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CALCIUM CARBONATE OBTAINED FROM MUSSEL SHELL WASTE: A NOVEL ENERGIZER IN THE CARBURIZATION OF MILD STEEL

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ABSTRACT

The rising cost of the conventional energizers couple with the effect of the residue may have on the environment, waste materials especially those that are widely reported to possess carbonate and are eco-friendly are gradually attracting attention globally. This paper therefore, presents the opportunity of using mussel shell waste as an alternative energizer to the conventional energizers in the pack carburization of mild steel. The carburization treatment was carried out at the austenitic temperature of 925°C, 950°C and 975°C, and holding time of 1, 2 and 3 hours respectively. Thereafter, the test samples were quenched in water to obtain a hardened martensitic steel surface. Standard method was adopted to determine the surface hardness of the carburized and un-carburized steel samples. Micro-structural examination was also performed using standard metallographic techniques to observe the microstructure formed. The results showed a progressive increase in the surface hardness of the carburized steels as the temperature and holding time was increased for all the steel samples carburized with and without the energizer. However, the steel samples carburized with prosopisafricana seed pod and mussel shell waste mixture exhibited higher surface hardness compared to those carburized with prosopisafricana seed pod waste alone. The maximum surface hardness values of 452.8 VHN, 503.5 VHN and 581.3 VHN were obtained at the carburizing temperature of 925 °C, 950 °C, and 975°C respectively, for 3 hours holding time with the steel samples carburized with prosopisafricana seed pod and mussel shell waste mixture. The control sample gave a surface hardness value of 122.3 VHN. The results of the study have shown that the calcium carbonate from mussel shell waste acted as an effective energizer and would provide good economics in the carburization of mild steel.

Keywords: *Carburization, surface hardness, mild steel, mussel shell, prosopisafricana seed pod.*

INTRODUCTION

The ease with which the properties of steels can be altered at will to obtain certain desirable properties suitable for specific application has made steels to be the most widely used engineering material. There are several processes and methods available to achieve these; one notable method that has continued to be applied to impart enhanced surface hardness in steel is carburization. Carburization is the process of introducing carbon into solid iron-based alloys such as low carbon steels in order to produce a hard case and tough inner core, at the temperatures generally between 850°C and 980°C which is the temperature at which austenite, with its high solubility for carbon is the stable crystal structure. (Khanna, 2008, Higgins, 1993, George and Gabriel, 2009, Sanjib, 2009, and Dosset and Totten, 2013). One of the most common methods utilized to achieve increased carbon in the surface of steel is the pack carburization (Rajput, 2010). The pack carburizing medium usually consists of solid carbon sources essentially wood, animal bone, charcoal, shells of different organisms, charred leather or waste from different agricultural products mixed together with energizer such as sodium carbonate, barium carbonate or calcium carbonate, etc, (Khanna, 2008). Energizers are carbonate materials that are usually mixed with carbonaceous materials to raise the carbon potential of the carburizing medium in order to facilitate the release of nascent carbon into the surface of steel to a level that is dependent on temperature and holding time as well as the carbon potential of the carburizing medium. Energizers function usually by serving as the initial source of supply of carbon dioxide which reacts with the carbon in the carbonaceous material to form the carbon dioxide necessary for the carburizing reaction. Therefore, to ensure continuous supply of CO_2 in order to obtain a better carburizing efficiency; the use of energizer have become necessary. The conventional energizers that are widely used in industry are mostly inorganic synthetic chemical. The increasing cost of the conventional energizers as well the danger, the residue may posed to the environment as it is being disposed of, after utilization, has made researchers to begin to source for other better alternative materials that are eco-friendly, abundant and readily available at little or no cost with viable carbon potential. The new focus which is attracting attention currently is the utilization of waste materials, particularly those that are widely reported to contain carbonate, and are considered less hazardous to human health and the environment. Waste as defined elsewhere in Adzor *et al.* (2017) is anything un-used or not used to full capacity or excess of what is required or garbage, rubbish or trash. Different kinds of wastes are generated from one industry and community to another depending on the activities that are practiced or engaged in. According to Adzor *et al.* (2017) waste will continued to be generated in our environment as long as household, industrial and agricultural activities go on. Therefore, one practical approach that is widely encouraged and promoted as one of the best means of tackling waste disposal problem is recycling or conversion of waste to other reusable products that can be utilize as sources of raw material for another industrial set-up. Although in the true sense, there is no such thing as waste, it is when man has no purpose for a thing that he regards as waste. Based on this, extensive research works have been carried out by seasoned researchers to find ways of converting wastes to other reusable material in order to enhance economic growth, create jobs for the teeming youths and to encourage proper utilization of the limited resources in the world.

Many research works have been carried out on the utilization of different agricultural waste; and some waste of marine organisms to establish their carbon potential as alternative energizers that could be mix with different carbonaceous materials for the enhancement of the surface hardness of low carbon steels for use in application requiring higher surface hardness such as in gears, shafts, cams, etc. However, there are numerous other solid waste materials whose carbon potential are yet to be explored for engineering application, as such are often discarded at different dump site as valueless material in Nigeria. Ihom *et al.* (2013) investigated the used of egg shell waste as an energizer in the carburization of mild steel. The result shows that the steel samples carburized in the carburizing mixtures of egg shell and different carbonaceous materials gave higher hardness values than those carburized using the carbonaceous materials waste alone. Ihom (2013) in his experimental result on the case hardening of mild steel using cow bone as energizer at varying percentage composition of the carburizer (charcoal) and energizer (cow bone) observed that the specimens carburized with 60wt% charcoal and 40 wt% cow bone mixture gave the best result compared to using charcoal alone. Fatoba *et al.* (2013) while studying the suitability of seashell, animal bone and sodium carbonate as energizers in the pack carburization of mild steel concluded that, seashell and animal bone acted as better energizers than sodium carbonate at most of the soaking time for 950^{o}C carburizing temperature. The results of these authors work have proved that waste materials that contain carbonate have great potential to replace the conventional energizers in the near future and their continued utilization for engineering applications would help to enhance the mechanical properties of many engineering components, thus expanding its scope of application. Thus, this will in turn help to solve waste disposal problem while creating wealth for the poor local people in the various communities in Nigeria where the wastes are generated.

Report (Britannica, 2009) described mussel as a family of bivalve molluscs which has two pairs of exterior shell to protect the interior soft edible tissues. It is commonly found in salt and fresh water habitat. In Nigeria, it is an important delicacy of the eople living in the coastal areas of the Southern part of the country. It is found that after the consumption of the meat the shells are often discarded as waste, thereby creating environmental issues. Reports [Britannica] and (Buasri, *et al.*, 2013) indicates equally that mussel shell contain calcium carbonate. Carbonates are effective compounds in carburizing treatment. Therefore, the presence of these compounds in mussel shell offers better opportunity to the replacement of the conventional energizers in the carburization of mild steel; this will in turn help to bring down the cost of carburization. The $CaCO_3$ from mussel shell have been utilized as bioresources of Calcium oxide in catalyzing transesterification to produce biodiesel (Buasri, *et al.*, 2013). No report currently exists in the public domain on the utilization of the calcium carbonate from mussel shell as an energizer in enhancing the carbon potential of carbonaceous materials in the carburization of mild steel.

This study is therefore, aimed at investigating the effectiveness of the calcium carbonate ($CaCO_3$) obtained from mussel shell; which is often discarded after the consumption of its meat as an alternative energizer to the conventional energizer in the pack carburization of mild steel. The based approach of the study consists of correlation of the microstructure with the mechanical property (hardness) and measure of the surface hardness of the carburized steels in order to establish the effectiveness of the $CaCO_3$ obtained from mussel shell waste in raising the carbon potential of the carburizing medium for improved surface hardness.

It is on this basis that the authors are exploring this great opportunity for its reusability as a source of raw material key to the success of pack carburization at significant economics than the conventional energizers.

MATERIALS AND METHODS

Materials and Equipment

The materials and equipment utilized for the study were; mild steel, *prosopisafricana* seed pod wastes, mussel shell wastes, BS sieve analyser, steel boxes, muffle furnace, emery papers, ethanol, nitric acid, metallurgical microscope, weighing balance, digital dynamic hardness tester (VHN), water, fireclay, tong, bench vice and hack saw.

Methods

Preparation of the carburizing agents

The commercial grade of the mild steel used in the study was sourced locally from the central store of the Metallurgical Training Institute, Onitsha. The chemical composition of as-received mild steel plate is shown in Table 1. Figures 1(a) and (b) represent the photographs of the *prosopisafricana* seed pod and mussel shell wastes, respectively. The *prosopisafricana* seed pod which was used as the carbonaceous material was sourced locally in Ikpayongo, Gwer East local government of Benue State, Nigeria. The mussel shell waste was collected from a waste dump site in Warri, Delta State, Nigeria. The *prosopisafricana* seed pod waste; prior to its use was pounded in a mortar using a pestle to extract the seed from the pod. Thereafter, the pod and mussel shell were packed separately in metal boxes and then calcined in a muffle furnace at 450^{o}C and 550^{o}C respectively, to carbonize them and thereafter grinded and sieved through BS sieve analyzer and the weight of the oversize and undersize particles obtained from the different mesh sieve were measured and use to compute the Grain Fineness Number (GFN). The computed values of the GFN of the *prosopisafricana* seed pod and the mussel shell waste particles used in the study were; 57.09 and 54.06 respectively.

Carburizing operation

A total of 19 test specimens prepared according to standard specification as per ASTM standard were used for the study. To obtain a smooth surface of the test samples, the steel samples were grinded using emery cloth of sizes 220, 320, 400 and 800 respectively. The carburization operation was performed in two stages. In the first stage, 9 test samples packed separately in metal boxes and embedded in 100 wt% *prosopisafricana* seed pod waste only. In the second stage, 9 test samples were also packed separately in metal boxes containing mixtures of 60 wt% *prosopisafricana* seed pod waste and 40 wt% mussel shell waste. Each of the metal boxes were sealed with fireclay made into paste with water and allowed to dry under the sun before placing each box in turn inside the heating chamber of the muffle furnace where they were heated to a controlled temperature of 925°C, 950°C and 975°C and held for 1, 2 and 3 hours respectively. After the carburization, the test specimens were removed from the metal boxes and then quenched in water to harden.

Micro - structural examination

For effective examination of the microstructure of the carburized steel specimens, standard metallographic techniques were adopted. After grinding and polishing operations were performed, the specimens were etched with 2% nital before being placed on a metallurgical microscope to view the microstructures and the photographs were taken using a magnification of x400.

Determination of hardness values

The hardness values were determined using a digital dynamic hardness tester. The hardness test was taken at three different spots on the etched surface of each of the carburized steel. After which the average hardness values were computed.

Table 1: Chemical composition of as-received mild steel plate

Element	% Composition
C	0.232
Si	0.220
Mn	0.381
P	0.026
S	0.004
Ti	0.002
Mo	0.004
Al	0.023
Fe	Balanced


(a) (b)

Figure 1: (a) Photograph of the *prosopisafricana* seed pod waste, and (b) Photograph of the mussel shell waste

RESULTS AND DISCUSSION

The results obtained from the study are presented in Table 2. The micrographs of the un-carburized test samples and the carburized steel samples are presented in Figures 2 to 11. Table 2 illustrates how the surface hardness of the carburized steel is affected by the carburizing temperature and holding time. The results in Table 2 clearly demonstrate that, as the carburizing temperature and holding time was increased from 925 to 975°C at the time intervals of 1, 2 and 3 hours respectively, there was a progressive increase in the surface hardness of all the carburized steel samples although not in direct proportion. However, the steel samples carburized with 60 wt% *prosopisafricana* seed pod waste and 40wt% mussel shell waste mixture exhibited higher surface hardness values compared to those carburized with 100 wt% *prosopisafricana* seed pod waste only. This is an indication that the $CaCO_3$ from the mussel shell waste assisted in raising the carbon potential of the carburizing medium which resulted in the higher surface hardness values recorded. The variation in the surface hardness of the carburized steel samples is as result of the quantity of carbon absorbed into the surface layer of the carburized steel samples and is dependent on the carburizing temperature, holding time as well as the carbon potential of the carburizing medium. Therefore, as the carburizing temperature and holding time was increased, the surface hardness also increased. This is in agreement with reports (Ihom, 2013, Nwoke, *et al.,* 2014 and Singh, 2007). Adzor, *et al.* (2016) stated that the higher the amount of carbon absorbed into the steel, the higher the hardness of the martensite formed as the steel is being quenched. Therefore, the quantity of carbon absorbed into the steel at the different carburization temperatures affected the morphology of the martensite formed during quenching. This is in line with earlier reports (Singh, 2007, Singh, 2011 and Raghavan, 1989).

Table 2: Mechanical property of the carburized mild steel

Sample ID	Premix composition	Carburizing tempt. (^0C)	Holding time (hr)	Hardness (VHN)
Control				122.3
A1	100wt% prosopisafricana seed pod	925	1	221.3
A2	100wt% prosopisafricana seed pod	925	2	323.7
A3	100wt% prosopisafricana seed pod	925	3	432.5
B1	60wt% prosopisafricana seed pod +40wt% mussel shell	925	1	318.5
B2	60wt% prosopisafricana seed pod + 40wt% mussel shell	925	2	449.8
B3	60wt% prosopisafricana seed pod + 40wt% mussel shell	925	3	452.8
C1	100wt% prosopisafricana seed pod	950	1	371.7
C2	100wt% prosopisafricana seed pod	950	2	438.5
C3	100wt% prosopisafricana seed pod	950	3	446.3
D1	60wt% prosopisafricana seed pod + 40wt% mussel shell	950	1	415.7
D2	60wt% prosopisafricana seed pod + 40wt% mussel shell	950	2	471.7
D3	60wt% prosopisafricana seed pod + 40wt% mussel shell	950	3	503.5
E1	100wt% prosopisafricana seed pod	975	1	435.8
E2	100wt% prosopisafricana seed pod	975	2	474.3
E3	100wt% prosopisafricana seed pod	975	3	495.5
F1	60wt% prosopisafricana seed pod + 40wt% mussel shell	975	1	489.5
F2	60wt% prosopisafricana seed pod + 40wt% mussel shell	975	2	535.7
F3	60wt% prosopisafricana seed pod + 40wt% mussel shell	975	3	581.3

Figure 2 shows the micrograph of the control sample (un-carburized). The micrograph consists of pearlite (dark patches) and ferrite (white patches) in ferrite matrix. The presence of these phases accounts for the lower surface hardness value recorded. Figures 3 to 5 (a) and (b) show the micrographs of the steel carburized with *prosopisafricana* seed pod waste only, and *prosopisafricana* seed pod and mussel shell waste mixture, at 925°C for 1, 2 and 3 hours holding time respectively. The micrographs revealed the presence of lath martensite and varying degrees of dispersed carbide in ferrite matrix. However, the micrograph of the steel samples carburized with 60 wt% *prosopisafricana* seed pod waste and 40 wt. % mussel shell waste mixture showed more amount of lath martensite in ferrite matrix with varying degree of dispersed carbide along the ferrite grain boundary as compared to the micrographs of the steel samples carburized with *prosopisafricana* seed pod waste alone. This clearly demonstrates that there was an increasing absorption of carbon in the steel surface as the carburizing temperature and holding time was increased. Figures 6 to 8 (a) and (b) show the micrographs of steel carburized with *prosopisafricana* seed pod waste, and *prosopisafricana* seed pod and mussel shell waste at 950°C for 1, 2 and 3 hours holding time respectively, while Figures 9 to 11 (a) and (b) show the micrographs of the steel specimen carburized with *prosopisafricana* seed pod waste only, and *prosopisafricana* seed pod and mussel shell waste mixture at 975°C for 1, 2 and 3 hours holding time respectively. The presence of plate martensite with varying degree of dispersed carbide along the ferrite grain boundary on the micrographs clearly indicates that there was indeed more absorption of carbon in the surface layer of the steel carburized with *prosopisafricana* seed pod waste and mussel shell mixture. Reports (Singh, 2007) and (Raghavan, 1989) posited that with increasing carbon concentration at the steel surface the martensite start ($M_{s)}$ and martensite finish (M_f) temperatures decreases. Thus, as the M_S temperature decreases, as a function of carbon content of the steel, the changed in the morphology of martensite, ie lath to plate and the corresponding increase in its hardness. The higher surface hardness of the plate martensite formed on the steel samples carburized using *prosopisafricana* seed pod and mussel shell waste mixture at the carburizing temperature of 950-975°C and holding time of 1, 2 and 3 hours shows that the carburizing medium was saturated with more carbon than the *prosopisafricana* seed pod waste carburizing medium, hence the increased absorption of carbon into the steel surface. This therefore accounts for the higher surface hardness values recorded at the different carburizing temperatures and holding time for the samples carburized with *prosopisafricana* seed pod and mussel shell waste mixture (Raghavan, 1989).

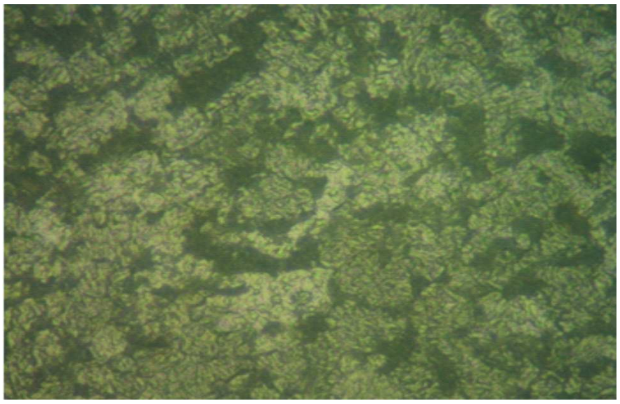
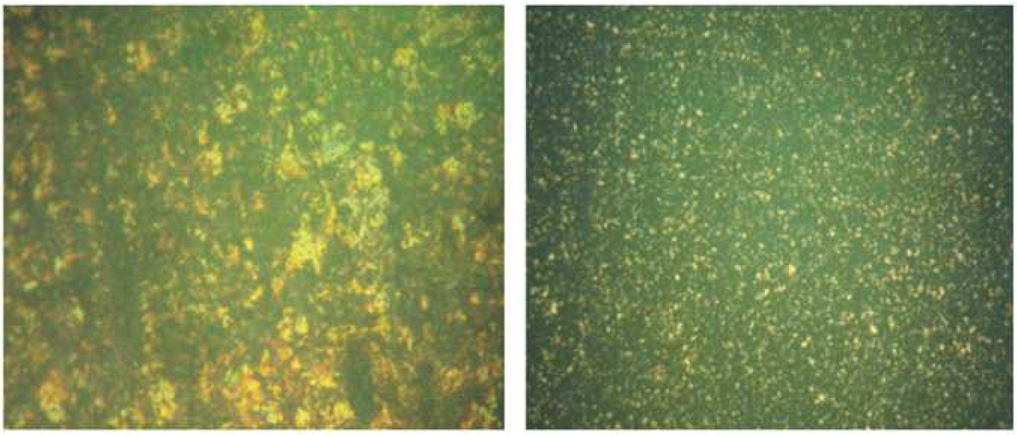
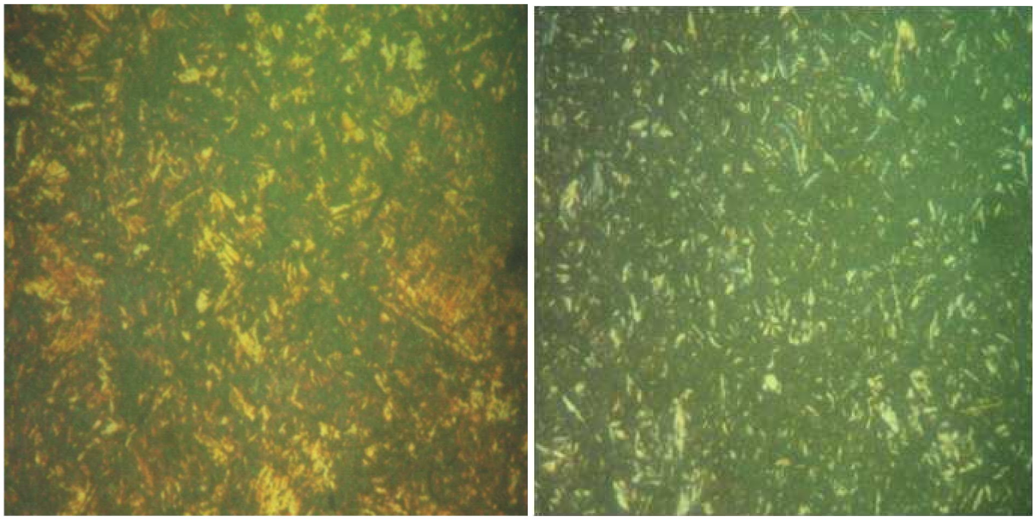


Figure 2: Micrograph of as-received mild steel plate, x400



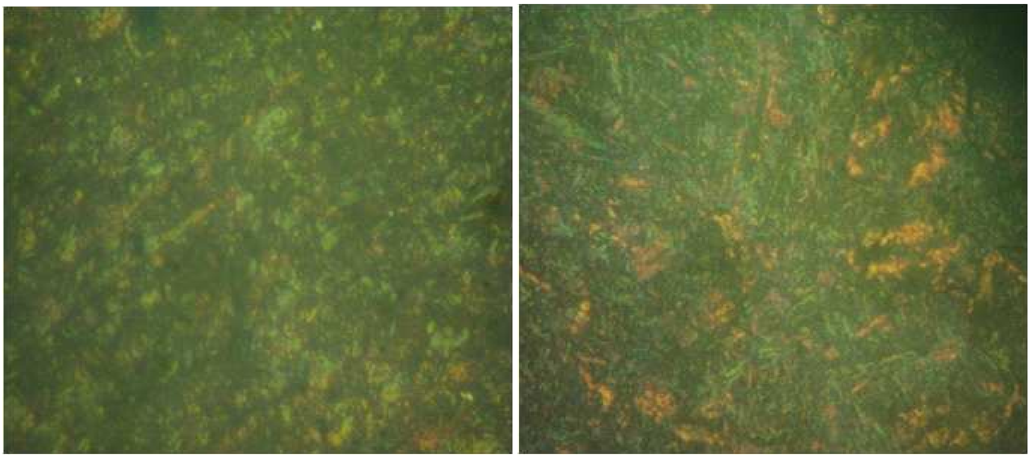
(a) (b)

Figure 3: Micrograph of steel carburized using *prosopisafricana* seed pod only at 925°C for 1 hour, and (b) micrograph of steel carburized using *prosopisafricana* seed and musse l shell mixture at 925°C for 1hour. (lath martensite, dark patches and carbide, white patches) x400



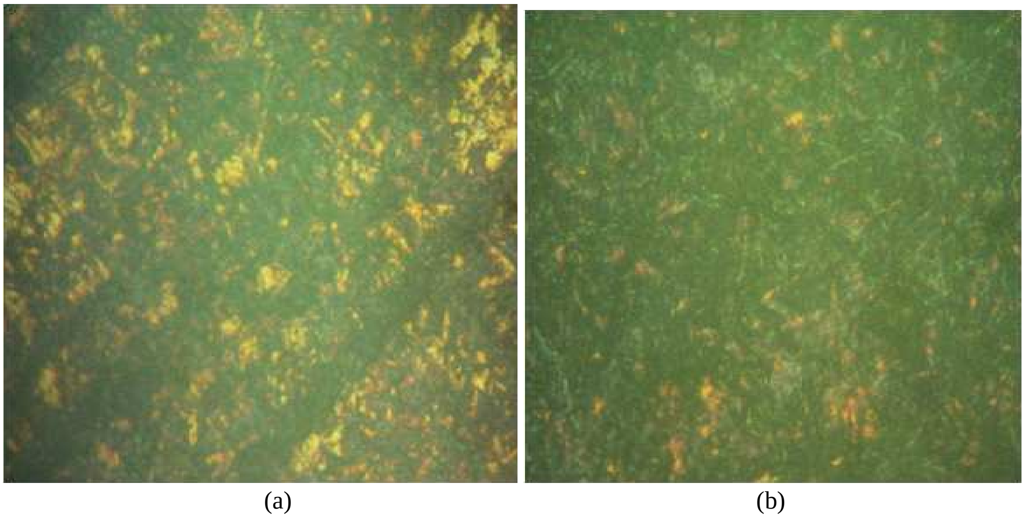
(a) (b)

Figure 4: Micrograph of steel carburized using *prosopisafricana* seed pod waste only at 925^{o}C for 2 hours, and (b) micrograph of steel carburized using *prosopisafricana* seed pod and mussel shell waste at 925^{0}C for 2hours. (lath martensite, dark patches and carbide, white patches) x400



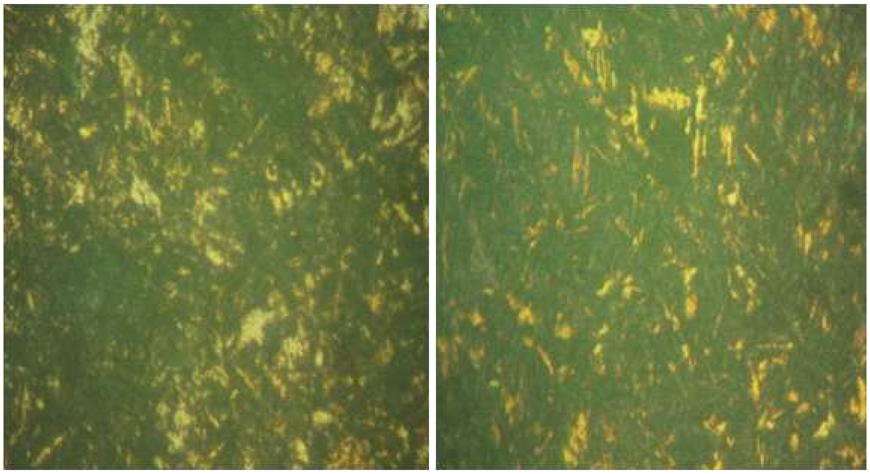
(a) (b)

Figure 5: Micrograph of steel carburized using *prosopisafricana* seed pod only at 925^{o}C for 3 hours, and (b) micrograph of steel carburized using *prosopisafricana* seed pod and mussel shell waste mixture at 925^{o}C for 3hours. (lath martensite, dark patches and carbide, white patches) x400



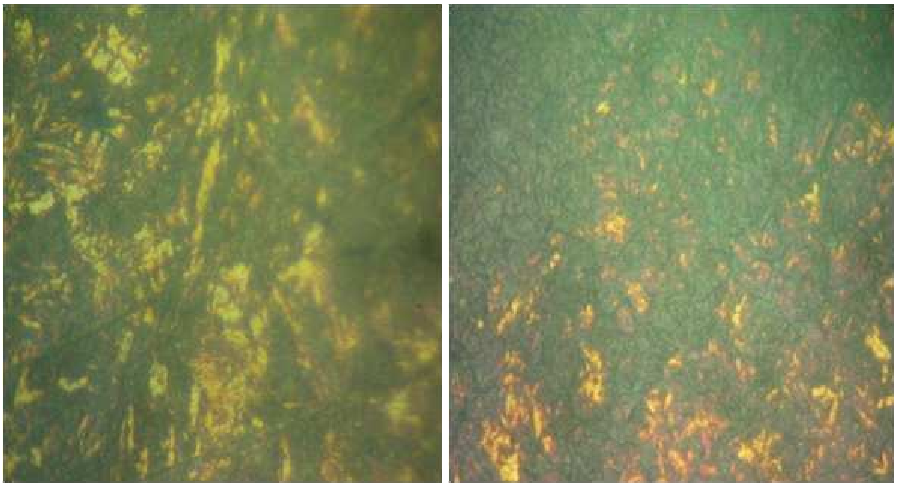
(a) (b)

Figure 6: (a) Micrograph of steel carburized using *prosopisafricana* seed pod only at 950 ^{o}C for 1hour, and (b) Micrograph of steel carburized using *prosopisafricana* seed pod and mussel shell waste mixture at 950 ^{o}C for 1 hour (plate martensite, dark patches and carbide, white patches) x400



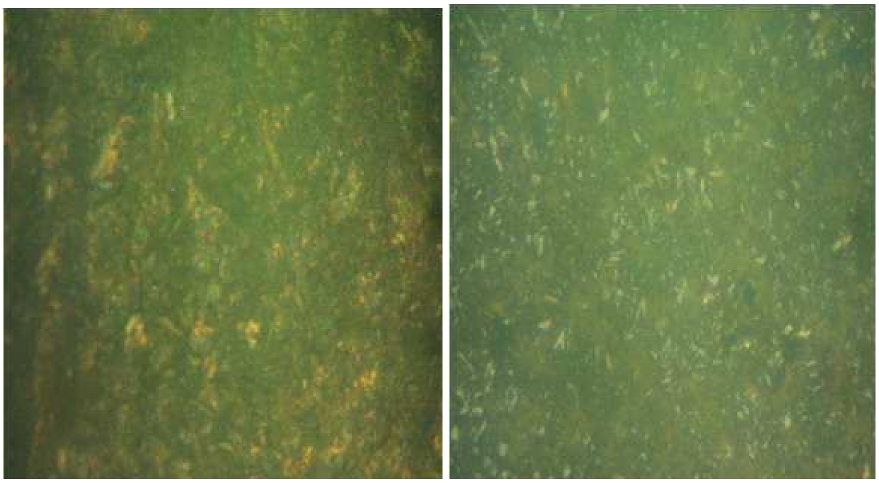
(a) (b)

Figure 7: Micrograph of steel carburized using *prosopisafricana* seed pod only at 950^{o}C for 2 hours, and (b) Micrograph of steel carburized using *prosopisafricana* seed pod and mussel shell waste at 950^{o}C for 2 hours, x400



(a) (b)

Figure 8: (a) Micrograph of steel carburized using *prosopisafricana* seed pod only at 950^{o}C for 3 hours, and (b) Micrograph of steel carburized using *prosopisafricana* seed pod and Mussel shell waste at 950^{o}C for 3hours (plate martensite, dark patches and carbide, white patches) x400



(a) (b)

Figure 9: (a) Micrograph of steel carburized using *prosopisafricana* seed pod only at 975^{o}C for 1 hour, and (b) Micrograph of steel carburized using 60 wt% *prosopisafricana* seed pod and 40 wt% mussel shell waste at 975^{o}C for 1 hour (plate martensite, dark patches and carbide, white patches) x400

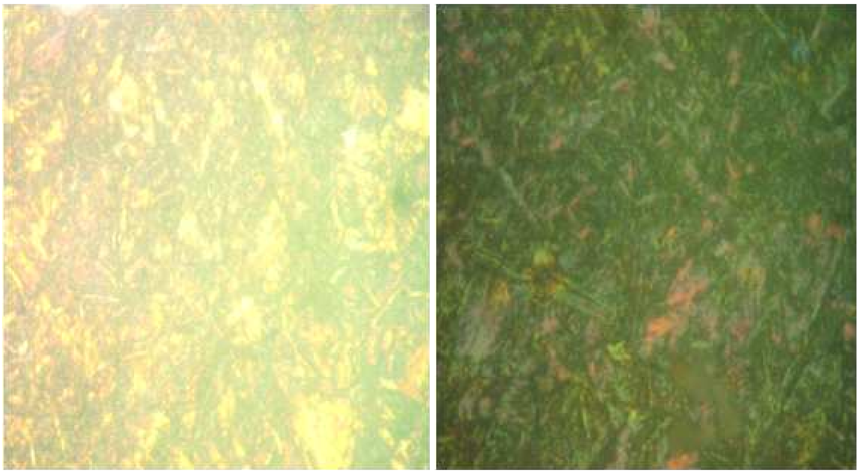


Figure 10: (a) Micrograph of steel carburized using 100 wt% *prosopisafricana* seed pod at 9750^{o}C for 2 hours, and (b) Micrograph of steel carburized using 60 wt% *prosopisafricana* seed pod and 40 wt% mussel shell waste at 975^{o}C for 2 hours. (Plate martensite, dark patches and carbide, white patches) x400

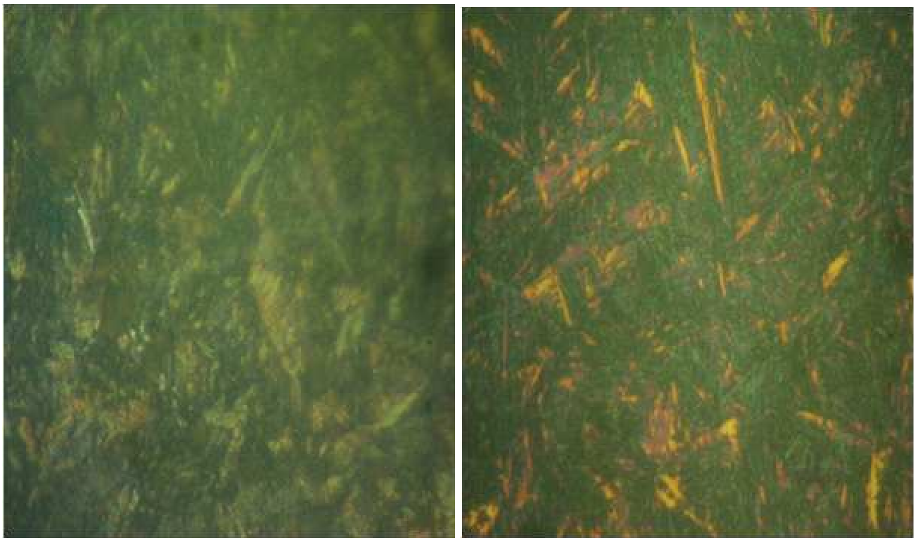


Figure 11: (a) Micrograph of steel carburized using 100 wt% *prosopisafricana* seed pod at 975^{o}C for 3 hours, and (b) Micrograph of steel carburized using 60 wt% *prosopisafricana* seed pod and 40 wt% mussel shell waste at 975^{o}C for 3 hours. (plate martensite, dark patches and carbide, white patches) x400

CONCLUSIONS

From the evidence gathered from the investigation the following conclusions have been drawn:

1. The study observed that the surface hardness of all the carburized steel samples' progressively increases as the carburizing temperature and holding time increased. This is a clear demonstration that the carburizing temperature and holding time had significant effects on the rate of liberation and absorption of carbon into the steel surfaces.
2. It was also observed that the carbon concentration at the carburized steel surface layer affected the morphology of the martensite formed.
3. The study also indicated that the samples carburized with *prosopisafricana* seed pod and mussel shell waste exhibited higher surface hardness values than those carburized with *prosopisafricana* seed pod waste alone.
4. The study have also revealed that *prosopisafricana* seed pod waste used as carbonaceous agent in the carburizing treatment has promising carbon potential as reflected by the hardness values of the samples carburized with it alone.
5. Finally, the study has unveiled a new opportunity for the utilization of mussel shell waste as an effective and alternative energizer in the carburization of mild steel; which before now is often discarded as waste material after the consumption of its meat.

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