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Oinu Timothy^a, D.T. Gundu^b, G. B. Nyior^c



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Oinu Timothy^a , D.T. Gundu^b , G. B. Nyior^c

^a Department of Mechanical Engineering, university of Agriculture Makurdi, Nigeria

^{bc} Department of Mechanical Engineering University of Agriculture Makurdi, Nigeria

Corresponding authors email: timoinu@gmail.com

Abstract

Purpose: In this study, selected calcareous wastes (cow bone and snail shell) were evaluated for use in surface carburizing of low carbon steel.

Methodology: Pack carburization process was used for the surface hardening of mild steel at 800 °C as the carburizing temperature. The soaking period of 2 hours was used and water was used as quenching medium. The specimens were subsequently tempered at 500°C. Mechanical tests such as hardness, impact, microstructural and wear resistance were carried out on the samples using standardized methods.

Results: The results obtained showed that the sample carburized with snail shell gave a higher hardness value than sample carburized with cow bone and untreated sample. In the charpy impact test carried out, it was observed that the sample carburized by snail shell has higher energy value than sample carburized with cow bone. For microstructural analysis test, it was seen that sample carburized by cow bone gave equiaxed and finer grains than sample carburized with snail shell and untreated sample, and for wear rate test, sample carburized by snail shell, has a better wear rate than sample carburized by cow bone and untreated sample.

Keywords: *Carburization, Calcareous wastes, Hardness, Impact, Wear, Untreated, Soaking, Quenching, Microstructure*

INTRODUCTION

Steel is essentially an alloy of iron and carbon containing up to roughly 2.0% carbon (Higgins, 1983; Shrager, 1961). By varying the carbon content and the heat treatment of the resultant alloy, enormous range of mechanical properties can be obtained. The addition of alloying elements such as nickel, chromium and molybdenum achieve further improvements in properties.

Mild steel contains 0.15 – 0.3% carbon. Steels used for sand castings usually contain 0.3 - 0.35% C, these are also considered as mild steel (Higgins, 1983; Shrager, 1961). These set of steels described above do not respond to hardening heat treatment, but only annealing treatment for grain refinement (Saita, 2008). However, given their wide area of application they do encounter service limitations, such as low hardness and wear resistance particularly when used as shafts and other rotating parts of machines. According to ASM, (1977, Iwata, 2008), to improve on the hardness and wear resistance several case hardening methods are normally used which include nitriding, carbonitriding, cyaniding and carburizing etc. The techniques involve the use of various raw materials for the impregnation of different elements on the surface of the mild steel to improve on the hardness and wear resistant properties which normally improve the service life of the part made of mild steel (ASM, 1977; Iwata, 2008).

Carburization is a heat treatment process in which iron or steel absorbs carbon liberated when the metal is heated in the presence of a carbon bearing material, such as charcoal or carbon monoxide, with the intent of making the metal harder. Depending on the amount of time and temperature, the affected area can vary in carbon content. Longer carburizing times and higher temperatures typically increase the depth of carbon diffusion. Carbonization is aimed at providing more carbon content at the surface than carbon content in the interior wall, so that the surface hardness increases. Carburizing can be done in four ways, namely pack carburizing, liquid carburizing, vacuum carburizing and gas carburizing (Heather, 2011).

When the iron or steel is cooled rapidly by quenching, the higher carbon content on the outer surface becomes hard via the transformation from austenite to martensite, while the core remains soft and tough with a ferritic and pearlite microstructure (Ober, et al, 1989). Carburizing is the addition of carbon to the surface of low carbon steels at temperatures within the austenitic region of the steel concerned, which generally is between 850°C and 950°C for mild steels. Within this temperature range, austenite which has high solubility for carbon, is the stable crystal structure. Hardening is accomplished when the subsequent high carbon surface layer is quenched to form martensite so that a high carbon martensitic case with good wear and fatigue resistance is superimposed on a tough core (Krauss, 1980).

The aim of this study is to evaluate cow bone and snail shell for surface treatment of low carbon steel.

MATERIALS AND METHOD

Materials that were used for the carburizing work consist of low carbon steel rods, carburizing media (cow bone and snail shell), carburizing boxes made of medium carbon steel, metal container to be used as bath for quenching and water as the quenchant. Equipment and tools consist of electric furnace, Vickers's hardness tester, metallurgical microscope with in-built camera and computer accessories, Charpy impact testing machine, tribometer, hand grinder, metaserv polishing machine.

Chemical composition of the low carbon steel sample

Analysis of chemical composition of low carbon steel used was done at Engineering Materials Development center in Akure in Nigeria.

Table 1. Chemical composition of the low carbon steel sample

Element	C	Si	S	P	Mn	Ni	Cr	Mo	V	Cu
Avg. Content	0.2427	0.2354	0.0379	0.0308	0.6970	0.0570	0.1223	0.0120	0.0022	0.1037
Element	W	As	Sn	Co	Al	Pb	Ca	Zn	Fe	
Avg. content	0.0005	0.0040	0.0095	0.0080	0.0032	0.0007	0.0000	0.0052	98.4279	

Test specimen preparation

Test specimen preparation implies cutting low carbon steel rods that were purchased from modern market in Makurdi and machining to standard test sample sizes. This was done according to America Society for Testing and Materials (ASTM) specifications on standard hardness, impact, wear and microstructural sample dimensions. Cow bone and snail shell, were collected from the surrounding compound and well sun dried. After sun drying, the samples were crushed into powder form using metallic mortar and pestle. They were then sieved out with a mesh sieve of about 600 μm . The carburizing boxes or metal boxes are made up of medium carbon steel material of thickness 4 mm and constructed using angle iron device. These materials are such that can withstand high temperature without melting.

Carburizing of low carbon steel samples

The metal boxes house the mild steel specimens and were heated with carbonaceous materials and covered with a lid and properly sealed with clay soil to prevent the carbon monoxide (CO) from escaping and to also prevent unwanted furnace gas from entering the steel boxes during heating.

The furnace temperature was adjusted to 800 °C and the loaded steel boxes were charged into the furnace. When the furnace reached the carburizing temperature, it is then held/soaked at the temperature for the required time (2 hours). After the materials were held at the time, the steel boxes were removed at various times from the furnace and the materials were quenched in water.

Tempering of the carburized samples

The carburized test samples were then tempered at a temperature of 500 °C for one hour and then cooled in air. After the cycles of heat treatment, the test samples were subjected to hardness test, Charpy impact test, microstructural and wear test

Proximate Analysis of cow bone and snail shell

Analysis of moisture, volatile matter, ash and fixed carbon content (estimated) of cow bone and snail shell. The samples were ground to pass through -72 mesh B.S. test sieve by the method given below.

Moisture determination

One gram each of air-dried cow bone and snail shell, were taken in a borosil glass crucible and then kept in the air oven maintained at the temperature 105 °C. The samples were soaked at this temperature for one hour and then taken out from the surface and cooled. Weight loss was recorded using an electronic weighing balance. The percentage loss in weight gave the percentage moisture content in the sample. Moisture content (Mc) = Loss in weight from 1g after heating at (105 °C).

Volatile matter determination

One gram of air-dried powdered samples each one of size -72 mesh were taken in a volatile matter crucible (made of silica) and kept in the muffle furnace maintained at the required temperature of (170 - 180 °C). The samples were soaked at this temperature for seven minutes and crucible was taken out from the furnace and cooled in air. Weight losses in the samples were recorded by using an electronic weighing balance. Volatile matter = weight after moisture content minus (-) weight after heating for volatile matters temperature (170 – 180 °C).

Ash determination

One gram each of air-dried powdered samples of size -72mesh were taken in a shallow silica disc and kept in the furnace maintained at the temperature of 500 °C. The samples were kept in the furnace till complete burning. Weights of ash formed were noted down and the percentage ash content in the samples was determined. Ash = the remaining weight after ashing at 500°C.

Estimated carbon/organic matter

Estimated carbon/organic matter = weight after volatile heating minus (-) ash weight

Table 2: Proximate Analysis of Carburizing Media

Samples (g)	Moisture (%)	Volatile Matter (%)	Ash Content	Estimated Carbon	Total (%)
Cow bone	11.04	1.79	55.63	31.54	100
Snail shell	0.42	0.32	55.83	43.43	100

Hardness test:

The test was conducted on vicker's hardness tester with direct reading scale for each of the samples see plate 1 below for Vicker's hardness machine. The test was conducted three times and the average of all the reading was taken as the observed value in each case.

Charpy impact test

The Charpy specimen have diameter of 23 mm with length of 55 mm and contains a 60° V notch, 17.1mm deep with a 0.25 mm root radius. The specimen was supported as a beam in a vertical position and loaded behind the notch by the impact of a heavy swinging pendulum. The specimen was forced to bend and fracture at a high strain rate of the order of 1000 per second. The principal measurement from the impact test is the energy absorbed in fracturing the specimen. The energy absorbed in fracture, usually expressed in joules, was read directly from a calibrated dial on the impact tester. The specimen is prepared in accordance with B. S. 131: part 1, 2 and 3 for impact test as shown in plate 2.

Microstructural analysis test

Microstructural examination of as received and carburized samples was carried out. Each of the samples was subjected to grinding with 200, 320, 400 and 600 grits silicon carbide abrasion paper and polished. The surfaces of the polished samples were etched in 2% nital by swabbing the surface with cotton wool soaked in the etchant. The microstructural examination of the etched surfaces of the specimens were made under a metallurgical microscope with an inbuilt camera through which the resulting microstructures were all photographically recorded. The setup is shown in Plate 3

Wear test

The machine used for this test is called Tribometer. Tribometer is an instrument that measures tribological quantities, such as coefficient of friction, friction force and wear volume between two surfaces in contact. Dutch scientist tribotester is the general name given a machine or device used to perform tests and simulations of wear, friction and lubrication which are the subject of the study of tribology. The setup is shown in Plate 4.



Plate 1: Vicker hardness testing machine Plate 2: Charpy impact testing machine



Plate 3: Metallurgical microscope with inbuilt camera and computer accessories



Plate 4: Tribometer for wear test

RESULTS AND DISCUSSION

Results

Table 3. Result of hardness test

Sample	Average hardness value (Hv)
Cow bone	108.92
Snail shell	138.8
Untreated	64.68

Table 4. Charpy impact test

Sample	Initial Energy (E1)	Residual Energy (E2)	Absorbed energy (E1 – E2) J
Cow bone	300	262	38
Snail shell	300	252	48
untreated	300	243	57

Microstructural Test Result

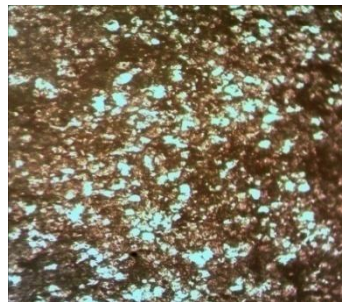
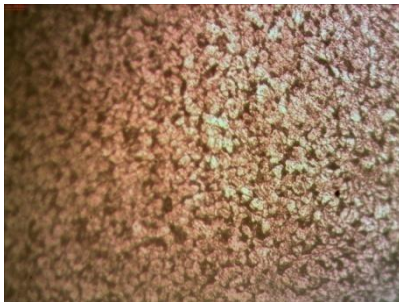


Plate 5. Cow bone

plate 6. Snail shell

plate 7. Untreated

Table 5 wear/abrasion test result

Sample	Wear rate (MM ³ /N/M)
A. Cow bone	0.01772
B. Snail shell	0.01672
C. Untreated	0.02735

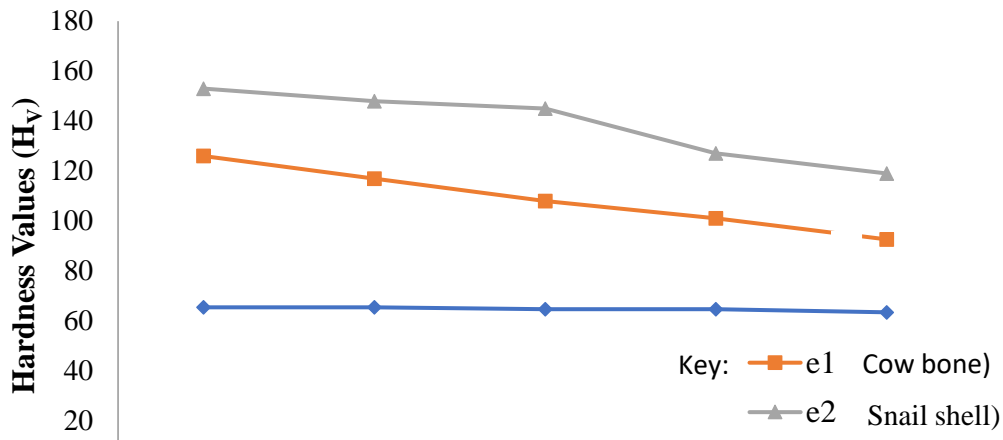


Figure 1. Hardness Values at Various Depths for Specimens carburized with Cow bone and Snail shell and untreated specimen.

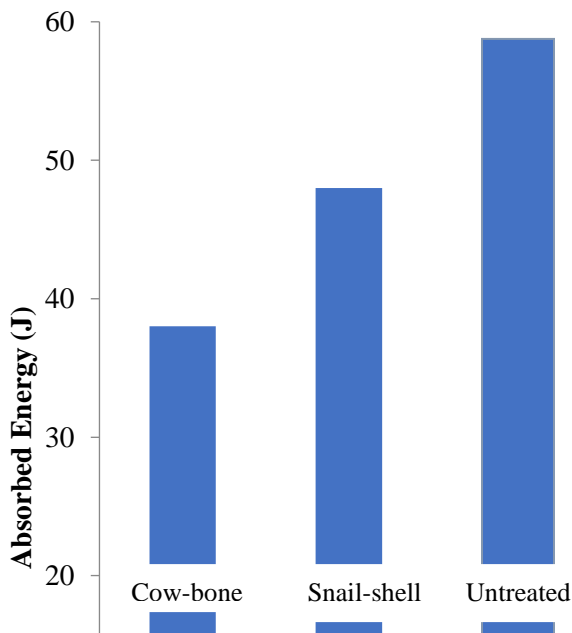
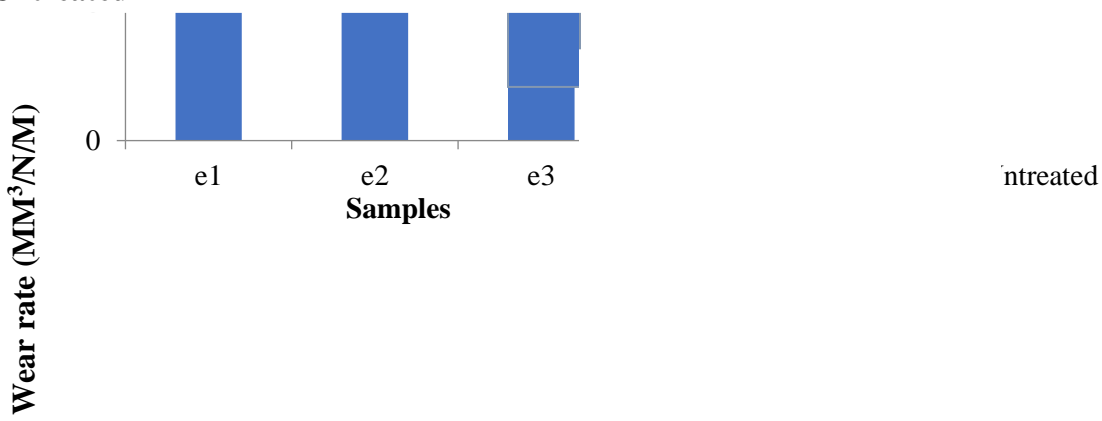


Figure 2: Impact Values for Specimens Carburized with Cow-bone, Snail-shell and Untreated



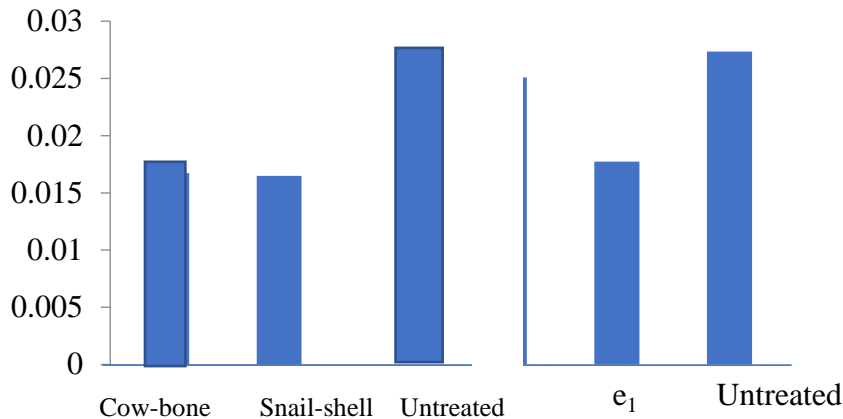


Figure 3: Wear rate of samples carburized by cow-bone, snail-shell and untreated

DISCUSSION

From the result obtained in table 3 and figure 1 from carburized samples and untreated, it was observed that sample carburized by snail shell has the highest hardness value of 138.8 Hv, followed by the hardness of sample carburized with cow bone with the value of 108.92Hv. The hardness value of the untreated is the least among the hardness values with the value of 64.68Hv. This explained that snail shell is a better efficient carburizer among the both calcareous carburizers used and has the better diffusion or penetration into the low carbon steel. The carbon deposited on the surface of the steel is dissolved by the austenite phase of the steel and diffuses into the steel which on subsequent quenching in water develops a hard case (Ihom, 1991 and Higgins, 1983).

The essence of carrying out the Charpy impact test is to determine the level of fracture toughness each carburized sample possesses. From table 4 and figure 2, the untreated specimen has the highest energy value/resistance to fracture of 57J. This is in contrast with the report of Agbeeze (1979) that the fracture strength of a metal increases with its hardness. Due to the refinement of the case and the core of low carbon steel with the two carburizers, it was observed that sample carburized with snail shell has higher energy value /resistance to fracture of 48J, while sample carburized with cow bone has energy value of 38J. The low hardness and impact values may be attributed to some high retained austenite occurring on surface hardening. This retained austenite can cause brittleness and loss of strength or hardness (Rajput, 2006).

From plate 5 and 6, it was observed that there were microstructural changes associated with the carburization process at the case area of the low carbon steel with the two plates while there is no change in the untreated specimen (plate 7). Plate 5 has equiaxed grains with pearlite phases dispersed in the ferrite matrix in a random pattern. In the plate 6, the sample was not well refined because of the appearance of large grains which might lead to low mechanical properties. Plate 7

is the microstructure of the untreated (as received) mild steel specimen showing pearlite structure in a ferritic matrix.

From table 5 and figure 3 the uncarburized (as received) sample has the highest wear rate of $0.02735 \text{ MM}^3/\text{N}/\text{M}$ compared to other samples that were carburized by cow bone and snail shell. Sample carburized by snail shell has a lesser wear rate of $0.01672 \text{ MM}^3/\text{N}/\text{M}$ while sample carburized by cow bone has wear rate of $0.0177 \text{ MM}^3/\text{N}/\text{M}$.

CONCLUSION

Evaluation of cow bone and snail shell Wastes for surface treatment of low carbon steel has been carried out and the following conclusions drawn from the work:

- 1 The sample carburized using snail shell as carburizer has the higher hardness value compared to the sample carburized using cow bone and untreated sample.
- 2 The impact resistance of untreated sample has higher value compared to samples carburized by cow bone and snail shell and can be attributed to retained austenite in carburized samples
- 3 The sample carburized using snail shell has a higher impact resistance than sample carburized with cow bone.
- 4 The sample carburized using cow bone has a better refined microstructure than sample carburized using snail shell and untreated
- 5 Sample carburized by snail shell has a lower wear rate than sample carburized by cow bone and untreated sample.

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