Exploratory study on Carburisation of Mild Steel using Selected Agroforestry Wastes

M.O.H. Amuda* F.T. Lawal and A. S. Adeyoye

Materials Development and Processing Research Group, Department of Metallurgical and Materials Engineering, University of Lagos, Lagos, Nigeria 101017
*Corresponding author e-mail: mamuda@unilag.edu.ng

Abstract
In the present work, exploratory study was conducted on selected agroforestry wastes as carbonaceous source for carburisation of mild steel in relation to conventional charcoal. Pulverised and carbonised coconut shell (CS), coconut bunch fruit (CBF), palm kernel shell (PKS) and palm kernel bunch fruit (PKBF) and their blends were combined in various proportions with $CaCO_3$ for pack carburisation of mild steel. Carburisation was accomplished by heating the samples to 950 °C for 8 hours followed by oil quenching and subsequent tempering at 450 °C and 550 °C, respectively. Microstructures in the agroforestry waste-carburised samples were similar to those obtained in charcoal carburised mild steel but with differential martensitic morphology and distribution. The hardness profile in the carburised samples correlated to the carbon potential of the carburising source in the trend PKS: 217.24Hv; CS: 203.32Hv; PKBF: 196.23Hv and CBF: 190.4Hv. Similar trend was obtained in the wear behaviour of the carburised samples. The impact strength of the selected agroforestry wastes compared favourably with the conventional charcoal and showed no correlation to the carbon potential. Whilst this study suggests that the selected agroforestry wastes are promising as carburising agents, a further investigation is imperative to determine the optimised carburising conditions as well as the technological framework for carbonisation of the wastes.

Keywords: Carbonised-agroforestry-wastes, carbon potential, charcoal, martensite morphology, wear characteristics

1.0 INTRODUCTION

Post-harvest and processing wastes collectively referred to as agroforestry wastes often constitute environmental nuisance and blight to sight due to lack of effective management framework (Ramírez-García *et al.,* 2019; Amulya, 2016; Nagendran, 2011). In some situations, the wastes are incinerated in open air contributing to climate condition alteration (London, *et al.,* 2011). Yet, these wastes are essentially carbonaceous that could be explored for both technological and economic benefits (Rajput *et al.,* 2014).

Agroforestry wastes contain significant amount of carbon. Nair *et al.* (2010) estimated the carbon stored in agroforestry systems to be in the range 0.29 - 15.21 Mg C ha^{-1} yr^{-1} above ground and in the range 30 - 300 Mg C ha^{-1} yr^{-1} in the soil up to 1 m deep. Agele *et al.* (2015) also reported that the percentage of carbon in a typical agroforestry waste is greater than 50%. As such, the carbon available in these wastes could be harnessed as substitute to green-house gas emitting charcoal for increasing the surface carbon of steel for regulated case hardening of steel. This is particularly significant in surface modification of low carbon steel for improved mechanical and tribological properties via using pack carburisation technique.

In this regard, several investigations have been conducted on the suitability of some agro-based by-products as alternative carbonaceous materials for carburisation of low carbon steel. Some of the investigated agricultural wastes include PKS, cow bone, sea shell, and egg shell wastes (Aslam, 2016; Ihom *et al.,* 2013; Ihom, 2013; Oyetunji and Adeosun, 2012; Aramide *et al*, 2010; Ohize, 2009). In related works, Akinluwade *et al*. (2012) and Arthur et al., (2016) reported better outcomes in contrast to the use of carbon only when cassava leaves were utilized as carbon and nitrogen sources for surface hardening of mild steel. The cyanide in the cassava leaves is the source of the carbon and nitrogen harnessed.

Whilst these investigations reported that agro-based by-products are suitable for pack carburisation of low carbon steels, there was inhibition and disincentive in some of the carbonaceous sources considered. For instance, cyanide has been established as a toxic carcinogenic substance with serious health implications. It is thus a health-risk and non-environment-friendly carbonaceous source (Jaszczak *et al.,* 2017). In other instances, the carbon potential of the applied wastes was affected by other phytochemical or volatile species in the wastes which could interfere with the diffusion reaction during the carburisation process (Mittemeijer and Somers, 2015). As such, there is lack of credible information on the active carbon potential of these agroforestry wastes in relation to the obtainable penetration depth in a typical pack carburisation process.

In addition to these process limiting challenges, the property characterization in most of the previous works on carburisation using agroforestry wastes was restricted to wear phenomenon with little or no consideration for microstructural characterization and mechanical properties (Aslam, 2016; Ihom *et al*., 2013; Aramide *et al*., 2010). Whereas, in most service situations, materials are exposed to loading conditions other than wear. Thus, in exploring the potential of other agroforestry wastes for carburisation, both surface and bulk properties such as wear and impact strength must be wholesomely investigated.

It is therefore imperative that in establishing the potential of these agroforestry wastes for carburisation, definitive answers must be provided to some posers. Some of these issues include: the microstructural evolution in mild steel carburised with agroforestry wastes in relation the conventional charcoal as well as the mechanical properties differential in agroforestry-waste carburised mild steel.

The success of the current investigation provides potential substitute for the conventional charcoal as well as the use of environment and health-risk free carbonaceous source for carburisation rather than the toxic carcinogenic cyanide from cassava leave. This opens a new vista of opportunity for generating economic activity and wealth from these abundant wastes which are currently being incinerated in the open or allowed to biodegrade over a long period of time.

2.0 MATERIALS AND METHODS

2.1 Preparation of Agroforestry Waste Materials

The agroforestry waste carbonaceous materials used in the present investigation were PKS, PKBF, CS and CBF. These materials sourced from various farmyards and markets in Lagos and Ogun States were afterwards sun-dried for three (3) weeks to reduce the moisture content. This was followed by further drying in a furnace at 110^{o}C to remove chemically combined moisture as shown in Figure 1, and then pulverized into fine powder using a fabricated pin cutter. Carbonization of the selected agroforestry wastes was accomplished by following the protocol described by Ntuli and Hapazari (2013) as well as Wiratmoko and Halloran (2009) using a 1500 ^{o}C rated muffle furnace. Eighty grams each of PKS, PKBF, CS, and CBF was individually weighed into a clean cylindrical crucible and placed in the furnace at a pre-determined temperature of 850 ^{o}C. They were held at this temperature for 4 hours in order to completely remove the impurities, volatile materials and eliminate the compound carried by flue gas. The carbonized pulverised agroforestry wastes are presented in Figure 2 whilst the carbon distribution in the samples determined via atomic absorption spectroscopy is presented in Table 1.

Figure 1: Agro waste materials samples (a) dried CS (b) dried CBF (c) dried PKS and (d) dried PKBF

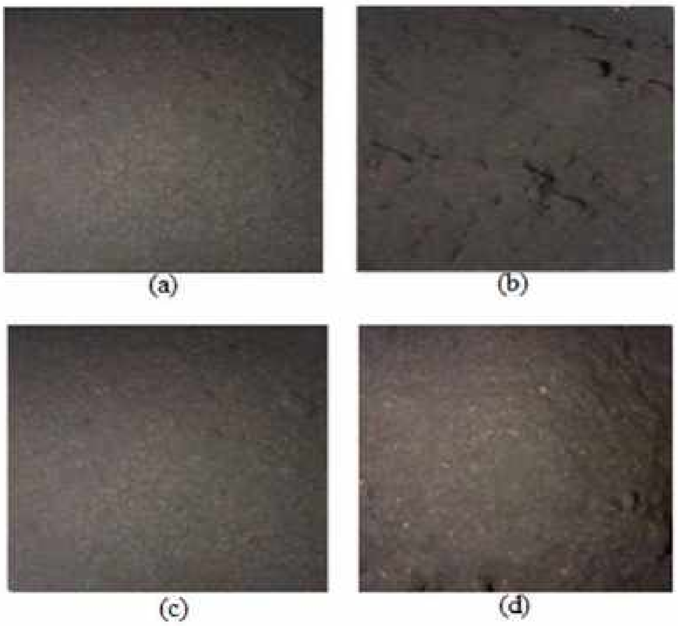

Figure 2: Carbonized agro waste material at 850 ^oC for 4 hours: (a) PKS, (b) PKBF, (c) CS and (d) CBF

Table 1: Carbon compositional analysis of the carbonised agroforestry-waste materials and Charcoal

Agroforestry Waste Materials	*Percentage Composition (wt.%)*	
	% Carbon	% Organic Carbon
CS	12.300	6.26
PKBF	10.920	5.53
PKS	12.530	6.34
CBF	1.335	0.67
C	30.000	-

2.2 Preparation of Mild Steel Material

The mild steel used for the present investigation was sourced from Owode International Steel Market, Lagos, Nigeria on GPS 6° 36' 38.88'' E 3° 25' 8.039''. The sample shown in Figure 3 was cleaned and machined to the specification presented in Figure 4 for pack carburisation. Optical emission spectrometer was used to determine the chemical composition of the sample which is presented in Table 2.

Table 2: Composition of Mild Steel rod used for pack carburisation

Materials	*Elemental Composition (wt.%)*								
	C	Si	Mn	P	S	Cr	Ni	Mo	Fe
Mild Steel Rod	0.16	0.054	0.405	0.011	0.018	0.0076	0.025	0.010	Bal.

Figure 3: Prepared specimen for carburisation

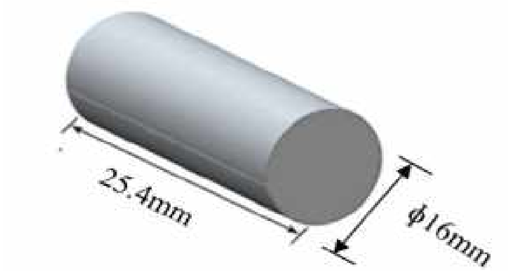

Figure 4: Dimension of carburised specimen

2.3 Pack Carburisation Process

The distribution of various agroforestry waste used for pack carburisation in the present investigation is presented in Table 3.

Table 3: Distribution of the selected agroforestry waste for pack carburisation

Agroforestry Waste Materials	Carbonised Blend	Composition of Blend (wt. %)
	PKS_1	100 PKS
PKS	PKS_2	80 PKS – 20 $CaCO_3$
	PKS_3	75PKS – 25 $CaCO_3$
	$PKBF_1$	100 PKBF
PKBF	$PKBF_2$	80 PKBF – 20 $CaCO_3$
	$PKBF_3$	75 PKBFS – 25$CaCO_3$
	CBF_1	100 CBF
CBF	CBF_2	80 CBF – 20 $CaCO_3$
	CBF_3	75 CBF – 25 $CaCO_3$
	CS_1	100 CS
CS	CS_2	80 CS – 20 $CaCO_3$
	CS_3	75 CS – 25 $CaCO_3$

Using 80g basis, evaluation by weight of the carbonaceous material as well as the energizer in the carburizing compound was conducted using Eq. 1.

$$\text{Weight of each carburiser} = Composition\left(\frac{wt\%}{100}\right) * 80\,g \tag{1}$$

Thus for 80/20 composition of carburiser and $CaCO_3$

20wt% $CaCO_3$ = $\frac{20}{100} * 80$ = 16g of $CaCO_3$

80wt% Carburiser = $\frac{80}{100} * 80$ = 64g of Carburiser (CBF, CS, PKBF and PKS).

The adapted weight of the carbonaceous materials to the energizer based on different weight ratios is presented in Table 4.

The prepared mild steel samples were placed in and covered fully with the prepared carburisers' ratio (Table 3) in a fabricated steel box. The box was kept tightly sealed using fireclay to restrict gas diffusing into and out of the box.

Table 4: Weight of each agro-waste carburisers

Compound	*Ratio of Carbonaceous Material/Energiser*		
	Wt		
	100	80/20	70/30
CS	80	64	56
PKBF	80	64	56
PKS	80	64	56
CBF	-	16	24

The carburizing box was placed in a muffle furnace and heated to a carburizing temperature of 950 ^oC for 8 hours to allow diffusion of carbon at austenitic temperature. The carburised samples were then removed from the furnace with a tongue and quenched in oil. They were then sectioned into two, and finally tempered at 450 °C and 550 °C, respectively for 1 hour.

2.4 Microstructural Characterization

The tempered samples were prepared for microstructural examination by mechanical grinding in a series of emery papers in sequence of grit sizes 240, 320, 400 and 1000. Final surface preparation was conducted by mechanical polishing using 0.05 µm alpha agglomerated alumina suspension. They were finally etched in 2% Nital solution for about five seconds and then rinsed in water followed by a spray of analytical grade methanol before microscopic examination.

2.5 Mechanical Property Characterization

Microhardness distributions in the polished samples were acquired at three different locations (0, 8 and 12 mm) on both tempered samples in the longitudinal direction using Vickers tester. For each of the samples, the test was conducted thrice and the average value recorded. Wear test was conducted on the samples using an improvised pin-on-disk type machine in which a mechanical abrasive was mounted on a metallurgical polishing machine. The samples served as the pin while the abrasive material served as the disk that rotated about its central axis. The characterizations were conducted at a predetermined speed but varying loads and times as provided in Table 5.

Table 5: Matrix of wear test schedule

Load (N)	Time (sec)	Sliding Distance (rev)
11.76	60	12,566
15.68	120	25,133
19.6	180	37,699
23.52	240	50,265

Impact values were estimated using Rolfe-Novak-Barsom developed correlation between derived Charpy V-notch (CVN) data and fracture toughness as shown in Eq. 2 (Barsom and Rolfe, 1970) as well as Cahoon's expression presented in Eq. 3 (Pavlina and Van Tyne, 2008),

$$\frac{(k_{IC})^2}{\sigma ys} = 5\frac{cvn}{\sigma ys} - 0.25 \quad (2)$$

$$\sigma_{ys} = -90.7 + 2.876HV \quad (3)$$

Where,

σys = Yield Stress

HV= Vickers's Hardness

CVN= Impact V. notch test

K_{Ic}= Fracture Toughness

RESULTS AND DISCUSSION

3.1. Carbonisation Potentials of the Agroforestry Waste Materials

The result of carbonisation for each of the carbonaceous agroforestry material is shown in Figure 5. The figure revealed that PKBF, PKS and CS have carbon potential averaging 6% whereas it is about

1% in CBF. This in relation to the almost 15% in charcoal indicates that though the carbon potentials in PKBF, PKS and CS are about 45% of that of charcoal, they could be explored for carburisation whilst the possibility of further exploration in CBF is quite low.

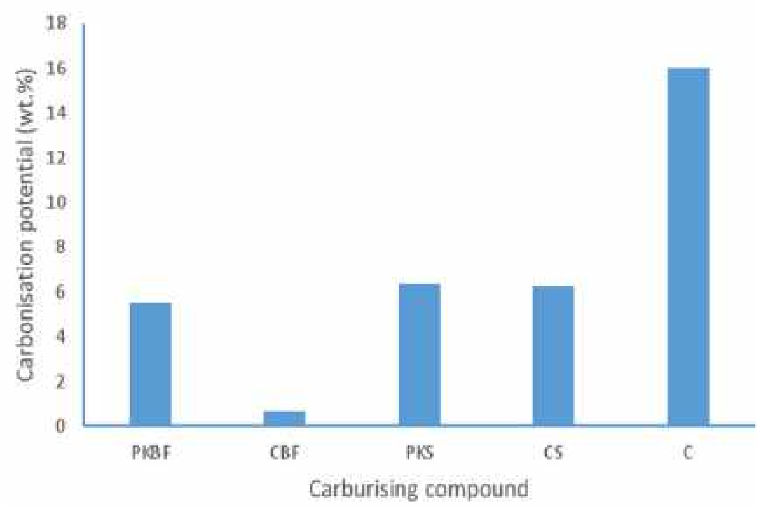

Figure 5: Carbonisation potential of the various carburising compounds

3.2 Microstructural Analysis

Figure 6 presents the micrograph of the uncarburised sample obtained from optical microscope. The figure represents a microstructure that is characterized by fine pearlite (dark) in the matrix of the ferrite (white) which is typical of all reported micrographs of uncarburised low carbon steels (Klenam *et al.,* 2015).

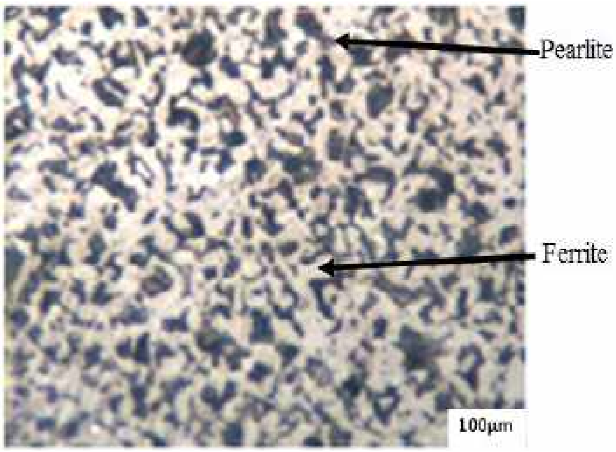

Figure 6: Microstructure of uncarburised sample

Figure 7 presents the microstructure of the charcoal carburised sample. The microstructures of the cases produced consist essentially of plate martensite, retained austenite and coarse-grained martensite. The samples carburised at 100 wt. percent charcoal possess a matrix with intergranular carbide whilst there is substantial retained austenite in the samples carburised with 80 and 75 wt. percent charcoal. These differential distributions of phases in the microstructure of the carburised samples are expected to have significant influence on their mechanical properties.

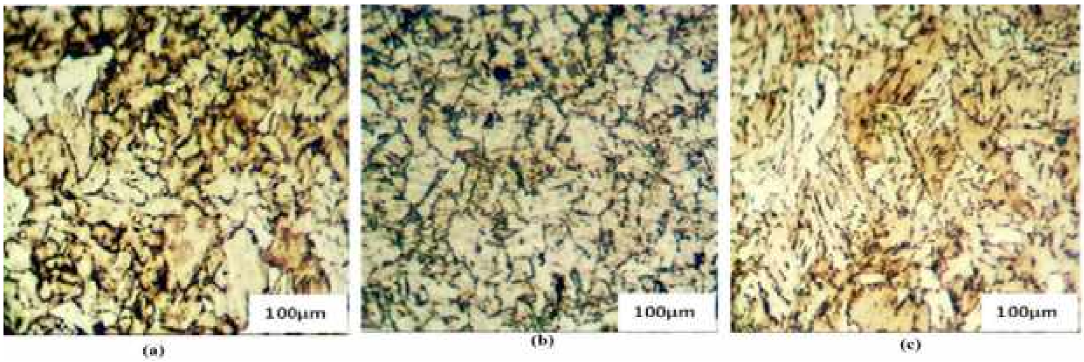

Figure 7: Microstructure of samples carburised in Charcoal matrix at: (a) 100wt%, (b) 80/20wt% and (c) 75/25wt%

Figure 8-11 presents the microstructure of agroforestry-wastes carburised samples. The microstructure consists essentially of martensite and retained austenite but the morphology and distribution of these phases vary with different carbonaceous sources. The morphology and distribution of martensite in the samples carburised with PKBF is shown in Figure 8. Samples carburised with 100% PKBF show lower distribution of martensite but with plate-like morphology similar to those carburised with charcoal. Those carburised with both 80% and 75% PKBF show higher distribution of martensite and more retained austenite. The morphology of those carburised at 75% PKBF shows a refined morphology.

The morphology and distribution of martensite in the samples carburised with 100% CBF shown in Figure 9 revealed higher martensite distribution with plate-like morphology similar to those carburised with charcoal. Those carburised with both 80% and 75% CBF shows lower distribution of martensite and more retained austenite.

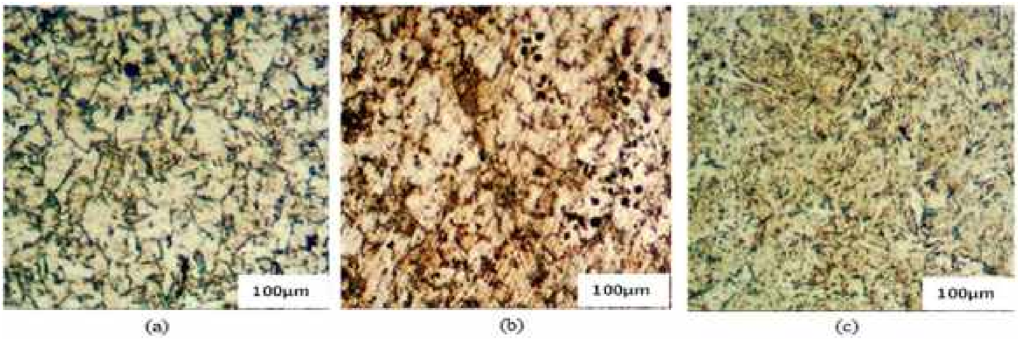

Figure 8: Microstructure of samples carburised in PKBF at: (a) 100wt%, (b) 80/20wt% and (c) 75/25wt%

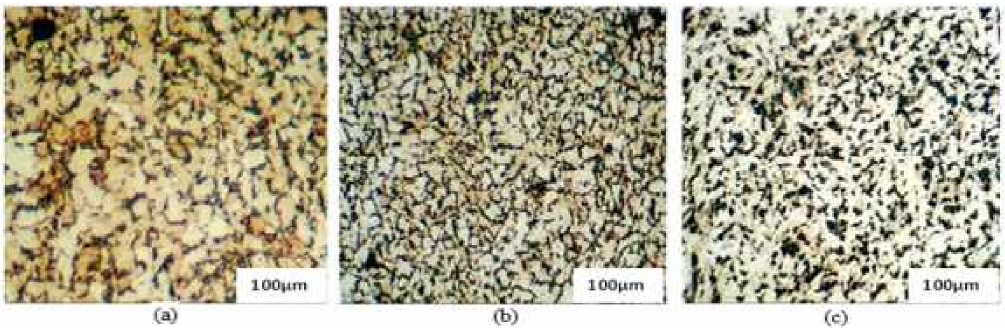

Figure 9: Microstructure of samples carburised in CBF at: (a) 100wt%, (b) 80/20wt% and (c) 75/25wt%

The microstructural characteristics in samples carburised with PKS and CS are presented in Figure 10 and 11. Similar to the trend in CBF, the microstructure of samples carburised with 100% PKS shows higher distribution of martensite whilst those at 80% and 75% show lower distribution of martensite and more retained austenite. However, unlike CBF, the microstructure of samples carburised with 100% CS shows lower distribution of martensite whilst those at 80% and 75% shows increased distribution of martensite but reduced retained austenite.

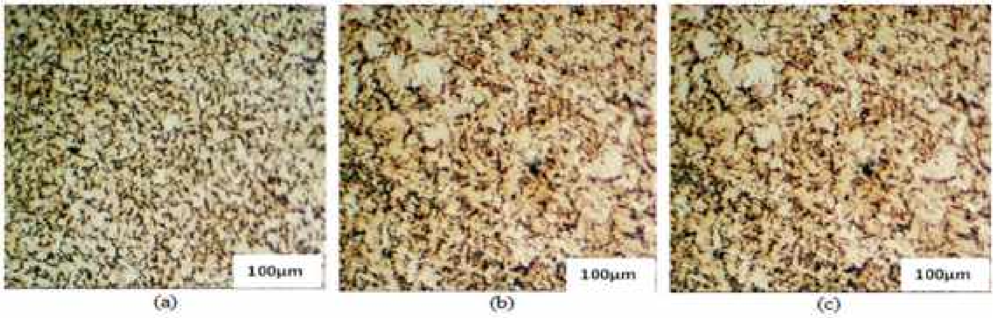

Figure 10: Microstructure of samples carburised in in PKS at: (a) 100wt%, (b) 80/20wt% and (c) 75/25wt%

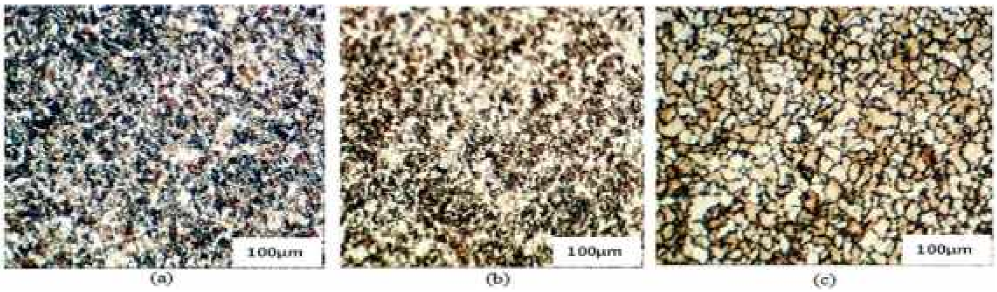

Figure 11: Microstructure of samples carburised in CS at: (a) 100wt%, (b) 80/20wt% and (c) 75/25wt%

3.3 Microhardness Characteristics

The hardness profile in agroforestry waste carburised samples tempered at 450 ^oC is shown in Figure 12 whilst that in those tempered at 550^oC is shown in Figure 13. Figure 12 shows that the hardness value in the various agroforestry-wastes carburised samples progressively decreases from the surface to the core of the samples in the radial direction. In the figure, PKS exhibits the highest surface hardness value of 217 HV, followed by CS with 203.3 HV and then PKBF with 196.23 HV. CBF exhibits the least surface hardness with a maximum value of 190.4 HV. The hardness in the charcoal carburised sample however is lower compared to any of the agroforestry-wastes carburised samples.

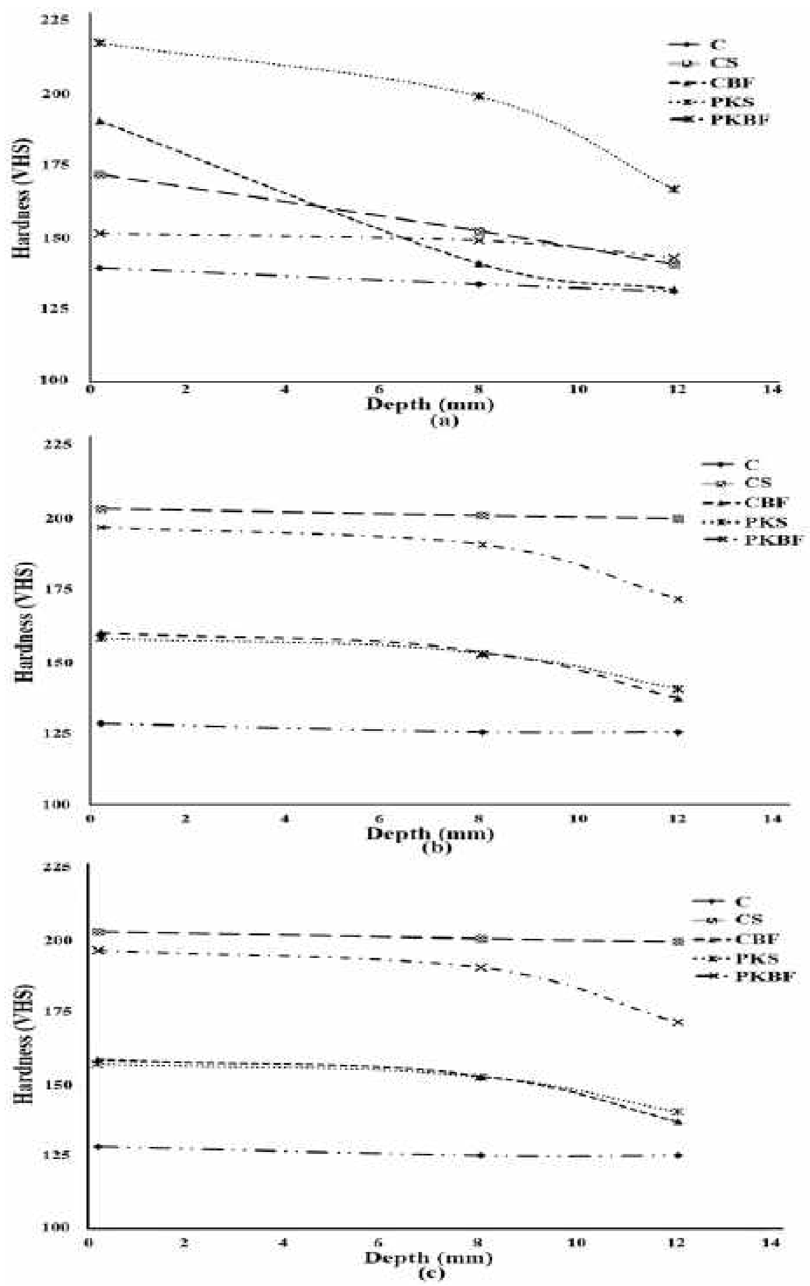

Figure 12: Vickers hardness distribution in carburised samples tempered at 450°C: (a) 100 wt.%, (b) 80/20 wt% and (c) 75/25 wt.%

Figure 13 exhibits similar trend to Figure 12. The hardness value in the various agroforestry-wastes carburised samples equally decreases progressively from the surface to the core of the samples in the radial direction and the hardness in the charcoal carburised sample is lower compared to any of the agroforestry-wastes carburised samples.

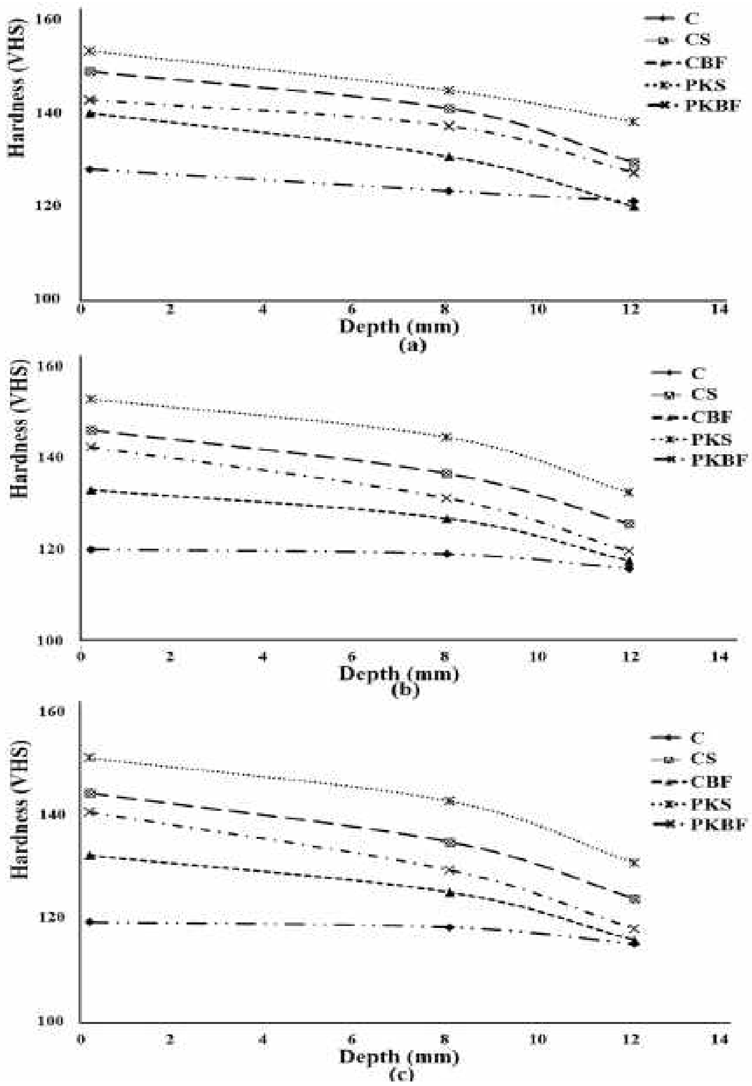

Figure 13: Vickers hardness distribution in carburised samples tempered at 550 °C: (a) 100 wt.%, (b) 80/20 wt% and (c) 75/25 wt.%

But unlike Figure 12, the hardness values in the various agroforestry-wastes tempered at 550°C is lower. PKS exhibits the highest surface hardness value of 154.2 HV, followed by CS with 149.8HV and then PKBF with 143.7HV. CBF still exhibits the least surface hardness with a maximum value of 140.8HV.

The range of hardness values in the agroforestry-wastes carburised samples in relation to the carbon potential of the carburisers suggests that whilst the carbon potential provides an indicator for carburisation, it does not reflect in the depth of carbon diffusion in the samples; rather it appears there is another phenomenon controlling the carbon diffusion. Literature suggests that the diffusion co-efficient and kinetics are likely to be the controlling parameters in this instance.

3.4 Analysis of Estimated Impact Strength of Carburised Samples

Figure 14 presents the impacts toughness in the agroforestry waste carburised samples tempered at 450 °C whilst that in those tempered at 550 °C is presented in Figure 15. In Figure 14, the impact toughness in the charcoal carburised sample is 28.15 J which is lower relative to any of the agroforestry-wastes carburised samples. CS exhibits the highest impact toughness value of 31.50 J followed by PKS with a value 29.80J. PKBF and CBF exhibit similar impact toughness with a value 29.18 J.

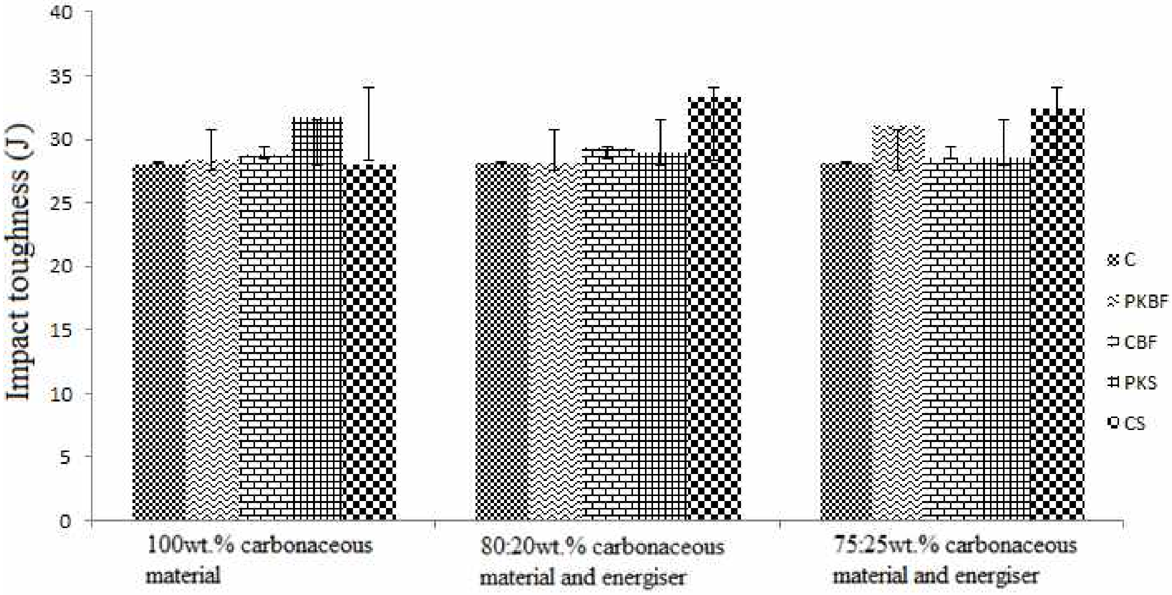

Figure 14: Impact toughness of carburised samples tempered at 450°C

The trend obtained in agroforestry carburised samples but tempered at 550 °C shown in Figure 15 is different from that obtained in Figure 14. In Figure 15, PKS exhibits the highest impact toughness with the 28.15J followed by CS with the value 28.13J. But similar to Figure, PKBF and CBF exhibits similar impact toughness with a value of 28.10J. The impact toughness in the charcoal carburised samples is equally lower compare to any of the agroforestry-waste carburised samples.

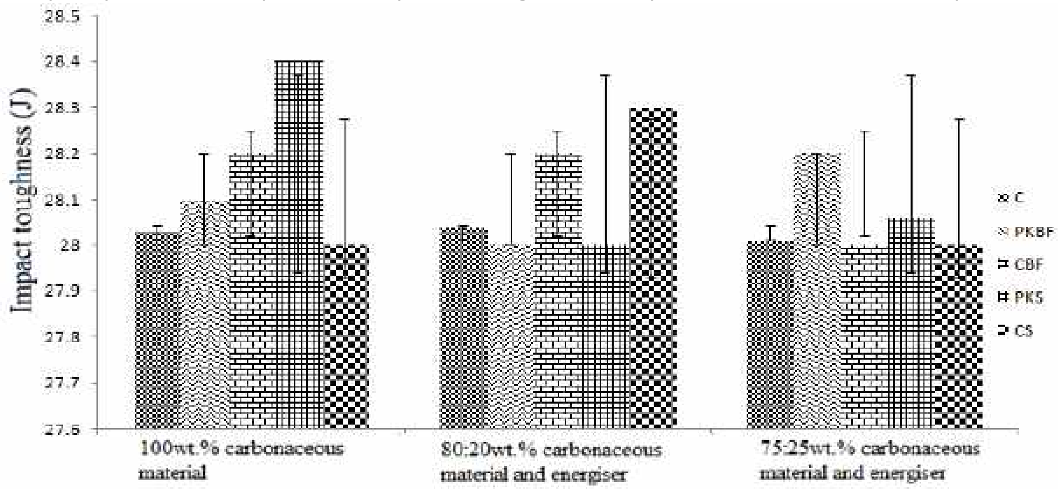

Figure 15: Impact toughness of carburised samples tempered at 550°C

3.5 Wear Characteristics

The wear characteristic of charcoal and agroforestry-wastes carburised mild steel tempered at 450°C is shown in Figure 16. The figure shows the wear rate parameter against time under a loading condition of 11.76N at a constant speed 250 rev/min. The figure indicates that the wear rate increases with increasing time for both charcoal and agroforestry-wastes carburised samples. Similar to the trend observed in microhardness characteristics, the samples carburised with charcoal has the highest wear rate whilst the samples carburised with PKS has the least average wear rate of

0.825cm^3/s followed by CS samples with 0.96 cm^3/s. PKBF and CBF samples have an average wear rate of 1.055 cm^3/s and 1.225 cm^3/s, respectively.

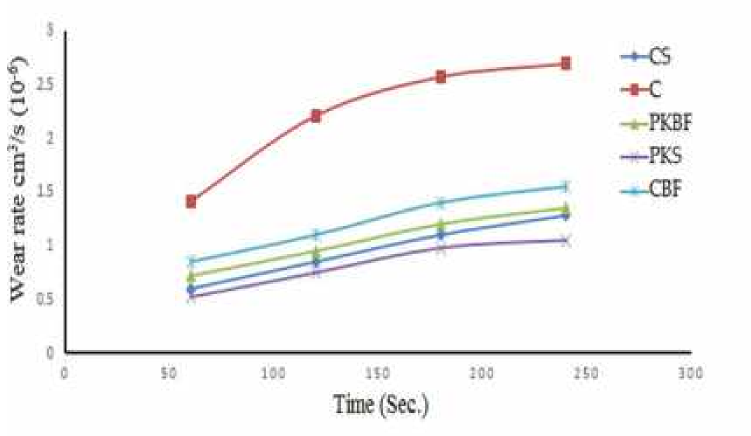

Figure 16: Influence of time on the wear rate of the carbonaceous samples at constant load

Figure 17 shows the wear rate parameter against load at a constant time of 240 seconds. The figure reveals a trend similar to that present by the wear characteristics against time with the charcoal carburised samples showing the highest wear rate. The trend in agroforestry-waste carburised sample is similar to the character against constant time shown in Figure 16.

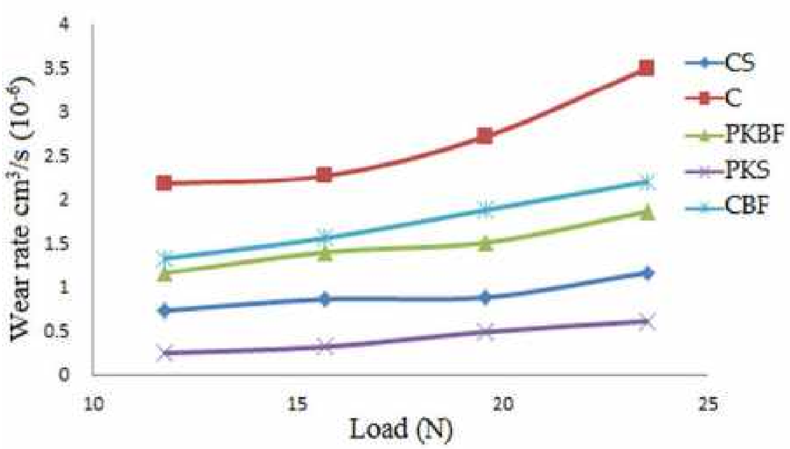

Figure 17: Influence of applied load on the wear rate of the carbonaceous samples at constant time

The influence of varying time on the wear resistance of the carbonaceous samples at constant load and speed is depicted in Figure 18. It is observed that the magnitude of the wear resistance decreases with increasing carburising time, a trend that correlates inversely to that of wear rate with the charcoal carburised samples showing the least wear resistance. In this figure, PKS carburised samples show the highest value of wear resistance whereas CS, PKBF and CBF maintained their trajectory just as in wear rate.

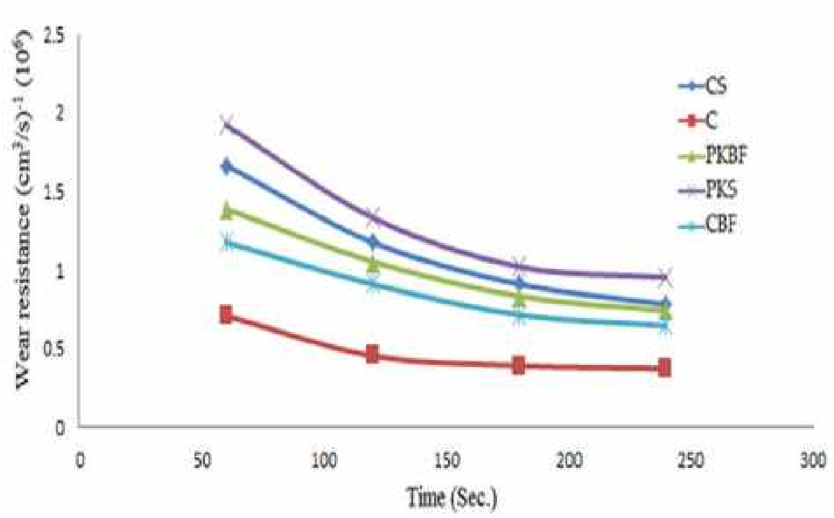

Figure 18: Influence of time on the wear resistance of the carbonaceous samples at constant load

Figure 19 shows the wear resistance parameter against load, at a constant time of 240secs and speed of 250rev/min. The results show that wear resistance decreases with increasing load for all carburised specimens. The sample carburised with PKF has the highest average wear resistance followed by the sample carburised in CS and the specimen with the least wear resistance is C.

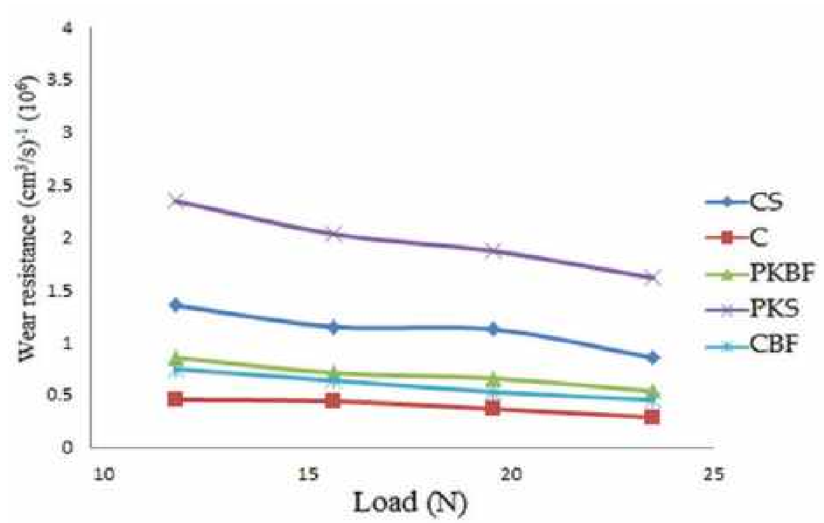

Figure 19: Influence of applied load on the wear resistance of the carbonaceous samples at constant time

4.0 CONCLUSIONS

The present study investigated the potential of pulverized and carbonised CS, CBF, PKS and PKBF agroforestry waste as carbonaceous source for pack carburisation of mild steel relative to the conventional charcoal. The following were established from the study:

(i) The carbonised agroforestry wastes have appreciable carbon potential to be explored for carburisation of mild steel.

(ii) Microstructural characteristics in the agroforestry-waste carburised mild steel exhibit identical microstructure to those presented by charcoal. However, the morphology and distribution of martensite and retained austenite are different.

(iii) Carbon potential is not the only parameter controlling the depth of penetration of the carbonaceous materials. It is postulated that both the diffusion co-efficient and kinetics are significant parameters other than the carbon potential. .

(iv) The impact toughness compared favorably with those of conventional charcoal with about 11% differential.

Whilst this exploratory study established that the carbonised selected agroforestry wastes are potential carburisers, the disparity in the mechanical properties of the materials with identical carbon potential suggests a need for further investigation on the diffusion kinetics. Other than this, the optimal carburizing conditions as well as the technological framework for carbonization of the waste require further research.

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