



## Research article

## Investigation of the effects of selected bio-based carburising agents on mechanical and microstructural characteristics of gray cast iron

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## ABSTRACT

The mechanism of graphite formation on gray cast iron metal during carburisation process using organic nano-carbon (ONC) was investigated at 900 °C for a holding time of three (3) hours. TEM and XRD were employed to characterize the pulverised nano-carbon to determine their phases and bonding potentials. Also, SEM/EDS, XRD and Vickers' hardness tester were employed to determine the microstructure, phase compositions as well as hardness and wear properties of the carburised material. The microstructural result showed that, there was uniform carbon diffusion into the substrate material which led to layers of graphite formation and subsequent surface modifications for each of the selected nano-carbon used. Also, the XRD results revealed variations in the peak patterns for each of the substrate carburised with different organic carbon with substrates showing graphite and iron phases as observed in that carburised in pulverised palm kernel shell having broad peaks at 35.50°, 44.4°, 65.12° and 82.395°. This is traceable to amorphous properties and crystalline behaviour of the organic carbon. Further to this, the micro-hardness measurement showed that substrate carburised using pulverised palm kernel shell performed better compared to other substrates in other media with a micro hardness value of 355.8 (HV) against as-received which is 116.9 (HV). Thus, this is a novel and possible method of improving the properties of grey cast iron to meet the increasing demand in gear applications.

## 1. Introduction

The performance of heat treated alloy steel materials when subjected to different applications or test has been known to be associated with the improved mechanical properties [1]. However, the criticality of the wear behaviour of cast iron when subjected to industrial applications also necessitated the consideration for their improvement against incessant failures [2, 3, 4]. Organic carbon is receiving great attention which is attributed to their ability to improve hardness and strength of cast iron and steel material for better wear characteristics [5]. Technological advancement in engineering applications have necessitated material modifications for adequate design and reliability. Organic carbon such as pulverised palm kernel shell, coconut shell, wood charcoal and egg shell have been found to have improved the strength and wear resistance of low carbon steel due to the different proportion of carbon content present in them [6, 7, 8]. According to Ntenga et al [9], Palm kernel shell (PKS)

and coconut shell (CCS) are mainly amorphous and exhibit crystalline phases in their microstructure. Further processing of these organic carbon could suggest them useful to improve the mechanical properties of low carbon materials. This may be due to the presence of alkali metals, and the reactivity and porosity associated with the microstructure [10, 11]. It has been established by Kurosaki *et al.* [12] that the microstructure of wood charcoal is characterised by graphene layers which are arranged in irregular manner. However, the use of activated charcoal as additives have proven better in improving the modulus of rupture and strength of wood composite which implies that better result would be achieved in terms of metal heat treatment [13, 14, 15]. In addition, pulverised egg shell has been found to have excellent grain refinement when combined with the aforementioned organic carbon due to their excellent adsorbent property, thus giving better yield point to some alloys especially aluminium [16, 17]. Gray cast iron is mostly used in different engineering applications because of its weight, damping ability and lower

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cost. Heat treatment of gray cast iron is usually done to relieve stress due to casting and also to ease the machining process [19]. However, gray cast iron is mostly associated with delamination and adhesion when subjected to real life applications [20]. This can be minimized by altering the chemical composition through addition of some elements which can improve the graphite formation and eventual hard surface appearance [21]. Thus, the presence of graphite layers in the microstructures of gray cast iron can ease the machining process as compared to steel [22]. According to Giacomelli *et al.* [23], gray cast iron has excellent mechanical strength and low cost of production, hence, its wide industrial applications. However, the presence of the graphite flakes in the microstructure causes defects which contribute to incessant fatigue failure of components during application. Thus, one of the ways to minimise this, is to increase the carbon content as a means of heat treatment. This in essence, helps in modifying the morphology of the cast iron, thereby increasing its mechanical strength [24, 25]. The study therefore, aimed to modify the morphological characteristics of gray cast iron in order to improve its mechanical and microstructural properties. It also contributed to waste valorisation processes by employing bio-based carbon from agricultural wastes to enhance the carbon content of gray cast iron through carburisation. Three selected biomass materials of palm kernel, coconut and wood were used with egg shell as the energiser.

## 2. Experimental

### 2.1. Materials preparation

Gray cast iron of chemical composition 2.68 wt% C, 1.42 wt% Si, 0.63 wt% Mn, 0.13 wt% S, 0.28 wt% P and iron by balance was obtained. The material was sectioned into a rectangular shape with dimensions 30 mm × 20 mm × 10 mm. Each material was prepared using grades of silicon carbide abrasives to obtain polished and smooth surface for easy diffusion of carbon into the metal surface. Also, Pulverised palm-kernel shell, coconut shell, wood charcoal and egg shell were obtained and sieved to a particle size of 75 µm to obtain finer size.

### 2.2. Carburisation process

Carburisation of the gray cast iron was carried out using three (3) organic carburisers which include pulverised palm-kernel shell, coconut shell and wood charcoal and addition of egg shell as a catalyst. Each medium was mixed with pulverised egg shell in the ratio of 70–30 wt %. Muffle furnace with operating temperature 1200 °C was employed for the experiment. Each pulverised mixture was packed inside a stainless steel carburising pack to accelerate carbon absorption by the substrate material (gray cast iron) embedded into the carbon mixtures and the carburising pack was closed. Furthermore, the closed stainless carburising packs were charged into the muffle furnace and monitored at a temperature of 900 °C over a holding time of three (3) hours to be able to investigate the microstructure and the hardness properties of the heat treated material. The materials were unloaded at the end of the holding time and were allowed to cool slowly before quenching in distilled water (Figures 1 and 2).

### 2.3. Material characterisation

The chemical compositions of the as-received gray cast iron was determined using spark emission spectrometer. The pulverised organic carbon was characterised using transmission electron microscope (TEM) at 200 nm to study the morphology and the crystalline structure. More so, the phase compositions of each organic carbon was studied using X-ray diffractometer (XRD). A Tescan Vega 3LMH scanning electron microscope with an accelerating voltage of 20kV, beam intensity of 17 W/m<sup>2</sup> and the scanning speed of 10µs/pixel was used to check the morphology of each carburised sample. Also, the phase composition of the carburised substrate (gray cast iron) were equally determined using Rigaku Miniflex 600 powder diffractometer, which is equipped with Cu ka radiation source generated at 18 kW and 250 mA. The XRD spectra were obtained at room temperature.

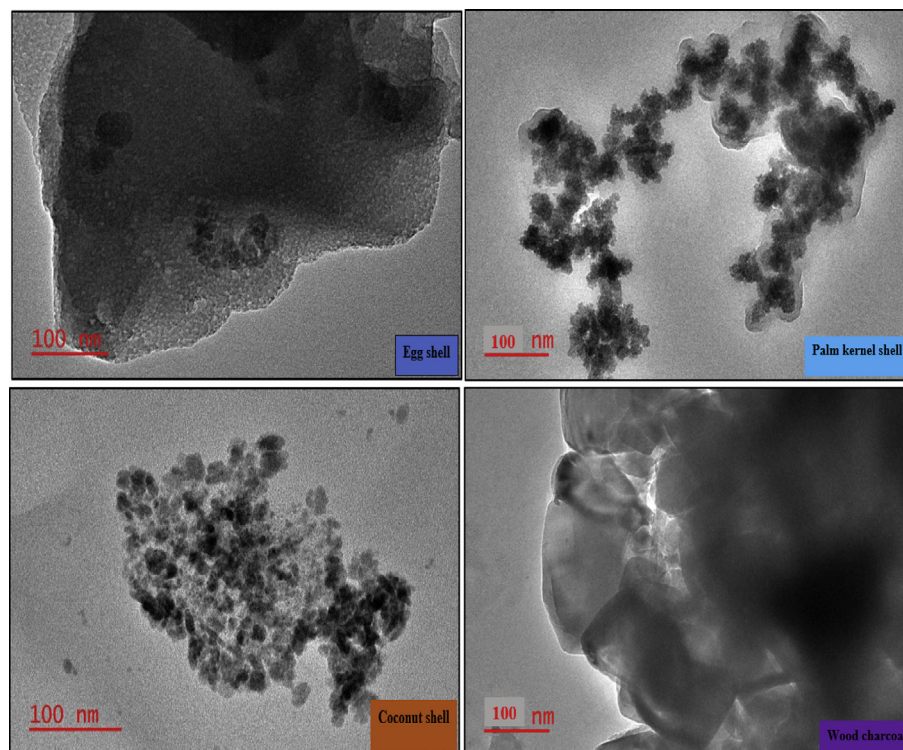


Figure 1. TEM microstructures of the pulverised organic carbon.

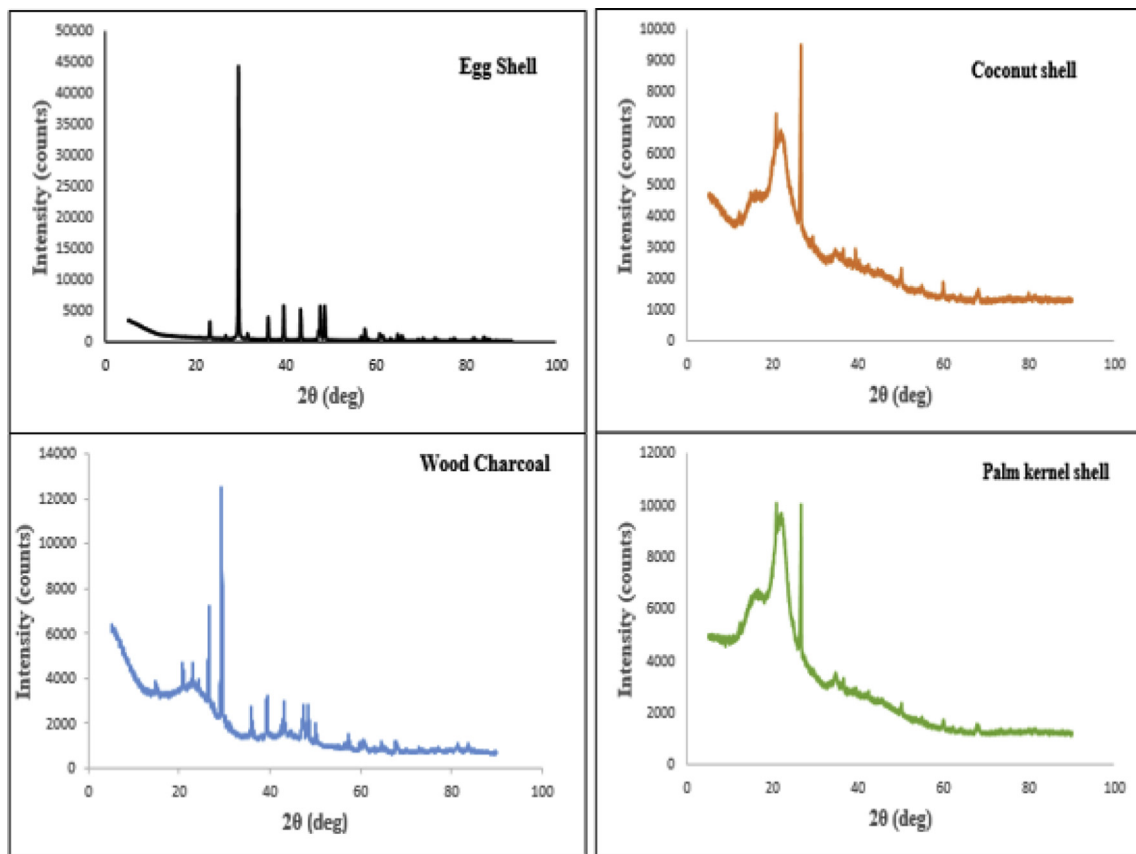


Figure 2. XRD profile of pulverised organic carbon.

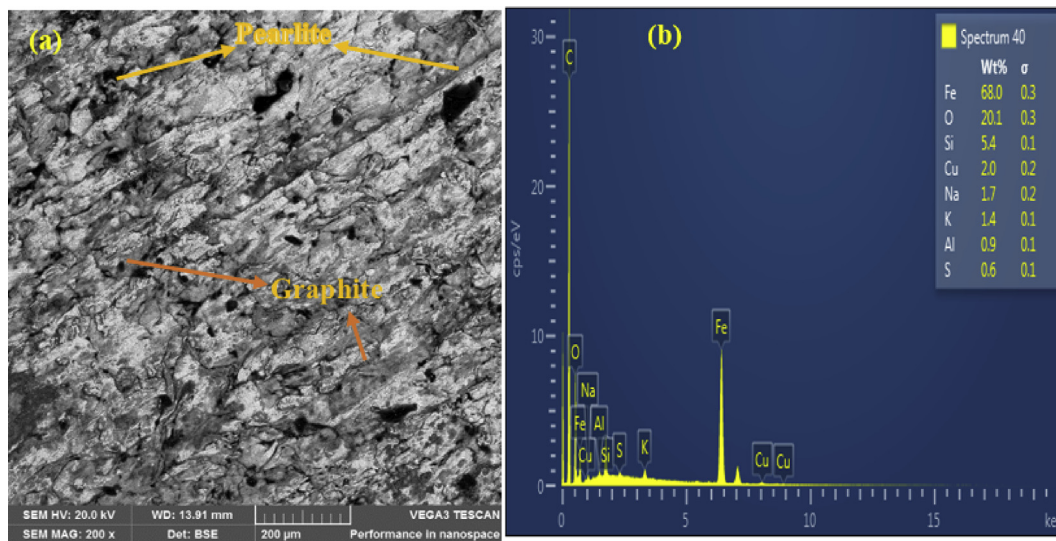


Figure 3. a: SEM Morphology of As-received gray cast iron, b: EDS Morphology of As-received gray cast iron.

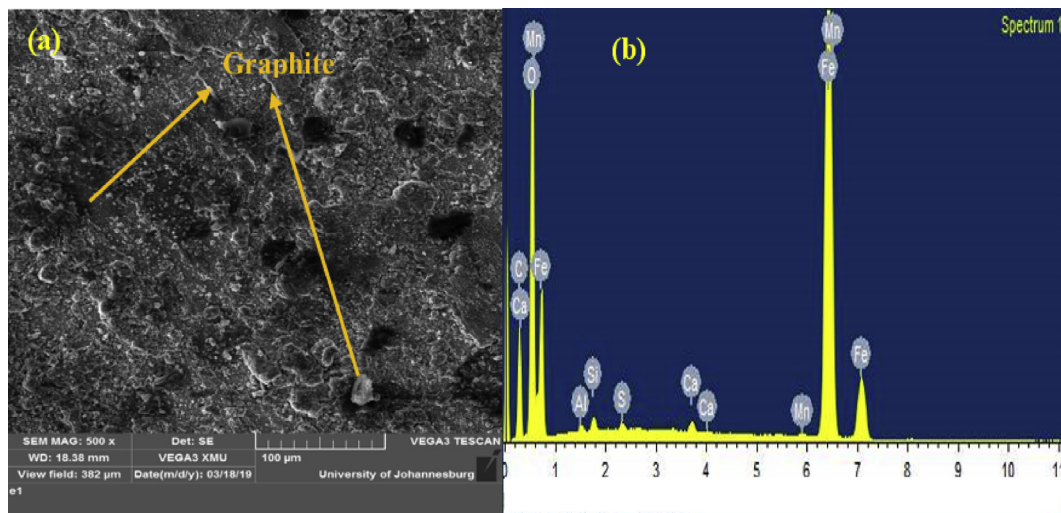
#### 2.4. Micro hardness measurement

The micro hardness tests for each sample carburised in different media were measured using a Vickers hardness test equipment with diamond indenter which has a triangular pyramid shape. Micro hardness tests were carried out with a load of 500 N which remain constant throughout the experiment, while the dwell time used was 15 s.

### 3. Results and discussion

#### 3.1. Microstructure and phase composition of pulverised organic carbon

The transmission electron microscopy (TEM) of each organic carburiser was carried out and presented in Figure 1. Figure 1, showed the topography, morphology and the crystalline structure of each carburising media. The microstructures revealed regions of light and dark zones



**Figure 4.** a: SEM/EDS Morphology of gray cast iron sample in Palm-kernel shell Carburised at 900 °C for 3hours, b: EDS Morphology of gray cast iron sample in Palm-kernel shell Carburised at 900 °C for 3hours.

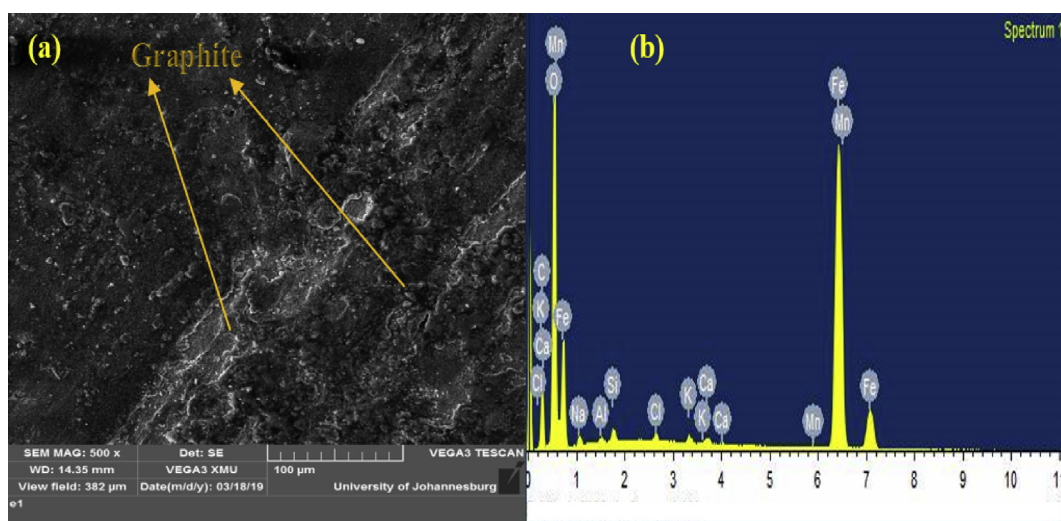
which is an indication of the penetrating behaviour of electrons into the pulverised samples. Invariably, the dark regions revealed the dense areas of object. This variation suggests the structure, sizes, textures and shape of the pulverised samples. Furthermore, observations from the microstructures showed that, each of the pulverised organic carbon sample formed an agglomerates of fine particles which accelerated their compaction and binding abilities on the substrate material (gray cast iron). Also, these properties gave insight on the formation mechanisms of graphite as observed in the various SEM samples.

Also, Figure 2. Showed the XRD profiles of the carburising media. The XRD of egg shell indicate a major diffraction at 30° and other variations of peak at 38°, 40°, 42° and 45° respectively. The peak patterns indicate the crystalline nature of the organic carbon. More so, the similarities in the lower peaks suggest its high bonding potentials which agrees with the study of Dagwal *et al.* [26]. However, the XRD of pulverised coconut and palm kernel shell showed similarities in their diffraction patterns but with slight variation at the peak levels. The highest peak level of coconut shell was observed at 25°, while palm kernel shell had its broad peak at 26°. This indicate that they exhibit crystalline properties that can improve the hardness of the substrate (gray cast iron) Okoroigwe *et al.* [27]. Also, the XRD of pulverised wood charcoal indicates the crystal growth of the organic carbon as observed from the peak patterns. Broader peak was

observed at 30° while subsequent peaks were observed at 28°, 40°, 42° and 48° respectively. This also showed that it contains some crystalline materials that improved the binding forces in the organic carbon.

### 3.2. SEM/EDS morphology of carburised gray cast iron

Figure 3 showed the morphology of as received gray cast iron while Figure 4 revealed the morphology of gray cast iron carburised at 900 °C for three (3) hours in pulverised palm kernel shell. From the microstructure, after carburisation, the pulverised palm kernel shell and the substrate material (gray cast iron) were found to have bonded together metallurgically and the resulting surface was characterised by graphite layers. The carburisation process revealed that carbon atoms diffused into the substrate material and this improved the surface morphology. However, the EDS analysis of the sample showed the major element present which include; Fe, Mn, Si, Al and C. Mn and iron content formed the highest element on the surface. Mn presence indicate that there was pearlite formation at the interface due to the cooling of austenite, thus, Mn presence will improve the wear resistance of the carburised material according to El Sawy *et al.* [28]. Gamma-phase ( $\gamma$ -Fe) were formed which can improve the hardness and strength of the material. Further to this, Figure 5 showed the microstructure and EDS profile of gray cast iron



**Figure 5.** a: SEM Morphology of gray cast iron sample in Coconut shell medium Carburised at 900 °C for 3hours, b: EDS Morphology of gray cast iron sample in Coconut shell medium Carburised at 900 °C for 3hours.

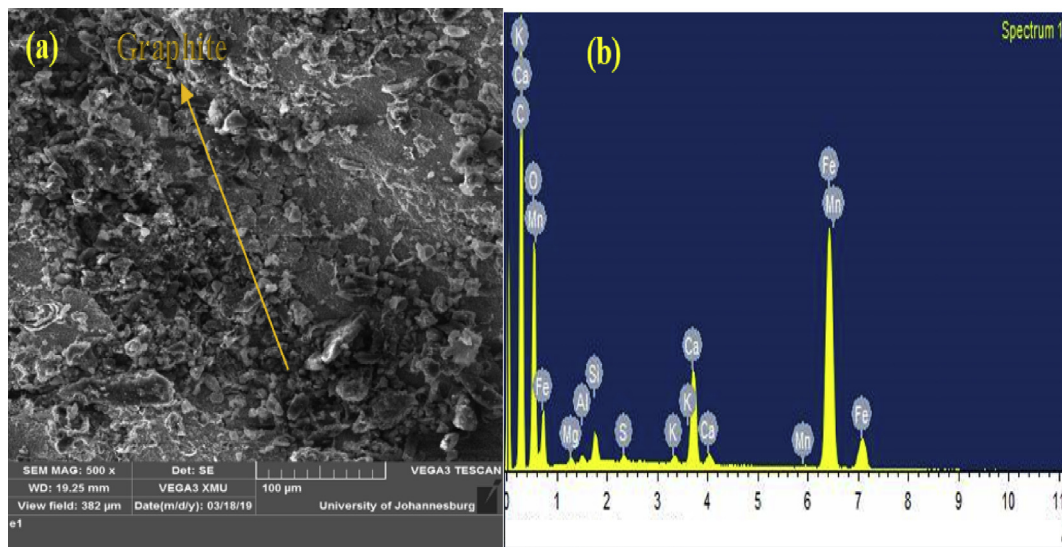


Figure 6. a: SEM Morphology of gray cast iron sample in wood charcoal Carburised at 900 °C for 3hours, b: EDS Morphology of gray cast iron sample in wood charcoal Carburised at 900 °C for 3hours.

carburised in a mixture of pulverised coconut shell at 900 °C for three (3) hours. The microstructure revealed a porous surface structure which is associated with the porosity of coconut shell and its high carbon presence. Thus, the coconut shell enhanced the strength and hardness property of the gray cast iron which is in agreement with the study of Raju *et al.* [29]. The EDS result revealed certain elements which were dispersed into the material during the heat treatment process. These include; Mn, K, Na, Al and P. Mn increases tensile strength and hardness, and also decreases the critical cooling rate during the process of hardening thereby improving the hardenability of the gray cast iron. Similarly, the presence of Phosphorus increases strength and hardness and also increased the resistance to corrosion while improving machinability. More so, Figure 6 revealed the microstructure and EDS result of gray cast iron carburised in a mixture of pulverised wood charcoal and egg shell. It was observed from the SEM morphology that wood charcoal showed a non-graphitization property which is linked to porous nature of the medium. The fine structure indicates the random behaviour of the carbon

atoms which also indicate its choice as a potential organic carbon for carburisation. The EDS profile showed that several elements were present in various percentages which are; Fe, Mn, Mg, S, K, Ca, Cr, Cl, P and Cu. The presence of Cr increases corrosion resistance as a result of oxidation process, hardenability and high temperature strength. Its ability to increase high temperature strength gives it advantage during the application as spur gear in mesh. Also, the presence of sulphur in small amounts improves machinability and weldability. The presence of Mg also improves toughness, and resistance to hydrogen induced cracking (which can come from the impacts of ethanol medium), while Ca addition to low carbon steel can improve the resistance to hot cracking, and weld-related cracking.

### 3.3. Phase composition of carburised gray cast iron

To further measure the rate of dispersion of carbon into the substrate (gray cast iron), X-ray dispersive spectroscopy was employed to measure

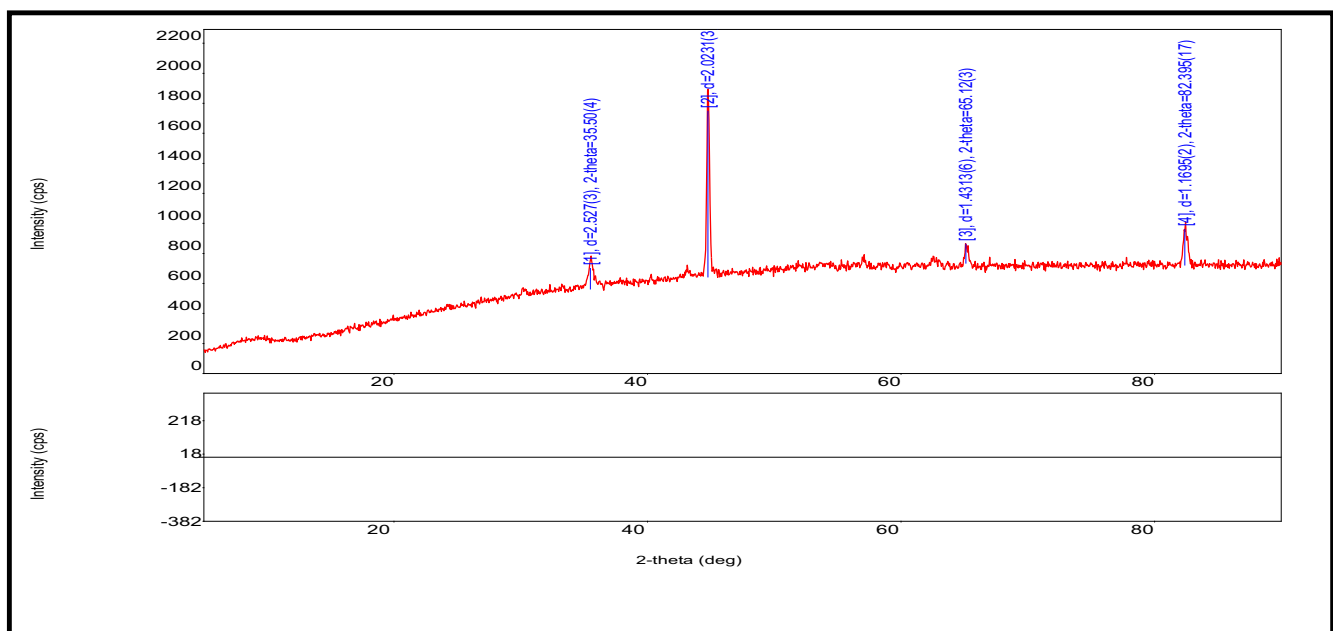


Figure 7. XRD profile of sample in Palm-kernel shell powder medium Carburised at 900 °C for 3hours.

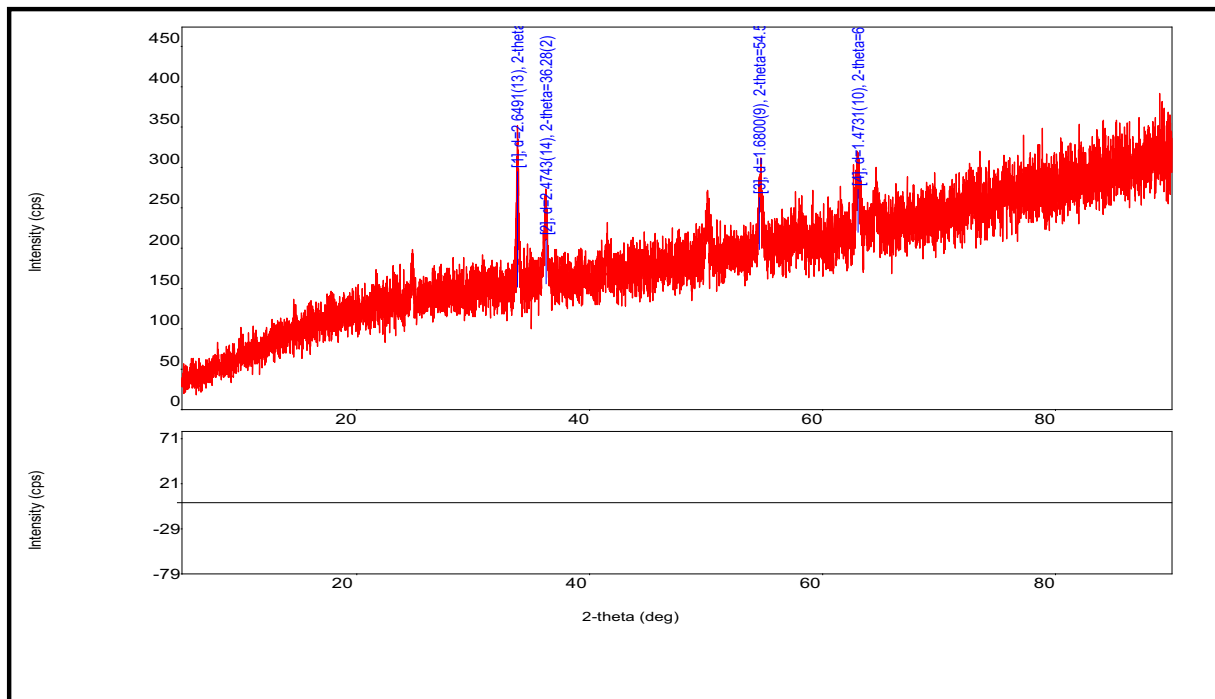


Figure 8. XRD profile of sample in Coconut shell medium Carburised at 900 °C for 3hours.

the distribution and peak levels of element dispersed. Figures 7, 8, and 9 showed the various pattern of distribution of carbon of the various pulverised organic carbon. From Figure 7, it could be observed that broad peaks dominated the surface ranging from 35.50°, 44.4°, 65.12° and 82.395° with martensitic microstructure as observed in the SEM image (Figure 4). The diffraction patterns are broad and indicates that there was even diffusion of carbon and other micro crystalline material present in pulverised palm kernel shell into the substrate. This suggests the

spherical shape of graphite as observed in the SEM image (Figure 4). The high percentage of carbon present suggests its use in the improvement of micro hardness. Also, Figure 8 showed the XRD profile of gray cast iron carburised in a mixture of coconut shell and egg shell. Peak levels were observed 36.28 °C, 54.3 °C and 62.4 °C respectively with mainly Fe phases and hematite iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>). These properties reinforce the carburised material against abrasive wear which is in line with the studies of Karunakara and Dinesh [30] and Phanibhushana et al. [31].

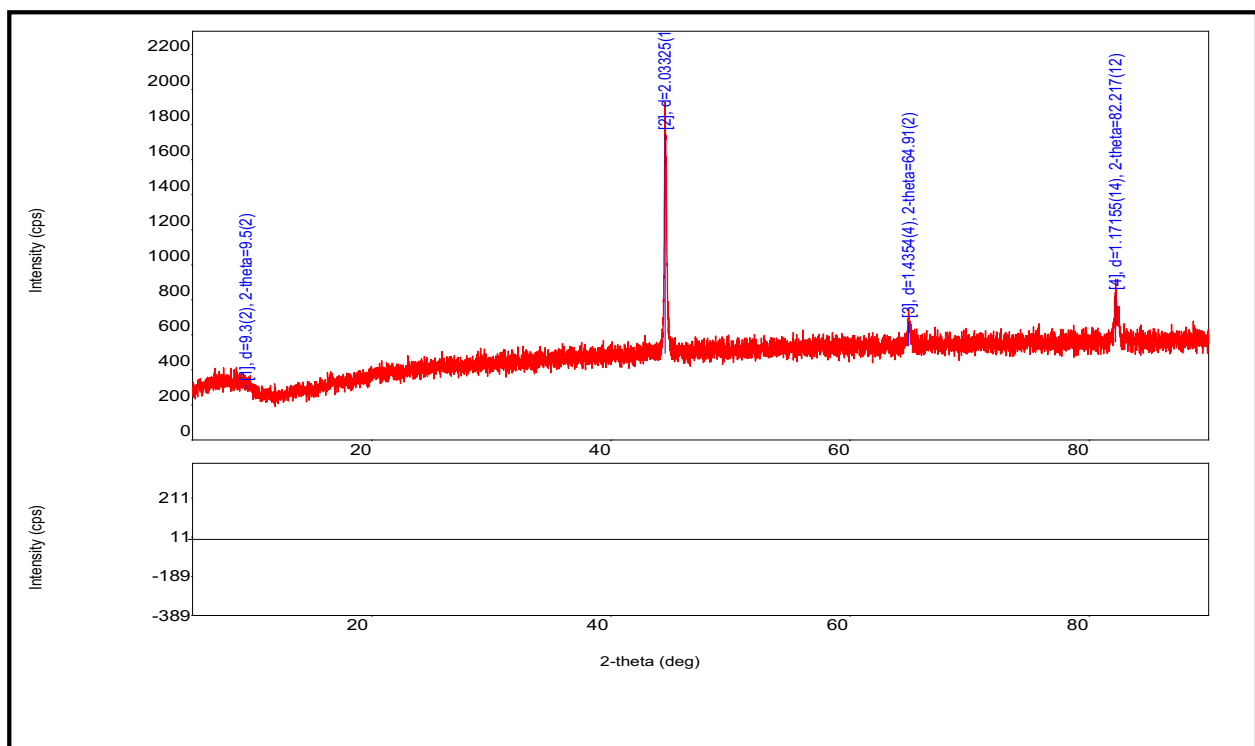


Figure 9. XRD profile of sample in Wood charcoal medium Carburised at 900 °C for 3hours.

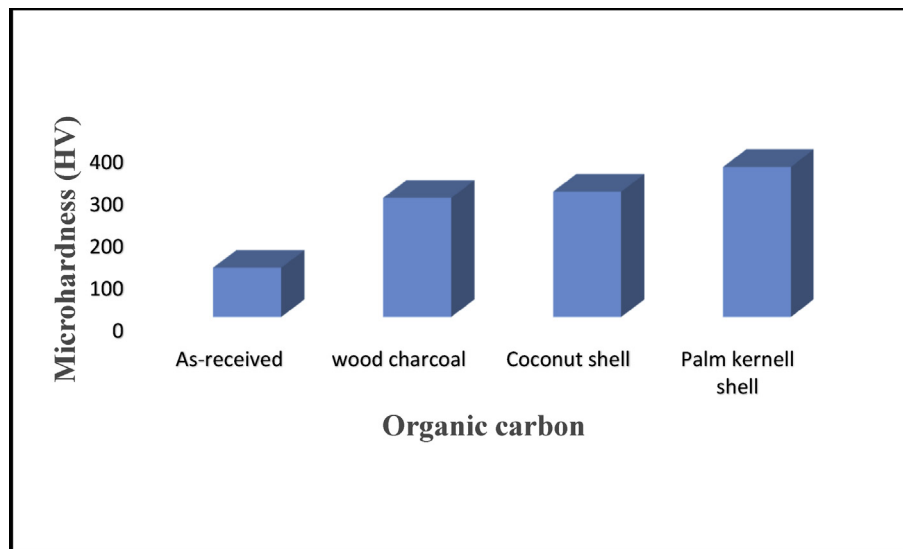


Figure 10. Micro hardness results of carburised gray cast iron in different organic carbon.

More so, Figure 9 showed the XRD profile of gray cast iron carburised in a mixture of pulverised wood and egg shell media. This was characterised by graphite and iron syn phases with peak variations of  $9.5^\circ$ ,  $45^\circ$ ,  $64.91^\circ$ , and  $82.217^\circ$ . The broad peaks revealed crystallite growth of carbon on the substrate forming curved layers on the surface of the substrate as observed on the SEM image (Figure 6). This also, increased the strength and hardness of the carburised material.

#### 3.4. Micro hardness measurement

Figure 10 present the various micro hardness values of carburised gray cast iron in various pulverised organic carbon. From the Figure, it could be depicted that gray cast iron carburised in wood charcoal showed an enhanced hardness of 282.5 Hv compared to as received which is about 116.9Hv. Also, gray cast iron carburised in coconut shell equally increased in hardness against the as-received and that of wood charcoal. However, palm kernel shell showed a better enhancement in hardness with a value of 355.8 Hv. The variation in the hardness results using different organic carbon thus, agrees with the study by Ramli *et al.* [32] and Kocaman *et al.* [33] on the choice of organic carbon for improving mechanical properties of steel.

#### 4. Conclusion

The mechanism of graphite formation using some selected organic carbon for the surface modification of gray cast iron was investigated via heat treatment under the same temperature and holding time. The microstructural analysis showed that there was a homogenous diffusion of carbon which subsequently formed graphite layers on the substrate. However, substrate carburised in pulverised palm kernel shell revealed excellent micro hardness result compared to as-received and the substrate carburised in other media. The finer microstructure and improved hardness properties of the carburised substrate will improve the tribological properties of the carburised substrate.

#### Declarations

##### Author contribution statement

Enesi Y. Salawu: Conceived and designed the experiments; Performed the experiments; Wrote the paper.

Oluseyi O. Ajayi: Conceived and designed the experiments.

Anthony O. Inegbenebor, U.O. Uyo: Contributed reagents, materials, analysis tools or data.

Stephen Akinlabi, Esther Akinlabi, A.P.I Popoola: Analyzed and interpreted the data.

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##### Competing interest statement

The authors declare no conflict of interest.

##### Additional information

No additional information is available for this paper.

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