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# SYNTHESIS AND CHARACTERIZATION STUDIES OF SI–BASED REFRACTORY COMPOUNDS DERIVED FROM CORN COB

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**Abstract:** The synthesis and characterization of Si–based refractory compounds derived from corn cob was reported. Size reduction of the corn cob was achieved using a planetary mills. Carbothermal processing route was used for the production of the Si–based refractory compounds after acidic treatment. The carbothermal processing was done at temperature window of 600–900°C. The reaction products were analyzed using Fourier transmission infrared spectrometer (FTIR), scanning electron microscope (SEM) and X–ray diffractometer (XRD). The results obtained show that, at 600°C, there were different polytypes of silica phases in the refractory compounds. However, as the temperature increase up to 900°C, there were SiC phases in the reaction products. This trend was also corroborated with the results obtained from the XRD and FTIR respectively. The morphological features also agrees with the trend reported in the XRD.
**Keywords:** refractory compounds, structure, corn cob, silicon carbide

## INTRODUCTION

Silicon carbide is among the most active compounds utilized in the design of construction and hybrid ceramic materials for high operating temperatures due to a combination of properties including low density, high decomposition temperature, lack of phase transitions across a wide temperature range, superior mechanical qualities, non–interaction with corrosive environments, and the highest oxidation resistance among the refractory carbides [1–3]. It has the potential to be used as a reinforcement material for nanoscale components, protective coatings, and refractory matrices [3]. Currently, a wide variety of techniques have been described for the synthesis of SiC, however the majority of them were only performed in laboratories. Mechanical milling [4,5], rapid carbothermal synthesis [6–8], SHS procedures [9], microwave synthesis [10], polymer pyrolysis [11], sol–gel processes [12], CVD [13], and laser synthesis [14] are a few of these techniques. Each of the aforementioned procedures has benefits as well as drawbacks over the others, such as cheaper precursors used, lower reaction temperatures, higher product purity, etc [15].

One of the simplest and most cost–effective methods for producing silicon carbide–based refractory compounds out of all of these techniques is carbothermal processing, according to reports [2]. The traditional approach for producing SiC powders is exceedingly expensive and has substantial processing costs, which are quite rare, particularly in the developing Nation. Due of the relatively high cost of producing SiC, researchers have explored an alternative route for this production, hence, the use of agricultural wastes.

Wastes are items that have been rejected, disposed of, or left unmanaged because they can no longer be used and were produced without the aim of being used again [16]. Agricultural wastes were described by [17], as organic byproducts of plant life, including oil palm, corn, rice, and coconut shells, as well as seeds, fruits, leaves, and roots.

Agricultural wastes are defined by [18] as the leftovers from the cultivation and processing of raw agricultural products such fruits, vegetables, meat, poultry, dairy products, and crops. Animal waste, food processing waste, and agricultural waste all fall under the category of agriculture. Techniques for managing waste including waste reduction, reuse, and recycling must be properly taken into account. Because it will be challenging to implement goals to achieve zero waste from agricultural activities, efforts should be strengthened to reduce waste. Using eco–friendly strategies that employ the waste created for alternative activities without any form of treatment is also essential.

The transformation of agricultural waste into useable forms looks to be the most environmentally benign, economically viable, and sustainable approach to waste management.

Several studies have reported the use of different agro wastes materials in the production of Si–based refractory compounds [2,19,20] with potential use as reinforcement in metal matrix composite. Corn cob is reach in silica content among other important refractory compounds, thus making it a potential material for the production of Si–based refractory compounds. The leftover from corn includes a sizable amount of corn cob. According to authors [21], maize cobs make up between 40 and 50 percent of all corn produced. One example of an agricultural waste is corn cobs (a byproduct of sweet corn). Due to inadequate waste management in society, this byproduct leads to environmental pollution, which causes health issues among others. In an effort to reduce production costs due to the availability of raw materials, and less expensive methods; as well as to control waste in society, efforts have been made to transform this agricultural waste into useful materials that will be highly appealing to scientific and technological communities, especially in developing countries. The presence of refractory compounds in corn cob (CC) makes it a candidate material for the synthesis of a Si–based refractory compounds. For the larger–scale development of SiC, corn cob is viewed as a very promising material. The purpose of this work is to analyze the effect of carbothermal treatment temperatures on the production of silicon based refractory compounds from corn cob.

## MATERIALS AND METHODS

### Experimental materials

The experimental materials utilized were corn cobs which were gotten from the Landmark University Farm, Omu–Aran, in large quantities, distilled water for the washing of the corn cob, leaching agent (Hydrochloric acid) which were used to remove impurities from the cob, a sieve which is used to drain the acid from the corn cob when rinsing it with water and a set of nose masks, gloves to protect from the acid and beakers which were used to dissolve the solute in the solvent.

### Preparation of Agro wastes

To remove impurities that might prevent the solid creation of silicon–based refractory compounds from corn cob, experimental specimens were carefully prepared according to recognized techniques adopted by [2, 22, 23]. The corn cobs for sample preparation were thoroughly rinsed in clean water to remove every forms of dirt and volatile materials which has been accumulated during the period of time it has been on the farm and during transportation to the lab. These materials were sun–dried for (as shown in the Figure 1) three (3) days (7 h/day) to remove the moisture or water content present as a result of the initial washing. After, they were subjected to a temperature of 100°C for three (3) hours in an electric oven to eliminate the residual moisture. The corn cobs were later pulverized using a ball milling machine to increase the surface area of the corn cob and to reduce the size of the corn cob to powder form. The reduction in size and increase in surface area is necessary to enhance the reaction process.

Figure 1: Sun drying process of the corn cob

### Synthesizing of Corn cob samples

Carbothermal processing was adopted in the production route for the production of Si–based refractory compounds from corn cob. The process sequence used have been earlier reported by authors [2]. An analytical four digit weighing balance, CY224C, 0.1mg was used to weigh a known mass of corn cob.

A 400 ml of hydrochloric (as the leaching agent) was added to the weighed corn cob and a magnetic steering machine was used for stirring operation. After a while, 40 g of the dried samples were collected and placed in the graphite crucible which was then charged at a temperature ranging from 700°C – 900°C in an electrical furnace for an hour as earlier reported by [24].

The furnace was later turned off to cool before samples were removed from the crucibles. Table 1 shows the designation of the samples and heat treatment temperature.

Table 1: Sample designation and heat treatment temperature

| Sample designation | Carbothermal temperature (°C) |
|---|---|
| (A) | 700 |
| (A0) | 800 |
| A1) | 900 |

The dried samples were gathered, weighed, and examined for structural characterizations such as Fourier–Transform Infrared Spectroscope (FTIR) and X–Ray Diffraction to determine their suitability for the Si–based refractory compounds.

### Fourier transform infrared (FTIR) spectroscopy

The functional groups existing in the reaction products were identified using FTIR.

About 100–250 mg of kBr powder was mixed with 8–12 mg of refractory compounds from corn cob (RCC). The mixtures were compacted under 20–40 MPa compaction load. The functional groups in the reaction products were later accessed through this treatment sequence.

### X–ray diffractometer (XRD)

The phases present in the reaction products after the carbothermal processing of the RCC were scanned by XRD.

A backloading preparation method was used for the XRD scanning, the analysis was done using PANalytical Empyrean diffractometer with PIXcel detector and fixed slits with Fe filtered Co–Ka radiation in a scan range of 2theta ranging from 10° to 90° at 10° scan step. The phases present were identified using X'Pert Highscore plus software.

### Scanning electron microscope (SEM)

The morphological features of the reaction products were accessed using an extreme resolution Analytical Field Emission Scanning Electron Microscope– (JEOL, USA) equipped with energy dispersive spectroscope (EDX) operating with an accelerating voltage of 15 kV.

## RESULTS AND DISCUSSION

### Mass recovery/ reduction

The representative recovery yield at different treatment temperature are displayed in Figure 2. Initially, a mass of 40g was kept constant for each batch (A0, A, and A1 respectively). It is evident from Figure 2, that there was sufficient time for the transformation of Si–based refractory elements in the corn cob at higher temperature as compared to samples A0 and A.

The reduction in the yield of A1 further corroborate this observation. The implication is that, at higher treatment temperature, the volatile materials are given off during the carbothermal treatment, hence, a reduction in the mass of the Si–based refractory compounds. It can be inferred from Figure 2 that sample A displayed a value of 73.88% by mass recovery, A0 had 76.65% while A1 had 83.05%.

At higher temperature, the reaction sequence favoured the formation of SiC phases, thereby, enhancing the mass recovery rate. This trend is in line with the pattern obtained from the XRD and FTIR spectroscopy.

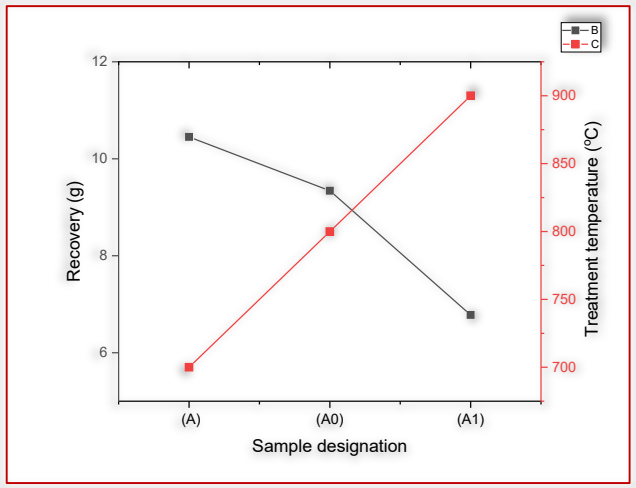


Figure 2. Variation in mass recovery rate with treatment temperature

### Fourier–Transform Infrared Spectroscope (FTIR)

Table 2 shows the pronounced peaks in the reaction products for the FTIR spectra of the RCC treated at 700°C, 800°C, and 900°C respectively. Also, previous work were benchmark with the current study, the trend of the results are as presented in Table 2.

Table 2: benchmarking the variations in the wavelength associated vibrations with previous studies and current work

| Wavelength ($cm^{-1}$) | Associated vibration | References |
|---|---|---|
| 3670 – 3000<br>1093<br>828 | O–H stretching<br>Si–O–Si<br>Si–C | [25] |
| 1091.75 – 3500 | O–Si–O stretching and bending | [26] |
| 821.92<br>1629.66 | Si–C bond<br>H–OH bending vibration | [26] |
| 3472<br>1740<br>800–679 | O–H stretching<br>O–H bending<br>Si–C stretching | Current study |

From Figure 3, the legend A, Ao, and A1 are the IR spectra for the corn cob ranging from the carbothermal heat treatment at 700°C, 800°C and 900°C respectively. It is evident from the carbothermal heat treatment at 700°C, the presence of O–H functional group and a stretching band was observed at a broad peak of wavelength 3390 $cm^{-1}$. Similarly, at a wavelength of 2450 $cm^{-1}$, N–O stretching at a low peak was observed.

The presence of C=C functional group as a bending band is located at about 930 $cm^{-1}$ wavelength value. A functional group of O–Si–O was observed at a wavelength of 1490 $cm^{-1}$. It is evident that Si–C presence was noted at about 679 $cm^{-1}$ of wavelength value in the stretching band.

At temperature of 800°C, it was identified that O–H functional group in stretching band was

present at wavelength of 3390 $cm^{-1}$. The presence of O–H functional group is as a result of dehydration during the process of treatment. At wavelength value of 1530 $cm^{-1}$ shows the presence of N–O functional group in the asymmetric stretching band. At a broad peak of 1100 $cm^{-1}$, C–C bending stretching band was indicated. Si–C shows its presence in the stretching band at wavelength value of about 610 $cm^{-1}$.

At a temperature of 900°C, a broad peak in the stretching band which indicates the presence of O–H functional group with a wavelength of 3390 $cm^{-1}$ was noticed in the spectra. It is noted that C=O functional group in the stretching band having a wavelength of 1800 $cm^{-1}$ was observed in the spectra. It is worthy of note that, N–O functional group in the stretching band occurred at a wavelength of 1530 $cm^{-1}$. A low broad peak of wavelength 1100 $cm^{-1}$ indicated the presence of C–C functional group in the stretching band. However, the presence of Si–C functional group is indicated with a sharp peak in the stretching band at a wavelength value of 850 $cm^{-1}$, this was within the margin earlier reported by authors [26,27] in the formation of SiC.

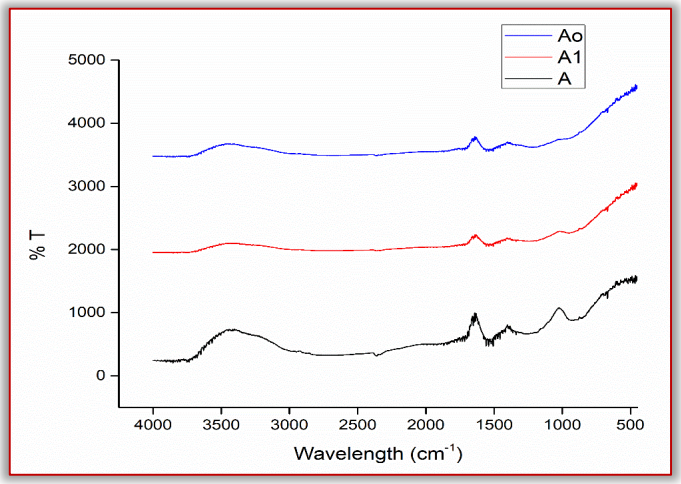


Figure 3. FTIR Spectra for Corn Cob at 900 °C

## X–ray Diffraction

The XRD of the RCC are as displayed in Figure 4. Sample A and Ao showed a similar trend in peak values, hence, the legend displayed in Figure 4 was used to capture Ao. It was observed at a temperature of 700°C that at a 2Theta range of 20°–30°, $SiO_2$ was formed with a peak value of percentage transmittance of 2000 and 4000 respectively.

At a 2Theta range of 30–40°, the presence of SiC phase (showing a low peak) and $SiO_2$ were observed at a peak value of transmittance of 9000 which is the most pronounced peak in these spectra and 5000 respectively. It was also observed that at 2Theta range of 55–58°, there was a formation of SiC at a lower peak value. The formation of SiC was low in this spectra due to low treatment temperature that the corn cob was subjected to.

However, at a temperature of 900°C which is a higher treatment temperature compare to the former, the formation of SiC was observed to be more in this spectra. Also, $SiO_2$ was observed at a 2Theta range of 20–30° at a pronounced peak with a value of percentage transmittance of 18000. The temperature was sufficient for the phase transformation reaction of $SiO_2$ and C to yield SiC.

At 2Theta range of 30–80° the formation of SiC was observed in this spectra at peak values of percentage transmittance of 10000, 4000, 6000, 2500, and 5200 respectively. The formation of more SiC in this spectra is as a result of higher treatment temperature of 900°C required for transformation of $SiO_2$ and C to SiC. However, at relatively lower treatment temperature of 700°C, it was noted that some unreacted phase of $SiO_2$ polytypes precipitate in the reaction products.

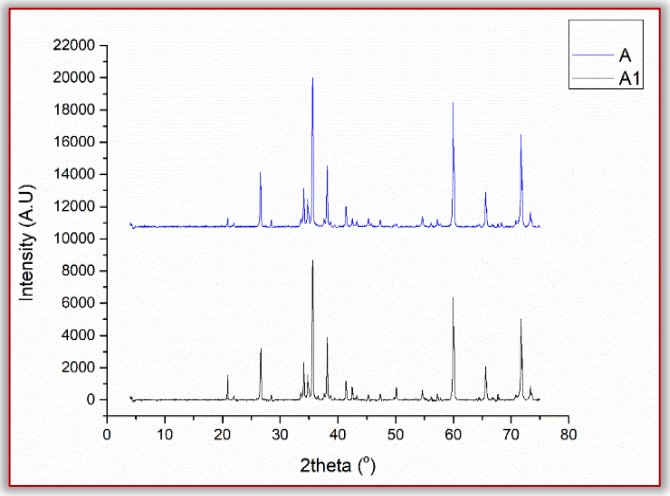


Figure 4. XRD Pattern for Corn Cob at 700 °C and 900 °C

## Morphology assessment

The variation in the morphological features of the RCC at varying temperatures are as shown in Figure 5(a) and (b). Evidently, Figure 5a shows the representative scanning electron image (SEM) with EDX of an amorphous phase of the RCC. It is evident from the EDX profile the presence of Silicon (Si), Iron (Fe), Oxygen (O), and Carbon (C) all establishing the presence of SiC (low peak), $SiO_2$ and $Fe_2O_3$. It is noted that the presence of Chromium (Cr) and Potassium (K) indicate impurities elements in the RCC. This trend is similar to the findings reported by [28].

The heterogenous nature of the RCC is well captures in the morphological features displayed in Figure 5b. Evidently, the peak in the spectra

show a high value of Silicon (Si), Oxygen (O), and Carbon (C). It is noted that the silica phase reacted with the carbon constituent in the structure thereby yielding SiC phase. This trend was also consistent with the results obtained in the XRD spectra.

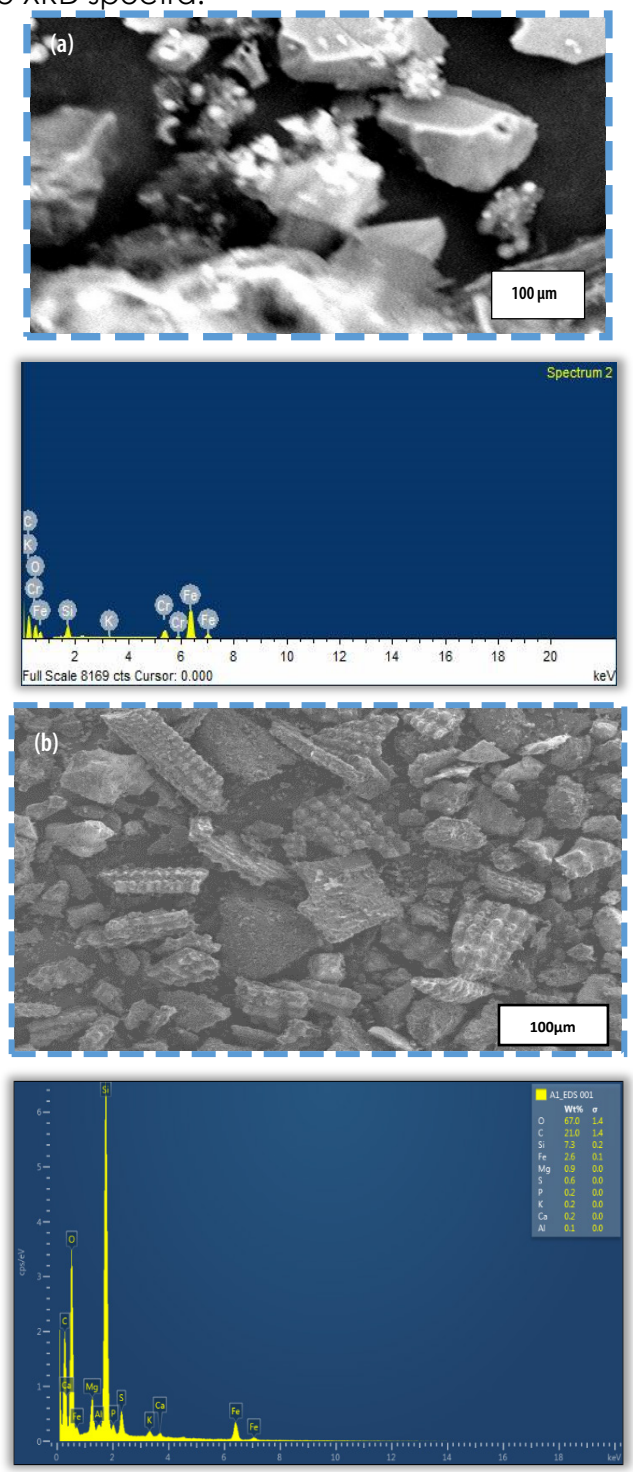


Figure 5: Showing the morphology of RCC (a) at 700 °C (b) 900 °C

## CONCLUSIONS

The study reports on the synthesis and characterization studies of Si–Based Refractory Compounds derived from Corn Cob. From the results obtained, higher temperature treatment favours the formation of SiC phase. The heterogenous nature of the RCC was evident in the morphology. The wave number of the FTIR spectra for the formation of Si–C was at par with previous reports in the literature.

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