Deciphering the Physicochemical and Thermo – Structural Evaluation of Peanut, Coconut ash using Advanced XRD, FTIR & SEM with EDX Techniques

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Abstract
The study presents a comprehensive characterization of amorphous silica derived from two agricultural waste materials: Coconut shell and peanut shell. A systematic investigation was conducted to evaluate their physical, chemical, and morphological properties for obtained silica. Fourier transform infrared (FTIR) spectroscopy was used to determine the chemical structure of the samples. SEM analysis confirmed well – developed irregular structures across all samples, with distinct morphological variations based on precursor materials. Thermo gravimetric analysis revealed that peanut shells exhibit a higher cellulose decomposition temperature at 350^0C, whereas coconut shells manifest in this phenomenon at 322^0C. The results show that the peanut shell ash had a higher heating value as they exceeded that of the coconut shell by 0.75 percent. These materials will offer great opportunities not only to reduce the environmental footprint, but also to increase the value of agricultural waste by promoting a circular economy.

Keywords: Peanut shell ash, Coconut shell ash, Agricultural waste, Amorphous Silica

1. Introduction

Nowadays, the global population enhances the sustainable production of crop plants to achieve global food demand. High abundance of those agro residues must be handled by applying some technological processes to retrieve their beneficial components. Silica work as a precursor for various applications includes ceramics, concrete, chromatography and optical materials. The employment of high purity silica in industrial applications will be costly because its processing temperature is high. Thus, the silica synthesis from agro waste materials will be an economical method to reduce the processing cost and to deal with some technological challenges. Agricultural wastes have been utilized for the production of several useful materials to reduce their disposal challenges. These wastes are often generated and disposed of indiscriminately or burnt, which results in environmental pollution with adverse effects on climate [1]. The side effects of climate have been a major challenge globally because it leads to the death of about 160,000 people annually, according to the World Health Organization (WHO) [2-5]. India

utilized 78% of its landmass, about 2,564,065 square kilometers, to produce several agricultural products and ranked third in eggs, oranges, coconuts, tomatoes, peas, and beans [6]. Literature has shown that agricultural wastes have been used as solid fuel briquettes, adsorbents, cellulose, activated carbon, silica, silicon, refectory, ceramic products, livestock feed, inhibitors, bio fuels, construction materials, lignin, and composite reinforcement [7-22]. Silica has been utilized for several applications across industries such as pharmaceuticals, archeology, biomedical, electronics, and silicon feedstock (birth of semiconductor revolution). Silica occurs naturally as quartz with the largest percentage in the earth's crust. It is crystalline in nature with low reactivity. Amorphous silica, on the other hand, finds applications in many areas owing to its advantage of being highly reactive. The non-crystalline type has been synthesized from sodium silicate [35], water glass [23], tetra ethoxylsilane (TEOS) [24-26]. It has also been produced from agricultural wastes [19] using several methods as reported in reviews [27, 28]. It is usually prepared by reacting sodium carbonate powder and quartz sand at high temperatures to form sodium silicate, which will then react with sulphuric acid to precipitate silica [26]. The methods used for synthesis from agricultural wastes involve a combination of processes like acid treatment, partial burning (charring), burning, calcination, leaching, enzymatic treatment [31], pyrolysis, hydrolysis, and sol-gel [28 - 30]. Among wastes generated in India, coconut wastes stand out because they liter our farmlands, residential and industrial areas because of coconut flesh/juice's economic and social importance. Coconut is a fruit of an Arecaceae plant (palm family) and can be found everywhere across India. India accounts for more than 11 % of the total world production after Indonesia and the Philippines [32]. The fruit is 55 % coir, 15 % shell, and 30 % flesh (meat) and juice [33]. The main part is the meat, where coconut oil and meals are produced. The coir (fibrous part) has been found to be useful for mats, ropes, sacks, door mats, brushes, boat caulks, compost, and fiber for mattresses [34,36]. Groundnut shells, also known as peanut shells or hulls are the outer covering of groundnut seeds. They are a by product of the agricultural processing of peanuts, typically discarded after the seeds are extracted for silica production. Groundnut shells are abundantly available agrowaste in India as it is the second largest country. Groundnut, also known peanut, belongs to the scientific name Fabaceae generally referred as the legume, bean or pea family (Royal Botanical gardens 2013). The reasons include (i) cheap cost of raw materials (ii) high percentage of silica content in the agricultural waste; (iii) comparable silica quality (iv)high energy content (v) low energy consumption.

In this study, it is easy and simple prepare for producing precipitated silica from discarded peanut shell and coconut husk and also compare the all the characterization. In the present work is a combination of bio resource engineering and applied chemistry and would develop a latest method to protect the natural environment by utilizing useful bio resource in the vast environment.

2. Experimental Detail

2.1 Raw Materials

Groundnut shells and coconut husks were collected from the local farmers in Tamilnadu, India. All the chemicals used were analytical reagent. Other materials used were nitric acid and sodium

hydroxide pellets. All the chemicals used directly and also methanol, distilled water were used as a solvent in sol – gel process.

2.2 Characterization

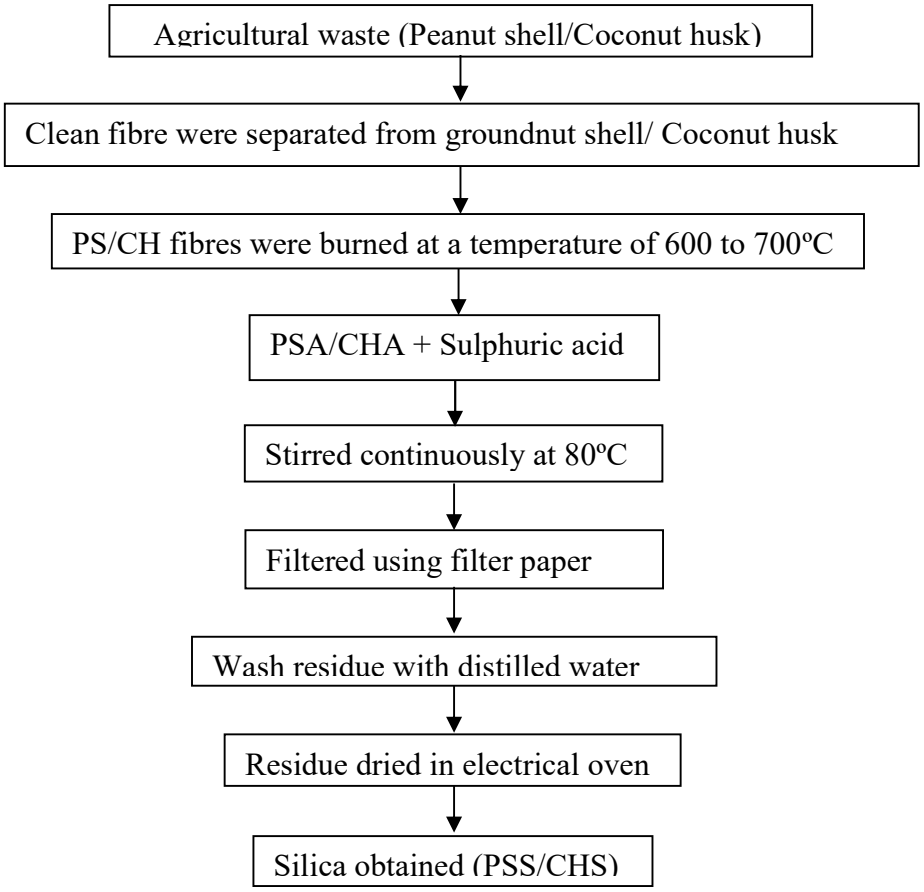
Powder X-ray diffraction pattern was obtained from a Rigakuminiflex diffract meter for 2Θ values from 100 to 900 using Cukα target at wavelength of λ = 1.5406Å. The FTIR analysis was carried out in a Perkin Elmer Spectrum 1000 using the KBr pellet method. Scanning electron microscopy (SEM), (JEOl – JSM – 6360LV) was used to record the morphology of the prepared materials and the elemental analysis was carried out by energy dispersive analysis (EDX) and Treated rice husk ash TGA and DTA analysis were performed in thermo gravimetric equipment (Pyris7, Perkin – Elmer).

3. Experimental Procedures:

3.1 Method for Peanut Shell Ash/Coconut Husk Ash

The main gram is separate from the coir and shell in this process, and moisture, volatile matter, and carbon are removed to generate ash at varying temperatures using muffle furnace. The resulting groundnut shell powder labeled as CHA & PSA was stored in an airtight container.

3.2 Method for Coconut Husk Ash (CHA) and Peanut Shell Silica (PSS)



Schematic diagram1. Silica preparation of Peanut shell silica/ Coconut husk silica

The soluble oxides of some metals are then removed using acid treatment. The sol – gel method initially with the formation of silicate solution from the ash and the neutralization of an alkaline solution to precipitate silica gel which is dispersed in NaOH solution in schematic diagram1.

4. Results and Discussion

4.1. Phase Analysis of Bio Silica from CHA

All extraction process involved the HCl treatment, calcinations at a temperature around 500 to 600ºC and NaOH treatment. We observed a change in the color of the ash from light brown to white, with the final result being bio silica. Fig.1 (a-c) shows the XRD patterns of various temperatures and finally CHS. The existing quartz is seen in the XRD pattern for the ash at 2Θ = 20º, 26º, 47º, 55º. 2Θ = 23º XRD spectra of isolated silica nanoparticles, which is characteristic of amorphous solid, confirms of amorphous silica formation, other researchers obtained similar results.

The other X-rays diffract grams showed semi crystalline and crystalline phase of silica for combustion at varying temperatures are 400ºC, 500ºC, 600ºC and 700ºC shows that amorphous substances display an atomic arrangement that is either random (or) has a very short range in order. The existence of several sharp peaks indicates the presence of crystalline phase in the matrix. Crystallization had taken place due to the high temperature employed during calcinations. The intensity of these silica peaks can increase with higher burning temperatures, which leads to a more crystalline structure. According to results, maximum efficiency was observed at 800ºC. In fig (e) shows, the SiO_2 structure of the amorphous silica present in the gentle slope the 2Θ angle 19.58º are recorded, in addition to 28.11º and 28.56º, indicating that the amorphous SiO_2 is generally amorphous and crystallized at 23º. In addition, some small peaks appear indicating the presence of weak traces of sodium [41].

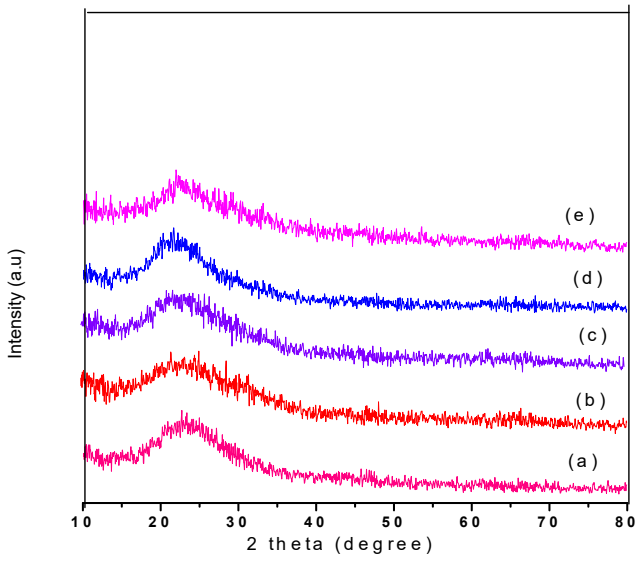


Fig.1. XRD Spectrum CHA a) 400ºC b) 500ºC c) 600ºC d) 700ºC e) CHS

4.2. Phase Analysis of Bio Silica from PSA

The XRD pattern for phase analyses was recorded in the 2Θ range 10–80º.The X-ray spectra and characteristic absorption peak of Peanut shell silica have been acquired at varying temperatures 400^0C, 600^0C,800^0C and displayed in Fig.2 (a - e). The sample is amorphous as pointed out by the featureless diffractograms and the appearance of a scatter maximum at $2\theta = 23^0$ characteristic of amorphous silica with hexagonal system according to the JCPDS 79-1915 [44].The structure of silica present in the selected as optimum temperature for bio silica synthesis.

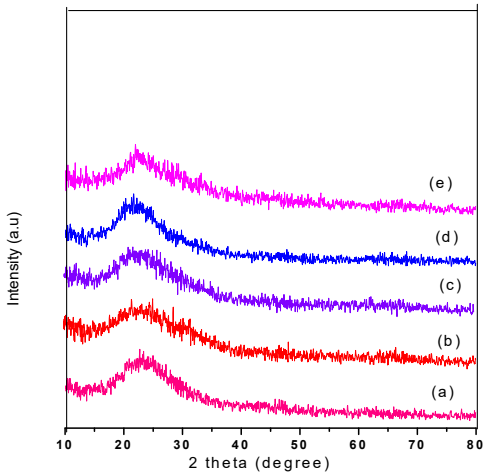


Fig.2. XRD Spectrum PSA a) 400ºC b) 500ºC c) 600ºC d) 700ºC e) PSS

This pattern exhibits a very broad line and no defined peaks due to crystalline are encountered. Figure 2 (a) also describes the theoretical postures of the main reflexion of the phase cristobalite (SiO_2) and graphite (C) and no peaks are discovered in these postures.

4.3 Spectral Analysis of Bio Silica from CHA

Fig.3 (a - c) shows the FTIR spectrum of CHA. A broad band in the range of 3375 cm^{-1} is due to the stretching vibration of the O – H band. This band is due to silanol groups (Si – OH) and the adsorbed water bound to the surface. The weak band 1627 cm^{-1} is assigned to H – O – H bending vibration mode. The predominant peak at 1350 cm^{-1} is due to silaxone bonds (Si – O – Si). The adsorption band at 893 cm^{-1} is because of silica structures and other peaks observed at 1627 cm^{-1} are because of impurities such as carbonate and sodium groups. The most intense bands at 1100 cm^{-1} and a peak 762 cm^{-1} are due to asymmetric and symmetric stretching mode at Si – O – Si. The vibration of oxygen atoms joined with the adjacent atoms in the asymmetric stretching vibrations of Si – OH bond which appeared at 1100 cm^{-1} [35].

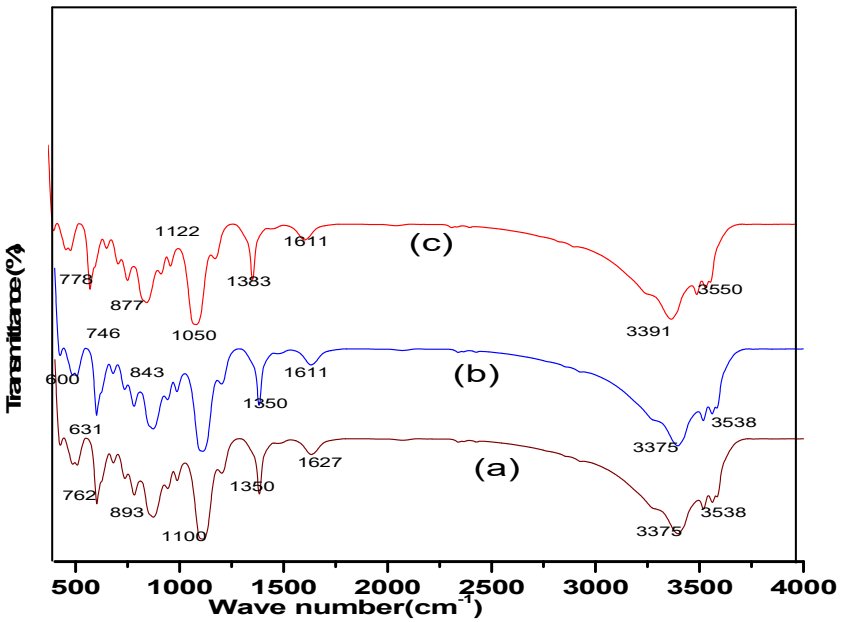


Fig.3. FTIR Spectrum CHA a) 600ºC b) 700ºC c) CHS

4.4 Spectral Analysis of Bio Silica from PSA

In Figure 4 (a -c) shows an FTIR spectrum of PSA. FTIR spectrum also exhibits bands at 600 cm^{-1}, 900 $cm^{-1}$and 1050 cm^{-1}characteristic of symmetric and anti symmetric stretching of Si – O bonds [42].The peak 1200 and 700 $cm^{-1}$are attributed to the vibration modes of the gel network [33]. The presence of absorption peak at 1611 and 1122 cm^{-1}suggest that the sample is still dominated $Si(OH)_4$. A slightly broad peak around 877 cm^{-1}shows the presence of silanol group (Si-OH) bending mode which associated with silicon atom linked with hydroxyl group in the framework [43].

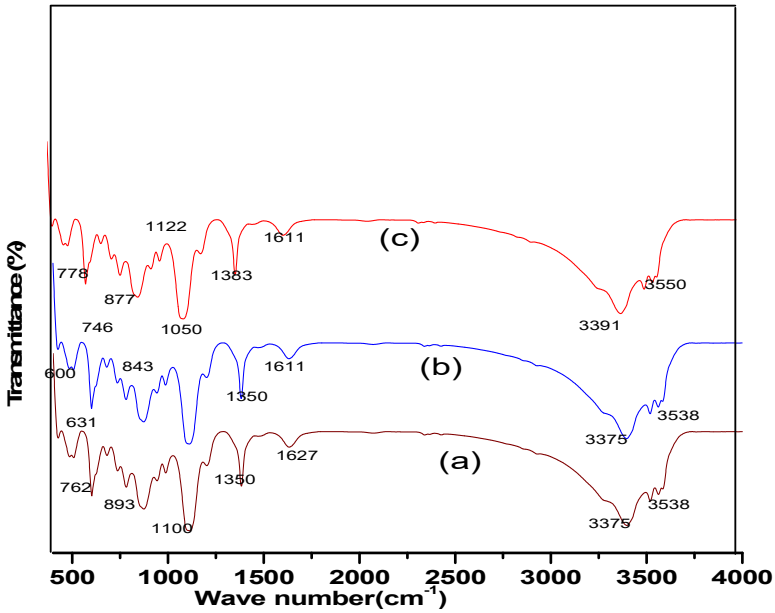


Fig.4. FTIR Spectrum PSA a) 600ºC b) 700ºC c) PSS

4.5 Morphological Analysis of Bio Silica from CHA

Fig .5 (a & b) exhibits the SEM and EDX analysis of Coconut husk silica. In SEM image, CHS particles are polygonal shape with different sizes. A strong intensity of Si in the EDS spectrum is confirmed the silica at higher weight percentage [43].

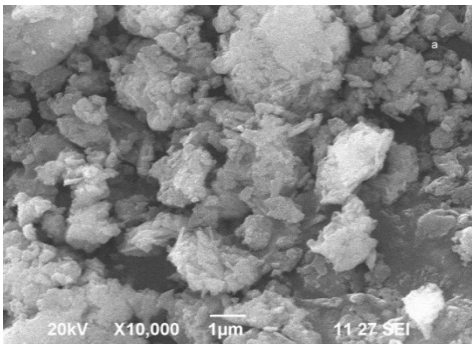


Fig.5 (a) SEM image of CHS

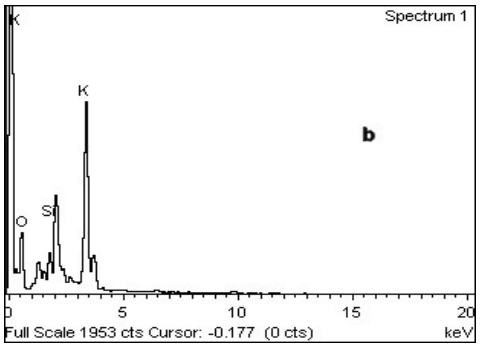


Fig.5 (b) EDX spectra of CHS

4.6 Morphological Analysis of Bio Silica from PSA

The SEM image and EDX spectra of peanut shell silica are shown in figure 6 (c & d). It is seen that the PSS has irregular shape that are loosely packed structure [40]. An EDX spectrum as shown in fig 6 (d) reveals a strong intensity of Si alone in the spectrum which confirms predominant of silica in the sample and some traces of impurities [39].

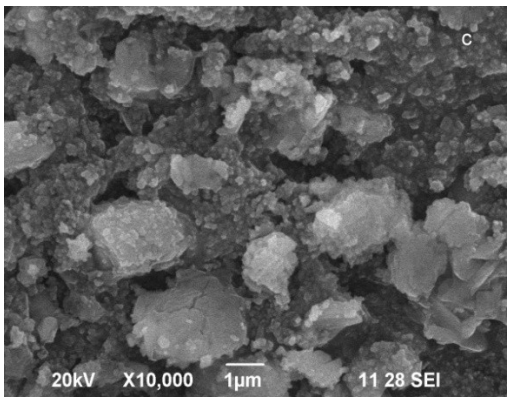


Fig.3.6 (a) SEM image of PSS

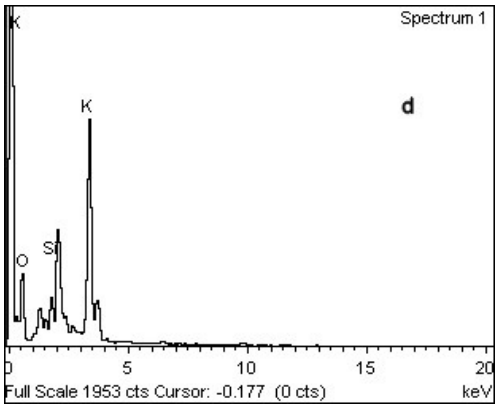


Fig.3.6 (b) EDX spectra of PSS

4.7. Thermal Analysis of Coconut Husk Ash

The results of TG/DTA analysis considered for an estimate of the temperature range that corresponds to important chemical and structural transitions of the obtained sample. The TG analysis revealed three distinct stages of mass loss namely, removal of moisture content, release of volatile matter and burning of combustion material.

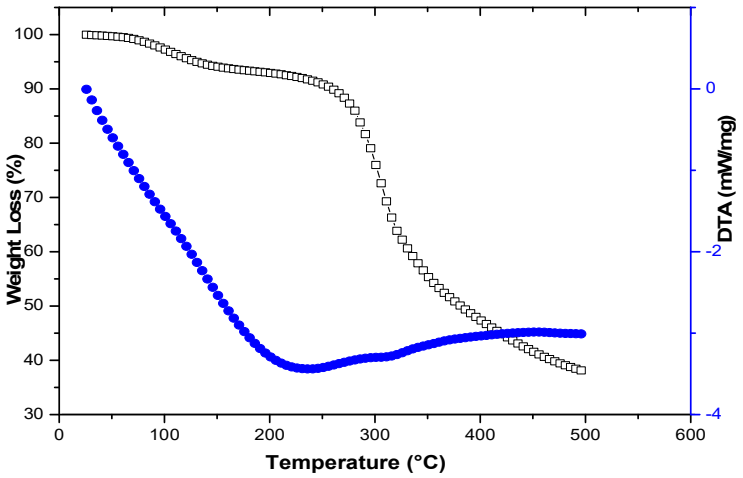


Fig. 7 TG-DTA Curve for Coconut Husk Ash

In fig. 7, the overall decomposition behavior of CHA was attributed to decomposition of hemi cellulose, cellulose and lignin. Previous reports claim that contributors to the evolution of the volatile matter. In the study on the thermal degradation, it can be reduced from these removals that decomposition of hemicellulose starts first followed by cellulose and finalizes with decomposition of lignin. The gaseous volatiles were released from organic and inorganic material during thermal degradation [38]. In this figure, it was noted that there was a decrease in the temperature of the sample around 200^0C. The weight of the sample remains almost constant for certain duration of time with decreasing temperature and after certain duration there is again increase in the temperature. This behavior of coconut husk ash sample of very fine size must be connected with some structural changes, since whenever there are any structural changes associated with sample, a certain amount of energy has been adsorbed and there is an endothermic reaction. The weight trace suddenly acquired along the abscissa and then continued its forward movement as usual. The TGA were mainly exhibits the initial weight around 100^0C which is due to loss of water content of the sample. The sharp decrease in weight over a temperature range of 150^0C can be attributed to the thermal combustion of organic groups present in the ash and rapid decomposition of the sample.

Coconut Husk ⟶ Intermediate + Volatiles ---------- (1)

In DTA curve, there are two exothermic peaks at 250^0C and 450^0C which are correlated to the removal of organic groups by oxidation and rapid decomposition of the sample [37].

4.8 Thermal Analysis of Peanut Shell Ash

Thermo gravimetric analysis shows that the activation process can be divided into three stages. This indicates that organic matter decomposes into intermediate of smaller molar mass in the initial stage of the activation reaction. The reaction releases the gaseous volatiles. Thus, the reaction mechanism at the first stage can be expressed as equation (1), at the second stage, the intermediate decomposes to form other volatile species, char and tar [36].

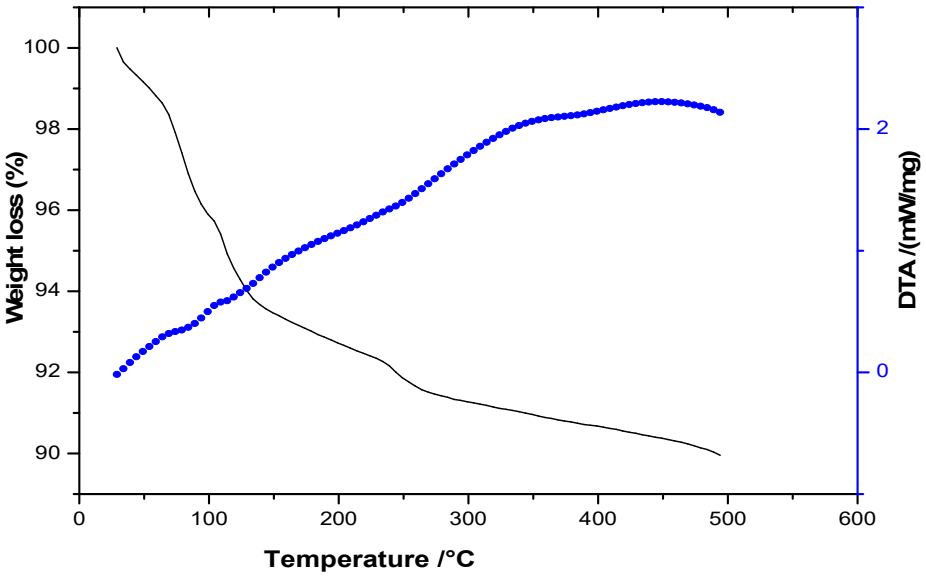


Fig. 8 TG-DTA Curve for Peanut Shell Ash

Intermediate ⟶ tar + char + volatiles _____ (2)

Fig.8 shows TG curve for Peanut shell Ash. The removal of moisture took place at temperatures ranging from 40 to 150^{0}C. The volatile matter released from husk in the range of 220- 325^{0}C and the last stage of mass loss that corresponds to the combustion process

Conclusion

The present study highlights the comprehensive characterization of peanut & coconut ash through physicochemical, thermo gravimetric & morphological analyses using XRD, FTIR & SEM/EDX techniques. The results confirm the presence of silica rich crystalline phases and reactivity. Thermo gravimetric analysis reveals good thermal stability, while SEM observations demonstrate porous and irregular surface morphology. EDX analysis further validates the elemental composition dominated by silica and other mineral constituents. Overall, both peanut and coconut ash exhibit significant potential as value added materials for applications in construction, catalysis and environmental remediation. This study reinforces the importance of utilizing agro waste as sustainable and eco friendly resources.

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