Si-O-Si and Si-O peaks appear at  $1028\text{ cm}^{-1}$  and  $801\text{ cm}^{-1}$ , indicating improved silica structure. The 700°C sample shows the sharpest peaks at  $1021\text{ cm}^{-1}$  and  $898\text{ cm}^{-1}$ , confirming high-purity, well-formed silica with minimal impurities. However, at 800°C, silica is still present ( $1006\text{ cm}^{-1}$  and  $898\text{ cm}^{-1}$ ), but with reduced peak intensity, suggesting possible structural degradation. Overall, 700°C is identified as the most effective temperature for producing high-quality silica.

### 3.3 SEM and EDX Analysis

The SEM analysis of silica samples at various combustion temperatures shows clear differences in particle morphology. At 400°C and 500°C, the particles are large, irregular, and porous, indicating incomplete combustion with residual organic matter. At 600°C, particle size becomes finer with improved definition, though some clustering remains. The best morphology is observed at 700°C, where particles are uniform, smoother, and more spherical, with minimal porosity indicating effective combustion and high silica purity. At 800°C, while fine particles are present, signs of sintering and partial fusion appear, reducing surface area and potentially affecting performance as a filler. Overall, 700°C produces the most favourable silica structure, supporting previous findings from FTIR and yield analysis. The visual result at 700°C and 800°C are shown in Figure 3.6 and 3.7

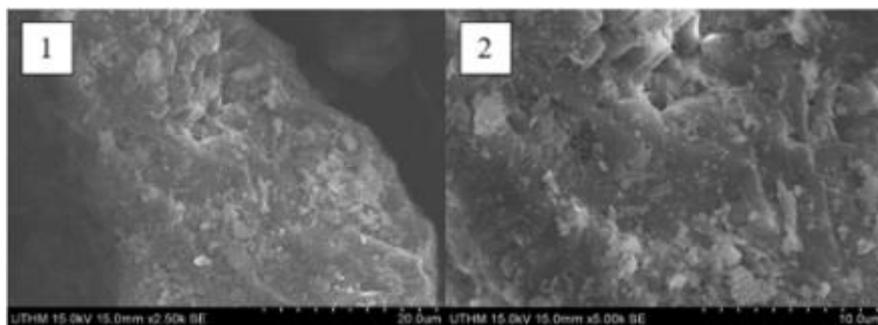


Figure 3.6: SEM images of visual morphology surface of (1) silica at 700°C 2.5k (2) silica at 700°C 5k

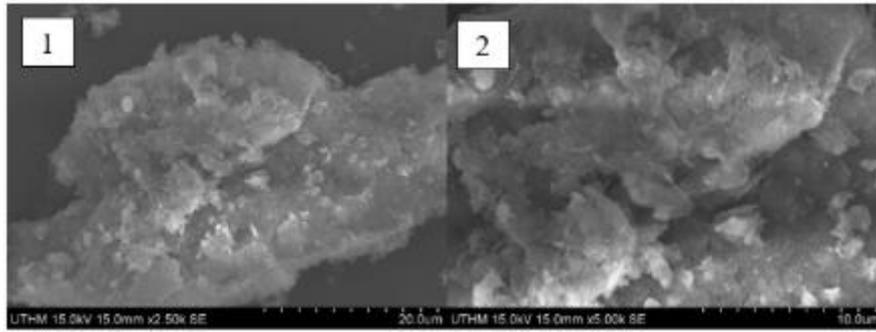


Figure 3.7: SEM images of visual morphology surface of (1) silica at 800°C 2.5k (2) silica at 800°C 5k

The EDX analysis shows that higher calcination temperatures improve the silica purity in cassava peel ash. At 800°C, the highest silicon content (40.39 wt%) and lowest potassium (0.40 wt%) indicate excellent purity. The 700°C sample also has high silicon (37.64 wt%) with low aluminium and potassium, making it efficient and energy-saving. In contrast, the 500°C sample shows lower silicon (27.35 wt%) and higher impurities, suggesting incomplete combustion. Samples at 400°C and 600°C have moderate silicon levels (around 36 wt%) but slightly more aluminium. Overall, 700°C and 800°C are the most effective temperatures for producing high-purity silica by reducing unwanted elements like aluminium and potassium.

### 3.5 Thermogravimetric Analysis

TGA analysis is to evaluate the thermal stability and degradation behaviour of the silica samples. The analysis has carried out by heating the sample under controlled conditions and monitoring weight changes, which reflect the presence of volatile components and the decomposition profile. Figure 3.8 shows the thermogravimetric analysis result of all the cassava silica sample.

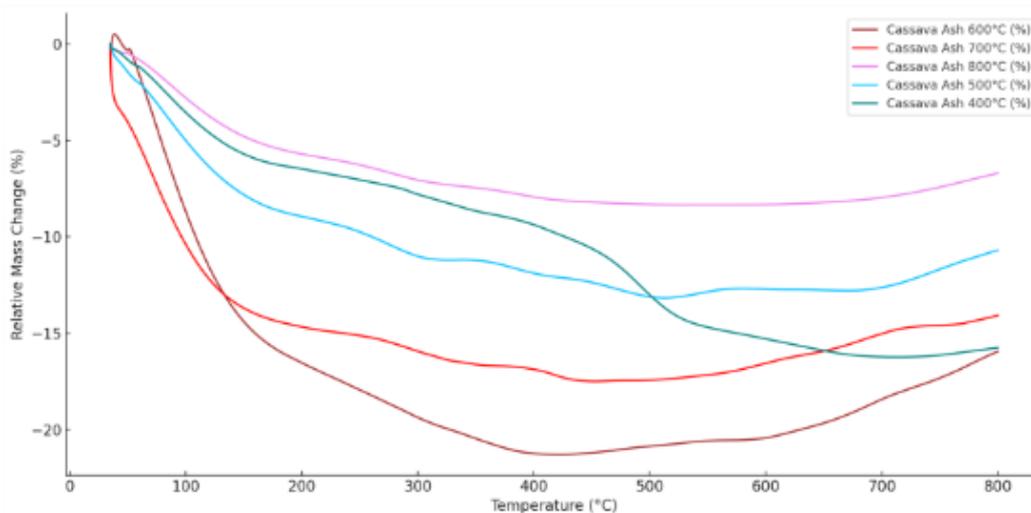


Figure 4.11: Relative mass change for all the silica sample

All samples showed a multi-stage weight loss pattern in the TGA analysis, with the most significant drop occurring below 150°C due to moisture evaporation. The 400°C and 500°C samples experienced greater mass loss, indicating incomplete combustion and higher residual organics. In contrast, the 600°C and 700°C samples exhibited more stable thermal profiles, with the 700°C sample showing the lowest total weight loss, suggesting higher purity and thermal stability. The 800°C sample remained relatively stable but displayed minor curve fluctuations at higher temperatures, possibly due to crystallisation or sintering effects, which may influence the consistency of the silica produced.

Table 3.2: Value of initial decomposition temperature, final decomposition temperature and weight percentage of residue

Sample	Initial decomposition temperature (°C)	Final decomposition temperature (°C)	Weight of residue (%)
400°C	35.42	713.6	-15.75
500°C	35.36	512.7	-10.69
600°C	35.08	422.56	-15.94
700°C	35.02	452.64	-14.07
800°C	35.4	531.2	-6.70

Based on table 3.2, shows that all cassava peel ash samples began to lose weight around 35°C due to moisture evaporation. The sample burned at 400°C had the highest final decomposition temperature and a relatively high residue, meaning it still contained many unburned components. The 500°C sample also retained more weight, indicating incomplete combustion. In contrast, the 600°C and 700°C samples decomposed earlier and had lower residues, showing they were more stable and had fewer remaining organics. Among them, the 700°C sample offered the best balance, with good thermal stability and a moderate residue of -14.07%, suggesting it was well-combusted and suitable for silica extraction. The 800°C sample had the lowest residue at -6.70%, showing most of the volatile material had already been removed, though its early decomposition may hint at some changes in silica structure.

#### 4. Conclusion

This study proved that cassava peel, a common agricultural waste, can be used to produce high-quality silica through a process of controlled burning and acid treatment. By experimenting with different combustion temperatures ranging from 400°C to 800°C, the research found that 700°C is the most effective temperature. At this point, the silica yield was highest (54.3%), and the quality based on purity, structure, and thermal stability was at its best. The silica produced at 700°C had properties similar to commercial silica, making it suitable for use as a reinforcing filler in biodegradable plastics and other composite materials. Lower temperatures resulted in incomplete combustion, leaving behind organic matter, while higher temperatures like 800°C risked damaging the silica structure by causing crystallisation. Overall, the study supports the use of cassava peel as a low-cost, eco-friendly alternative to mined silica and helps reduce environmental pollution by turning waste into a useful product.

#### 5. Recommendation

It is recommended that future production of cassava peel-derived silica follow the optimal combustion temperature of 700°C, which gives the best results in terms of yield and quality. To confirm its suitability in industry, further testing should be done on the mechanical properties of the final product, such as strength and flexibility. For a more sustainable approach, researchers can also try using natural or less harmful acids instead of hydrochloric acid during the leaching process. In addition, studies should be carried out to assess the cost and environmental impact of scaling up the process for commercial use. This method should be introduced to cassava-processing industries as part of a circular economy model to reduce waste and create valuable products. Governments and environmental agencies can help support this innovation by offering incentives, training programs, and awareness campaigns. Lastly, the use of cassava peel silica can be expanded to other fields such as rubber, paint, concrete, and packaging industries, increasing its value and usage.

#### Acknowledgement

The authors thank to Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia for its support.

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