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Characterisation of Cassava Peel-Derived Silica at Different Combustion Temperatures

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Cassava peel, silica extraction, agricultural waste, controlled combustion, acid leaching

Abstract

The growing need for sustainable materials and the environmental burden of agro industrial waste highlights the urgency of transforming biomass into high value resources. Cassava peel, typically discarded as waste, contains silica a valuable mineral composed of silicon and oxygen. 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). Acid leaching and drying process. FTIR, SEM, and TGA were used to look at the structural and thermal properties of the silica. The results showed that silica produced at 600-700°C had the best properties, with the 700°C sample achieving the highest silica yield of 54.3%, silicon content of 41.45wt%, and demonstrating sharp FTIR peaks at 1021cm^{-1} and 898cm^{-1} indicating a well-formed amorphous structure. It also exhibited the lowest total weight loss in TGA analysis and a moderate residue of 14.07%, confirming superior thermal stability and high purity. These properties are comparable to those of commercial silica, suggesting its potential as a cost-effective and 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. 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. Silica extracted from agricultural waste like cassava peel has shown positive capability characteristics such as thermal stability, mechanical strength, and compatibility with biodegradable polymers [2]. 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 [3].

Silica or silicon dioxide (SiO_2) is a common and versatile material used across various industries. Extracting silica from agricultural waste, such as cassava peel, offers an environmentally sound alternative to traditional methods [4]. This bio-derived silica is particularly promising as a reinforcing filler in composite materials, owing to its desirable properties, including thermal stability, mechanical strength, and compatibility with biodegradable polymer systems [5]. The research indicates that optimal silica characteristics, such as high purity and amorphous structure, are achieved when cassava peel is combusted between 600°C and 700°C.

Although other biomass sources such as rice husk and sugarcane bagasse have been extensively studied, cassava peel remains relatively underexplored. 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 [6]. Materials derived from recycled aluminium alloys such as AA6061 have also demonstrated notable mechanical performance and impact resistance under high-velocity conditions, offering insight into the broader potential of waste-derived materials in engineering applications [7].

2. Materials Preparation

The main raw material was cassava peels, which are high in silica and easy to find as farm waste near in Rengit, Batu Pahat, Malaysia. The peels were washed with clean water to get rid of dirt and other impurities on the surface. This was done to make sure cassava peels were clean and avoid contamination. Then, it was left out in the open air to dry under the sun for two days until become brittle and completely dry, which is an important step for lowering the moisture content before burning [8]. After the drying process take place, the peels were put away in tight plastic bags to keep them from absorbing more water and getting contaminated from the open area. The cassava peels used to weigh of 100 grams for each experiment. This was done to make sure that the sample size remains for all temperature treatments in the combustion process.

2.1 Combustion Process

A Protherm muffle furnace was used to carefully burn the dried cassava peels, turning them into ash. Five different temperatures were used to heat each batch of 100 g for 2 hours which the parameter sample 400°C, 500°C, 600°C, 700°C, and 800°C. The crucible is first to be cleaned. This range of temperatures was chosen so that it 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 [9]. 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 keep in to for further process sieving and leaching. Fig. 1 shows visual of peels after combustion process.

(a) (b)

Fig. 1 *(a) Peel before combustion; (b) Peel after combustion*

2.2 Ash Collection

Ashes were carefully collected with clean stainless-steel tools to keep them from getting dirty after the calcination process was completed and the furnace had dropped to room temperature. A 140-mesh sieve (106 µm) was used to get rid of any large pieces and make sure the bits were all the same size intake to be treated chemically. The ash that had been sieved was then weighed using a Mettler Toled analytical balance. 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.

2.3 Silica Extraction Process

The ash was then 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 [10]. 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 to 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 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 [11].

2.4 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 [12]. 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

2.5 Characterization and 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. Equation 1 was used to calculate the percentage of silica yield. Firstly, Fourier Transform Infrared Spectroscopy (FTIR) was employed to identify the functional groups present in the silica [13]. FTIR spectra were collected in the range of 4000–400 cm^{-1} 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 which 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 [14]. 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. 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 [15].

$$Silica\ Yield(\%) = \left(\frac{Weight\ of\ Silica}{Weight\ of\ Ash}\right) \times 100 \tag{1}$$

3. Results 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 due to incomplete burned off of organic materials. 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 [16]. 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 results silica at different temperature FTIR examination are shown in Table 1 and Fig. 2. The analysis was conducted of 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 cm^{-1}, 800 cm^{-1}, and 470 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 [17].

Table 1 *FTIR spectral data for functional groups*

Peak no.	Wave number (cm^{-1})	Functional group
1	3358-3362	O–H stretching
2	2996	C–H bending
3	1028-1043	Si–O–Si asymmetric stretching
4	800-898	Si–O symmetric stretching

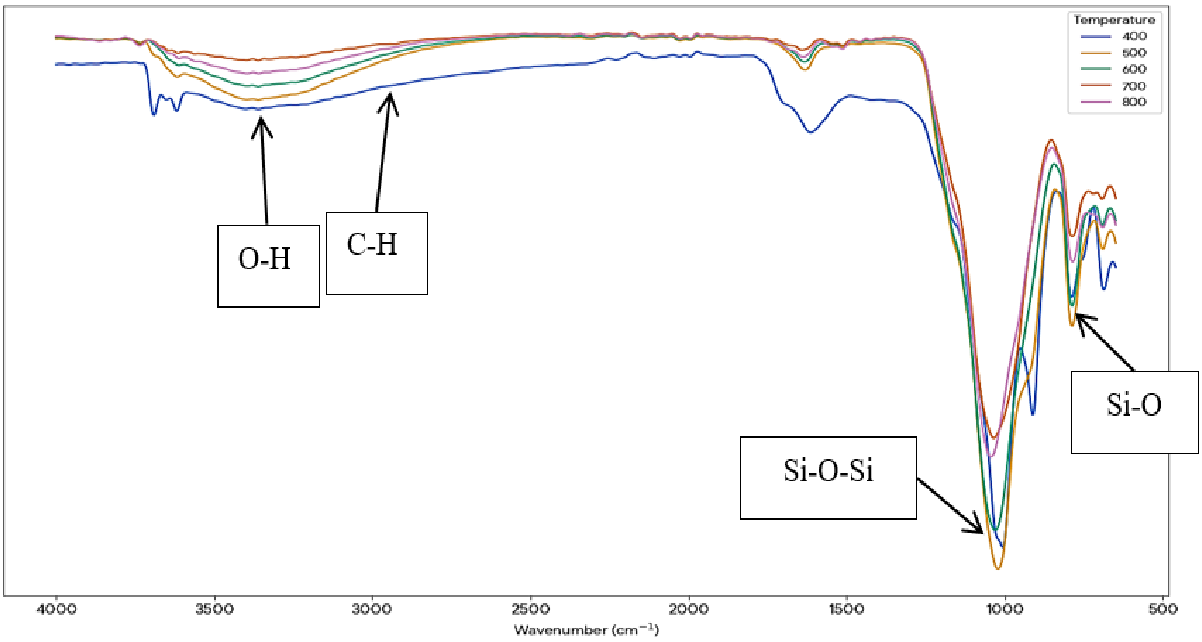

Fig. 2 *FTIR analysis on 400°C-800°C*

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 ~3360 cm^{-1} and ~2996 cm^{-1} indicate residual moisture (O–H) and organic matter (C–H), while peaks near 1043–1036 cm^{-1} and 801 cm^{-1} confirm initial silica formation. At 600°C, sharper Si–O–Si and Si–O peaks appear at 1028 cm^{-1} and 801 cm^{-1}, indicating improved silica structure.

The 700°C sample shows the sharpest peaks at 1021 cm^{-1} and 898 cm^{-1}, confirming high-purity, well-formed silica with minimal impurities. However, at 800°C, silica is still present (1043 cm^{-1} and 898 cm^{-1}), but with reduced peak intensity, suggesting possible structural degradation [18]. 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 [19]. 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 Fig. 3 and Fig. 4.

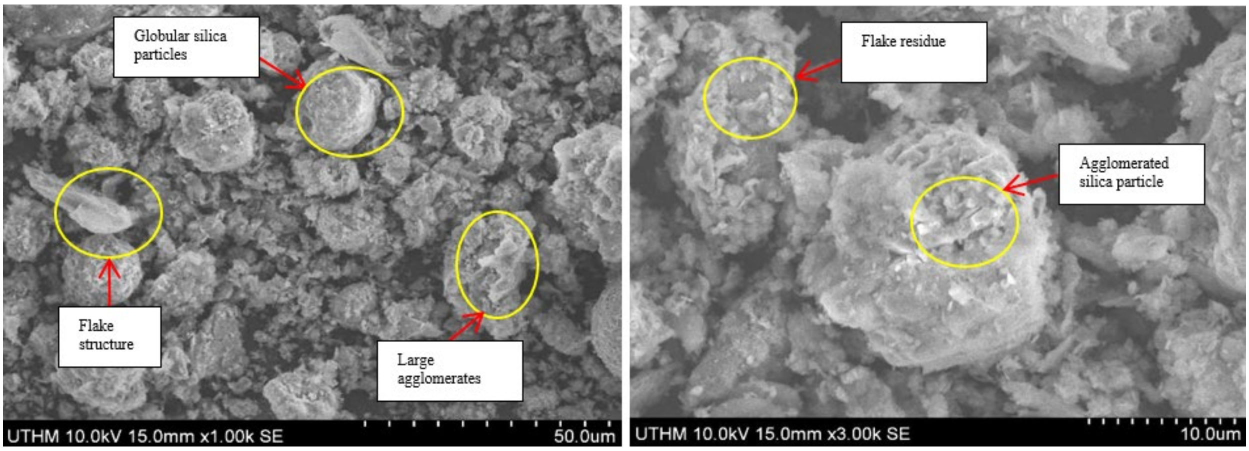

Fig. 3 *SEM images of visual morphology surface of silica at 700°C*

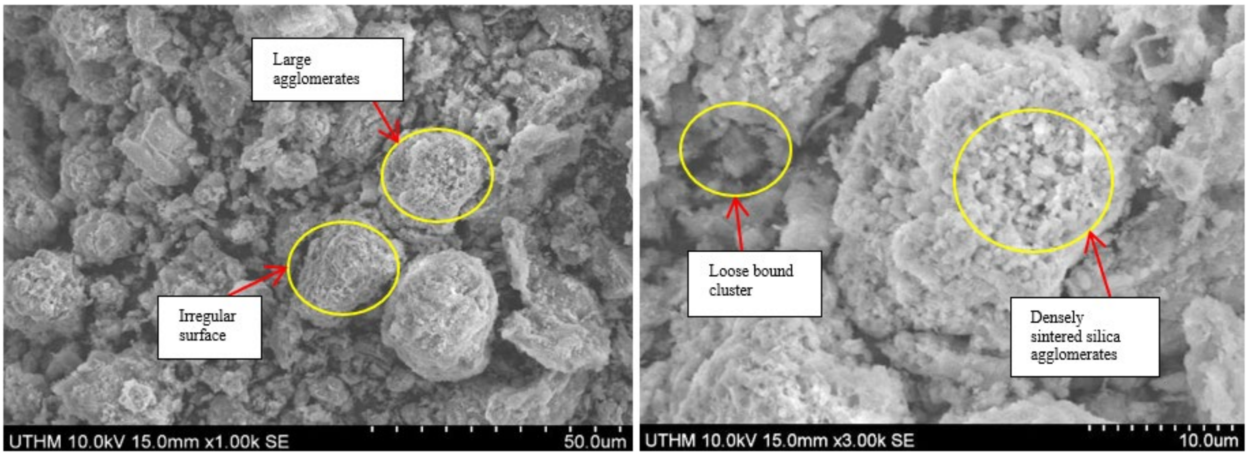

Fig. 4 *SEM images of visual morphology surface of silica at 800°C*

Table 2 shown the Energy Dispersive X-ray Spectroscopy (EDS) analysis of cassava peel-derived silica across various combustion temperatures shows a distinct trend in silica development. At 400°C, the silicon content was 36.85 wt% with 63.15 wt% oxygen, indicating the early stages of silica formation but with possible residual organic matter. At 500°C, the silicon content decreased to 31.59 wt% while oxygen increased to 68.41 wt%, suggesting incomplete combustion and the presence of hydroxyl groups or unburnt biomass. However, combustion at 600°C marked a turning point, where silicon content rose to 38.06 wt% and oxygen dropped to 61.94 wt%, indicating better organic removal and enhanced silica purity [20].

The optimal condition was observed at 700°C, where the silicon content peaked at 41.45 wt% and oxygen settled at 58.55 wt%. This composition closely resembles the ideal Si:O ratio in amorphous silica, suggesting well formed, high-purity silica with minimal impurities ideal for reinforcing filler applications. Although 800°C showed a slightly higher silicon content (42.18 wt%), the risk of sintering or crystallization at this temperature may compromise the surface area and reactivity of the silica. Therefore, while silica formation improves with increasing temperature, 700°C is considered the most effective point for producing structurally suitable and chemically pure silica from cassava peel.

Table 2 *Energy Dispersive X-ray Spectroscopy (EDS) analysis*

Temperature	Element	Weight (%)	Atomic (%)
400°C	O K	63.15	75.05
	Si K	36.85	24.95
500°C	O K	68.41	79.18
	Si K	31.59	20.82
600°C	O K	61.94	74.07
	Si K	38.06	25.93
700°C	O K	58.55	71.26
	Si K	41.45	28.74
800°C	O K	57.82	70.64
	Si K	42.18	29.36

3.4 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 [21]. Fig. 5 shows the thermogravimetric analysis result of all the cassava silica sample.

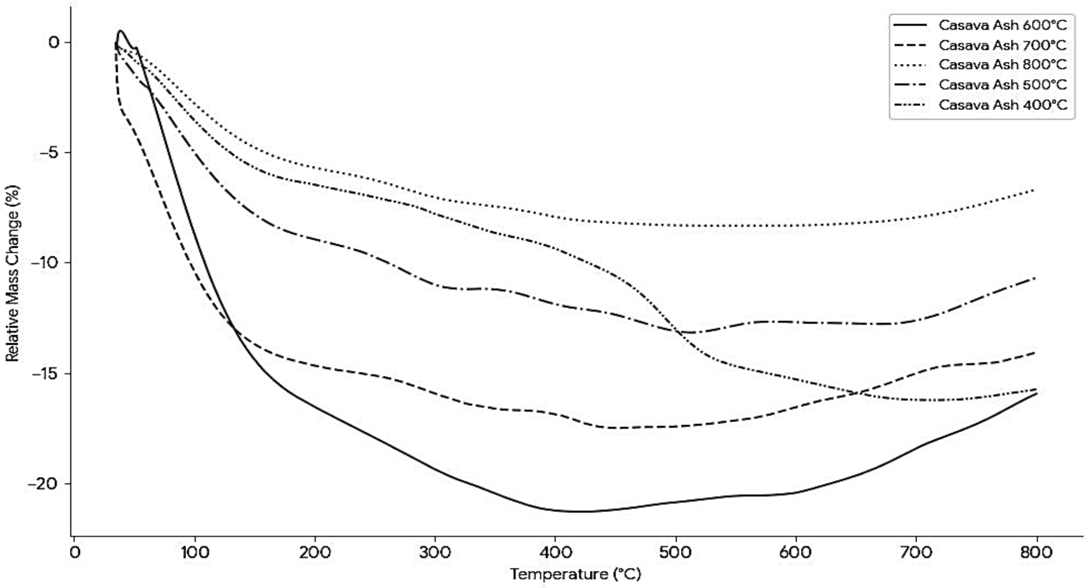

Fig. 5 *Relative mass change for all the silica sample*

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 [22]. 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 shows that all cassava peel ash samples began to lose weight around 35°C due to moisture evaporation. The sample were 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 more stable and had fewer remaining organics. 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.

Table 3 *Initial decomposition temperature, final decomposition temperature and weight percentage of residue*

Temperature	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

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 [23]. 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 almost 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 [24].

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