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Effect of walnut shell powder on the mechanical properties of case-hardened steel

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Abstract

In recent times, most previously-considered agricultural wastes are now gaining importance in the enhancement of some engineering product. One of such cases is in *case hardening* of steel surface. These works focused on the use of walnut shell powder to case harden steel. Pretreated and as-received powder was used in the experiment at 850^{o}C and varied soaking times of 45, 60 and 70 minutes designated as A, B and C respectively. Uniaxial tensile test method was adopted to characterize the tensile properties and the fracture toughness, while metallurgical microscope was used to view and analyze the microstructure of the samples. From the result, Sample A which was case hardened in pretreated walnut shell powder at 850^{o}C and soaked for 45 minutes was observed to have the best combined improved properties with a uniquely reduced and more compacted grain size at the edge of the microstructure.

Keywords: Walnut shell, Case hardening, Steel, Heat treatment, Microstructure, Soaking time

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1. Introduction

Recently, steam and chemical activation techniques are currently adopted in the production of activated carbon. The former require the introduction of steam between temperature ranges of 600 – 1200^oC while the latter involved the use of impregnated material with an agent as a starting material prior to blending of both at elevated temperature (Lillo-Rodenas et al., 2003; Patil et al., 2013). Both activation methods yields 30-35 % of activated carbon on average, but in chemical activation yield is normally higher than physical method – based on weight of starting material (Gonza et al., 1997; Evans et al., 1999).

Walnuts has been found to exhibit high nutritive value and characteristic health promoting components enriched in omega-6 and omega-3 polyunsaturated fatty acids (PUFA) (Cemile et al., 2012). These PUFA are essential dietary fatty acids (Amaral et al., 2003). Although, activated carbon have been observed to have large surface area, the absorption selectivity is limited because it is based on the shape and size of molecules. An increase in selectivity and hence a narrower pore size distribution is highly desirable for separation purposes. This can be achieved by tailoring the preparation conditions. The product obtained behaves as molecular sieves and are referred to as carbon molecular sieves (Martinez et al., 2003).

Walnut shells have also find usage in the production of natural dies (Mohammad and Loghman, 2013) and in the removal of lead from aqueous solution (Radka et al., 2013). Active (activated) charcoals are carbon adsorbents commonly used in industry for the sorption of various substances from gas atmospheres and liquids. Thus, the use of activated charcoals is of considerable interest in the development of non-waste technologies and environmental improvement (Kambarova and Sarymsakov, 2008). This is aside other raw materials used for the manufacture of activated charcoals (Martınez et al., 2006).

The process of hardening the surface of metal is called *surface hardening* or *case hardening*. This technique has been used to improve the mechanical properties of low carbon steel which generally classified as non-hardenable steel using the conventional heat-treatment. These techniques which have been in operation for ages now involve essentially the addition of carbon at the surface of low carbon steels at appropriate temperatures.

Several researchers have been working tirelessly in optimizing the use and relevance of agricultural waste converting it from waste to worth. Some of these include Ohize who worked on the effects of local carbonaceous materials on the mechanical properties of mild steel. The study experimentally determined the effect of coal, bone charcoal and wood charcoal on the hardness, tensile and impact strengths of mild steel. It was observed that bone and wood charcoal as carburizing materials each had considerable increasing effect on some of the mechanical properties (hardness and tensile strengths) with wood charcoal having greater effect and coal having the least effect on hardness, tensile strengths (Ohize, 2009). The wear resistance is also observed to be enhanced (Jaykant, 2009). Alagbe (2011) also studied the effects of some carburizing media on surface hardening of mild steel. The outcome of the research indicate that all the carburizing media yielded well to all surface hardening with palm kernel shell giving the highest hardness value.

Using pulverized bone, Aramide and associates studied the pack-carburization of mild steel where it was observed that the mechanical properties of mild steels are strongly influenced by the process of carburization, carburizing temperature and soaking time at carburizing temperature (Aramide et al., 2009).

Ihom and associates (Ihom et al., 2013) investigated the use of Egg Shell Waste as an Enhancer in the Carburization of Mild Steel and found that the carbonaceous organic waste improved the case hardness of the steel. However, the use of walnut shells powder as carburizer in the enhancing some mechanical properties of mild steel has not been extensively researched into. This serves as an impetus to the adoption of this heat treatment technique to carburize the skin of mild steel using agricultural waste (walnut shell) with the sole aim of improving on the inherent mechanical property thus expanding its scope of application.

2. Materials and method

The main material used in this research is the walnut sourced from its tree found at Itaogbolu, Ondo State, Nigeria. It was segmented into two part of where was pretreated and the other half left as collected, it was subsequently cleaned, break to remove the shell which was further dried and grinded with a roller mill and further pulverized with a mini-pulveriser. The product was passed through sieves to obtain fine particle size of 36 microns. This product serves as the starting material - carburizer. Structural steel was procured and characterized to determine the chemistry using spectrometer as shown in the Table 1. It was subsequently machined to different configuration taking into consideration the predetermined properties to be studied (tensile test, impact, hardness, fracture toughness and microstructural evaluation).

The pulverized shell is heated in a muffle furnace alongside the machined samples, heated to austenitic temperature and cooled at different time of 45, 60 and 75 minutes prior to its characterization. This variation is necessary in order to investigate the depth of penetration of the activated carbon. The samples are designated as shown in Table 2.

Table 1. Spectrometric analysis of the steel used in the experiment

Elements	Composition (wt. %)
C	0.308
Si	0.289
Mn	0.3465
S	0.0485
P	0.0435
Cr	0.166
Ni	0.071
Cu	0.194
Nb	0.0001
Al	0.0001
B	0.0006
W	0.0001
Mo	0.0001
V	0.0001
Ti	0.009
Fe	Bal.

Table 2. Sample designations during experiment

Sample Designation	Features
A1	Pretreated/850°C/45mins
B1	Pretreated/850°C/60mins
C1	Pretreated/850°C/75mins
A2	As-Received/850°C/45mins
B2	As-Received/850°C/60mins
C2	As-Received/850°C/75mins

3. Results and discussion

3.1. Results

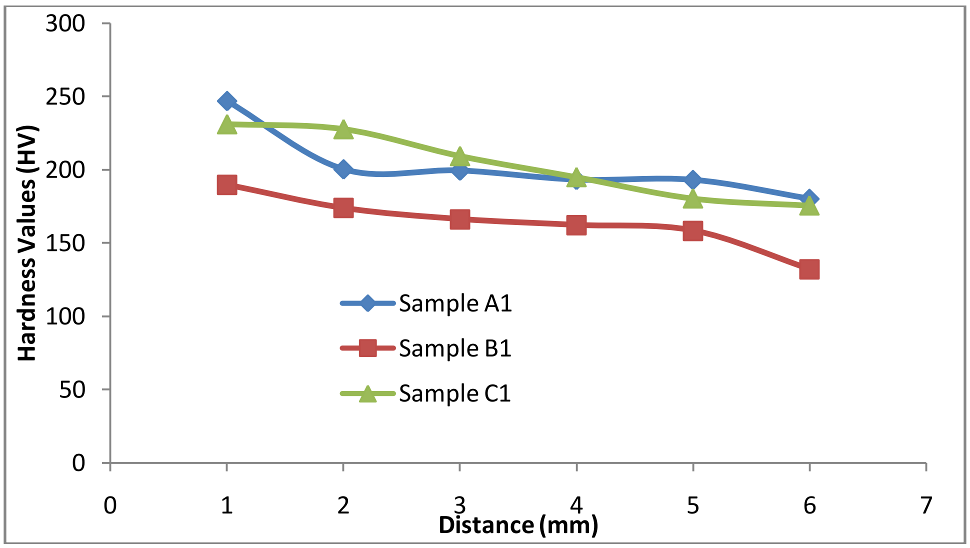


Figure 1. Variation of Hardness to distance after case hardening in a pre-treated walnut shell

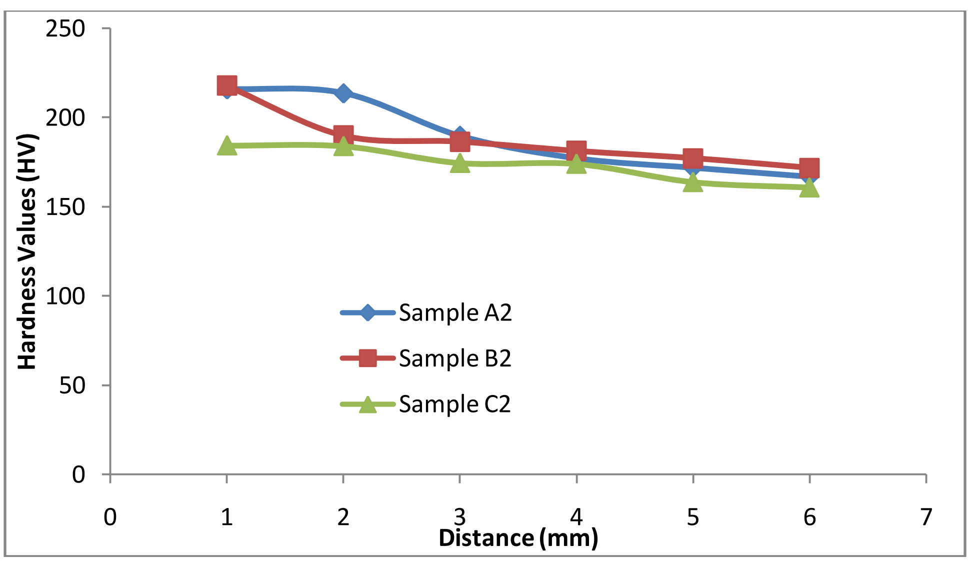


Figure 2. Variation of Hardness to distance after case hardening in as-received walnut shell

Table 2. Summary of mechanical properties of the case hardened steel

Sample	Eng UTS (MPa)	Peak Disp. (mm)	Strain-to-Fracture	Eng Slope MPa	Work done Nmm	K_{1C} MPa/mm$^{1/2}$	Impact (J)
A1	596.1	0.2684	0.3358	3087.9	33267.9	172.38	11.55
B1	506.1	0.2661	0.3304	2725.4	35685.4	120.96	16.97
C1	600.8	0.2358	0.2946	3082.7	41391.7	120.96	13.87
A2	596.1	0.2684	0.3358	3087.9	33267.9	169.69	10.90
B2	595.5	0.2848	0.3726	2862.3	41808.9	150.25	8.84
C2	596.1	0.2684	0.3358	3087.9	33267.9	172.20	6.53

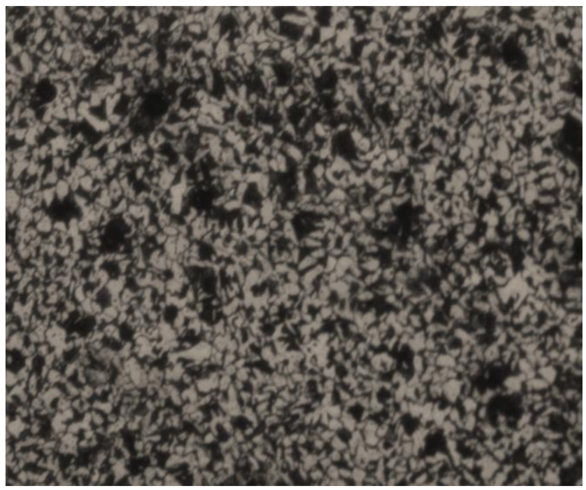
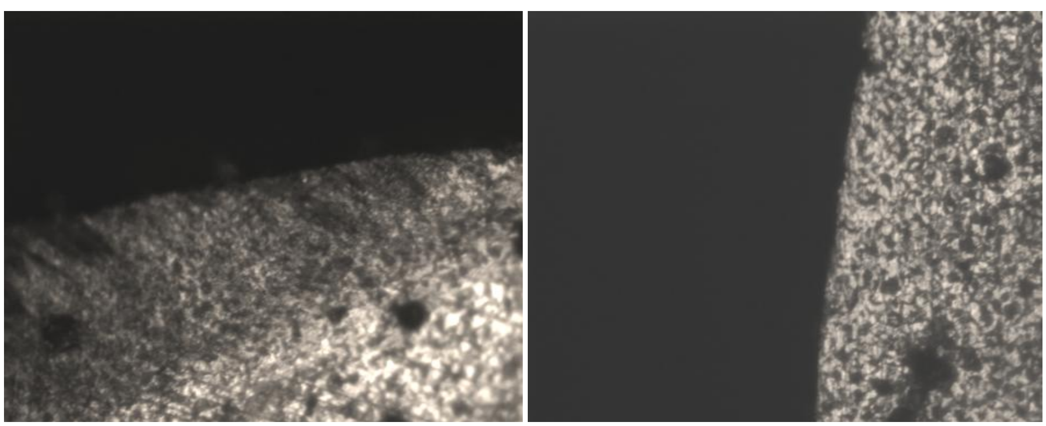
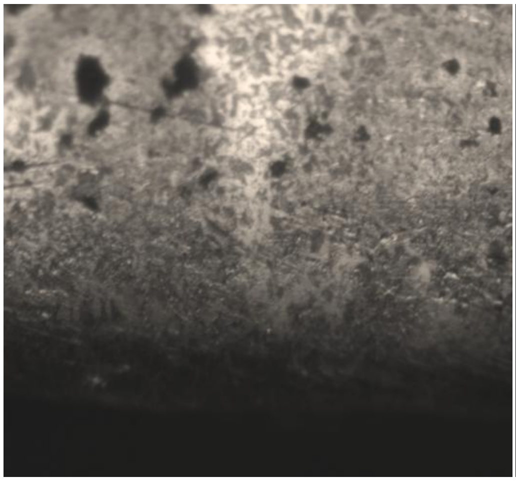
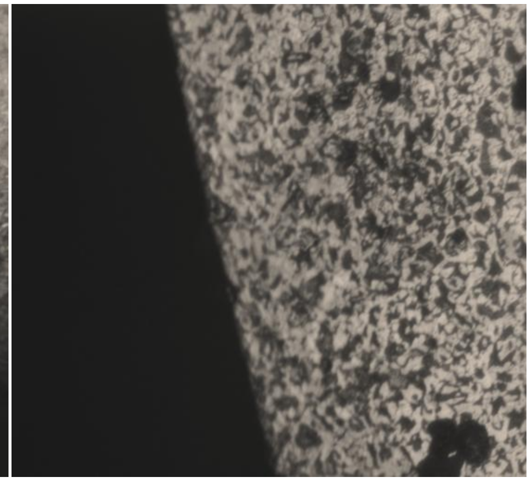


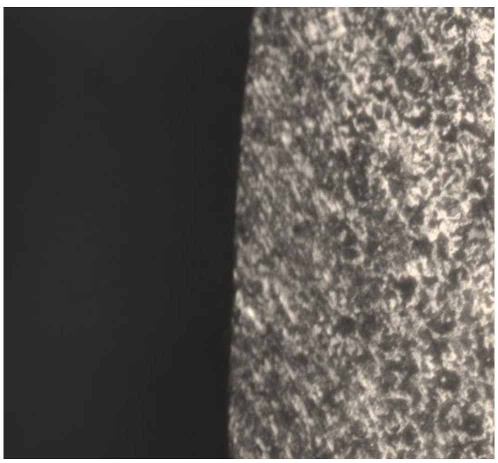
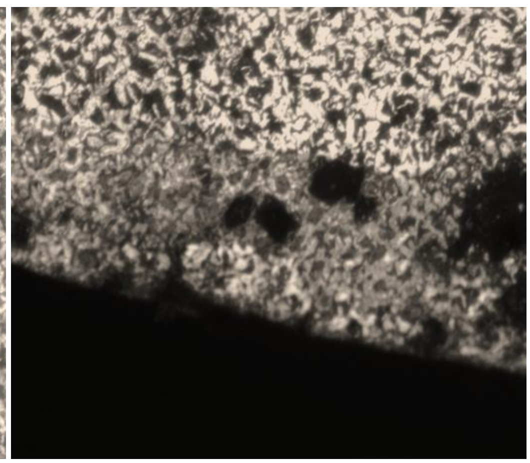
Plate 1. Photomicrograph of as-received steel prior to heat treatment (Mag. 100x)



(a) Sample A1 (b) Sample B1

(c) Sample C1 (d) Sample A2

(e) Sample B2 (f) Sample C2

Plate 2. Photomicrograph of treated steel showing different depth of carbon diffusion

3.2. Discussion

3.2.1. Mechanical properties

3.2.1.1. Hardness Behavior

Figure 1 and 2 shows the hardness property of the samples when hardened in a pre-treated and as-received walnut shell. The hardness test was conducted along a trend from the surface (case) of the core at a regular interval of 1mm. the plot indicates that the case of the sample possess a distinctively high hardness value and subsequently decreases with respect to distance through the core. The high hardness observed at the case is due to the presence of the carbon in the pulverized walnut shell which diffuses into the skin thus reacting with the inherent iron to form iron carbide which is a hard phase.

There is however, a variation in the plots explaining the hardness properties of the samples. When the carburizer was pre-treated prior to its usage in the hardening process, a relatively high hardness was observed especially more noticeable in sample A's and C's. Sample A1 was observed in total to exhibit the highest hardness value of 247.1HV while its corresponding value in the as-received shell displays 213.7HV. The reason for the high hardness in the pre-treated shell could be attributed to the initial carbon activation prior to usage as hardener.

3.2.1.2. Tensile Behavior

Table 2 show the summary of the mechanical properties of the case hardened steel. Uniaxial tensile testing technique was adopted to characterize the tensile properties; some of the properties characterized are the engineering ultimate tensile strength (UTS), peak displacement and the strain-to-fracture. Circumferential notch technique (CNT) was adopted in the characterization of the fracture toughness of the steel after treatment. This methods were as found in literature review (Dieter, 2004; Alaneme, 2011; Momoh, 2012).

From the data shown (See Table 2), the UTS were observed to be closely related with sample C1 exhibiting the highest value of 600.8MPa, the pre treatment the walnut shell was subjected to still account for the relatively high tensile strength. Moreover, the set of samples heat treated in as-received walnut shell display almost constant values in UTS and negligible differences in the peak displacement and strain-to-fracture regardless of the soaking time. Thus indicating that case hardening of steel does not take soaking or holding time into consideration.

The fracture toughness values however, was found to range between the value of about 150 – 172MPa/mm$^{1/2}$. Sample A1 and C2 were observed to posses the highest toughness value, thus explaining the minimum stress value it could accommodate prior to crack propagation which could leads to failure.

3.2.1.3. Impact Behavior

The impact analysis still supports the observation that the pre-treated shell has much effect on some mechanical properties when used to case harden steel. Sample B1 was observed to possess the optimum impact value of about 17J while sample C2 subjected to as-received walnut shell was observed to possess the least value of 6.53J.

3.2.2. Microstructural examination

Plate 1 shows the initial microstructure used in these experiment, it consist basically of ferrite (grey) and pearlite (dark) phase, the volume fraction of which confirmed it to be normalized medium carbon steel. This operation is initially carried out to annul its mechanical history introduced during machining. All micrographs in this work are viewed at various magnifications; for comparison sake however, all micrographs shown here are selected to be consistently x100.

The edges of all the structures are shown in Plate 2. General observation shows microstructural orientation in the entire sample at different degree though. Sample A (See Plate 2(a)) case hardened in pretreated walnut shell powder at 850°C and soaked for 45 minutes was observed to show a uniquely reduced and more compacted grain size at the edge. This is as a result of the carbon diffusion from the carburizer into the surface of the sample, thus, accounting for its unique improved mechanical properties as earlier discussed. The concentrated dark portion of the structure could be graphite concentration which could not distribute itself uniformly due to the predetermined insufficient soaking time used.

4. Conclusion

These works focused on the use of walnut shell powder to case harden steel. Pretreated and as-received powder was used in the experiment, and varied heat treatment temperatures and soaking time was adopted. Uniaxial tensile testing technique was used to characterize the tensile and fracture toughness (CNT) properties, while optical microscope was used to view the microstructure prior to its analysis. From the result, Sample A which was case hardened in pretreated walnut shell powder at 850°C and soaked for 45 minutes was observed to have the best combined improved properties with a uniquely reduced and more compacted grain size at the edge of the microstructure when viewed and analyzed.

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Appendix

Figure 3. Walnut tree species found at Itaogbolu, Ondo State, Nigeria

Figure 4. Selected walnut shells prior to subsequent treatment