The Suitability of Seashell, Animal Bone and Sodium Carbonate as Energizers in Case Carburization of Mild Steel

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Abstract: This work examines the suitability of using seashell (Oyster shell), animal bone and Na₂CO₃ materials as energizers for case hardening of mild steel. A carburizer consisting of charcoal was used for research with sea shell, animal bone and Na₂CO₃ as energizers. Samples were carburized in fabricated rectangular stainless steel boxes using different percentages of energizers (10, 20, 30, 40, and 50%) respectively. The samples were covered completely in each of the boxes with the mixture of carburizer and energizer placed in the chamber of the furnace. The process was carried out at carburizing temperature of 950°C, soaked for 4, 6, and 8hours and quenched in oil. Twenty samples were further tempered at 200°C for 1hour to relieve the stress built up during quenching. Hardness test, chemical analysis and impact test were carried out on the samples. The hardness values of the carburized mild steel were measured with a micro hardness tester. The results of the study showed that hardness values of the untempered mild steel samples were slightly improved than the tempered samples at carburizing temperature of 950°C in seashell energizer for 8hours have the highest impact values of 184 Joules for the tempered samples which are higher than the untempered samples due to increase in toughness resulting from tempering. The results also showed that seashell and animal bones are potential energizers in case carburization of mild steel.

Keywords: Carburizer, energizer, carburizing time, carburizing temperature, hardness, impact.

1. INTRODUCTION

Surface degradation by wear and corrosion is one of the complications associated with ageing facilities and components especially under some service environments. In today's competitive global market, surface engineering offers industries improve performance of engineering components, longer component life and failure prevention. The overhaul condition of many steel components such as gears, shafts, and cams, gears make it essential for them to possess both wear-resistant and hard surfaces at the same time with shock- resistant and tough cores. These two different sets of properties exist only in alloy steels. Higgins [1] reported that low carbon steel, containing about 0.1%C, will be tough and soft, while a high carbon steel of 0.8%C or more will be hard and brittle. However, all steel contain small amounts of carbon and manganese i.e. steel is a crystalline alloy of iron, carbon and several other elements, which hardens above its critical temperature. Steel has a carbon content ranging from 0.2 to 1.5%.

Mild steel, by reasons of its dominance and workability among the classes of steel [2], has found a broad application in the production of engineering components like cams, gears, shafts, pinions, keys, hand tools, agricultural equipment, etc. These components require the mechanical properties of impact strength, hardness and tensile strength for their safe and durable purposes.

Many different types of heat treatment processes are used to modify the surface and structural properties of engineering components as reported by Child [3]. Among the more important of these treatments are heat treatment processes such as induction hardening, case carburizing, and immersion hardening as studied by Child [3]. Various forms of steel are used in the manufacture of both major and minor machine parts. Bolts, cams, screw and cutting tools are among the essential engineering components usually made out from steel. The diffusion of carbon to the surface of low-carbon steels at temperatures within the austenitic region of the steel concern is called carburizing, which usually is between 850°C and 950°C for low carbon steels. The resulting interstitial solid solution is harder than the base material, which improves wear resistance without sacrificing toughness [4]. A great concern in manufacturing environments is optimum structural material, where there is demand for high performance in mechanical properties such as hardness. If the temperature of carburization is between 900°C and 950°C as is usually the practice – then the rate at which the case is produced depends on

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the characteristics of the carburizing compound employed.

The inward diffusion of carbon takes place at a rate which depends on the carburizing temperature, chemical composition of the carburizing mixture and chemical composition of the steel [5, 7, 9, 11]. For a successful carburization, a control of all these parameters; the soaking time, carburizing temperature, and chemical composition of the carburizing compound must be achieved [6, 8, 10].

[12-14] found that some carbonate salts when added to charcoal enhance their carburization process. These additives known as energizers are mainly carbonates of Barium (BaCO₃), Sodium (Na₂CO₃) and Potassium (K₂CO₃). Waste products like seashell, coconut shell and river clam shell from agricultural crops have been investigated for a possible use as energizers in case carburization of mild steel as reported by Ogo *et al.* [16]. Mild steel carburization rate was enhanced by sea shell addition (Oyster shell) to charcoal. Also, there was significant increase in the tensile strength of mild steel by the addition of seashell to charcoal as reported by Ogo *et al.* [17].

For some years, there has been interest in the study of process variables of metals during heat treatment as reported by [6, 7, 14, 15]. However, very few works has been done on process parameters during the surface hardening process as stated by aramide et al. [6] because parameters controlling carburization process is a multifaceted problem. The parameters majorly controlling the process of carburization are carbon potential, soaking time, quenching time and carburizing temperature as investigated by Aramide et al. [6]. Surface hardening processes are influenced by heat treatment, rate of heating and cooling, quenching media and temperature as investigated by Schimizu and Tamura [18]. Post heat treatment and pre-heat treatment processes are the major influencial factors, which affect the quality of the part surface hardened.

The aim of this research work is to examine the suitability of animal bones, seashells, and sodium carbonate as substitute energizers and by optimizing volume fraction of energizers and soaking time in enhancing the hardness and impact properties of mild steel applicable in gears, machines, springs, high strength wires and automobiles, by pack carburization.

2. EXPERIMENTAL PROCEDURE

A flat bar of mild steel obtained from Eldorado Steel Industry was analyzed at Nigerian foundry and its nominal chemical composition is given in Table **1**.

Pack carburizing process was carried out in a Muffle furnace. Seashell and animal bone were obtained and grinded in a ball milling machine into fine powder to increase the surface area. The prepared mild steel samples were embedded in the carburizing box, which were then tightly sealed with clay cover to avoid unnecessary gas furnace from inflowing the carburizing box during heating. The temperature of the furnace was attuned to the required temperature (950°C), and the loaded steel carburizing box was charged into the furnace. When the furnace temperature attained the required carburizing temperature, it was then held at the temperature for the required time (4, 6 and 8hours). The steel samples were soaked at the specified time, the steel carburizing box was removed from the muffle furnace and the samples were quenched in oil. The oil quenchant physical properties are shown in Table 2. The carburizing process was carried out in various batches in accordance with volume fraction set (100/0, 90/10, 80/20, 70/30, 60/40, and 50/50). The weight of the carburizer and energizer were calculated from the volume of the container used and a known weight of each of the carburizer and energizer was packed into the stainless steel box.

Twenty (20) of these samples were quenched and tempered at a temperature of 200° C for an hour to relieve internal stresses built up during quenching and to increase the toughness of the samples while the

Table 1: Chemical Composition of AISI 1018 Mild Steel (Eldorado Steel Industry, Lagos, Nigeria)

С	Si	Mn	Р	S	Cr	Ni	Cu	Sn
0.18%	0.215	0.51%	0.022%	0.005%	0.02%	0.02%	0.09%	0.0034

AI	Мо	Со	Nb	V	Fe
0.031	0.0043 0.0092		0.0047	0.0027	BALANCE

Characteristics	Values
Viscosity of cSt @ 40°C	14.0
Viscosity of cSt @ 100°C	3.2
Viscosity of SUS @ 100°F	74
Viscosity of SUS @ 210°F	37
Flash Point, ⁰ C/ ⁰ F	173/343
Ramsbottom carbon residue, mass %	0.2
Quench Time, seconds	
Nickel Ball	16
Chromized Nickel Ball	19

Table 2: Typical Characteristics of the Quenching Oil (As Specified by the Producer: Petro-Canada)

remaining twenty (20) samples were untempered after hardening. Micro-hardness and impact tests were carried out on the tempered and untempered carburized mild steel samples. For each of the sample, tests were carried out 3times, and the observed values were the average of all the samples taken.

Samples were prepared using modern Wet Grinding Machine. The samples were then pressed against the rotating emery paper with a stream of water acting as coolant and particles remover. Coarse grinding was done with emery papers of meshes 60, 120, 240 followed by fine grinding with 600 and 800 meshes. The ground and polished samples were etched in Nital solution for 20seconds.

3. RESULTS AND DISCUSSION

In Figure 2, at 4hrs and furnace temperature of 950°C, seashell and Na₂CO₃ energizers peaked at 50/50 volume fraction while animal bone peaked at 60/40. Seashell had the highest hardness value of 162HB, followed by 147HB and 139HB respectively by Na₂CO₃ and Animal bone. At 80/20 volume fraction, animal bone showed slight improvement above Na₂CO₃ but below seashell energizer. Moreover, the three energizers showed decrease in hardness values in subsequent volume fractions.

Figure **3** showed Na₂CO₃ with steady decrease in hardness values with increase in volume fractions till 90/10. After 6hrs, animal bone showed slight decrease with increase in volume fraction and rose sharply to peak at 90/10 at value of 145HB. Seashell on the other hand showed a sinusoidal decrease and increase in hardness values with increasing volume fraction. At soaking time of 6hours, seashell had the highest hardness value at 178HB while Na₂CO₃ displayed the lowest value of 118HB on untempered samples.

From Figure 4, after heat treating for 8hours with carburizing temperature of 950°C, seashell had a steady rise with peak of 179HB at 80/20 volume fraction and later showed slight decrease. Animal bone improved significantly at 50/50 but showed slight decrease with increase in volume fraction of energizer. Na₂CO₃ started slowly and peaked at 50/50 with significant decrease till 80/20 and rose slightly with increase in volume fraction.



(a)

Figure 1: a: Clay for sealing up the boxes. b: Samples removed from the box.

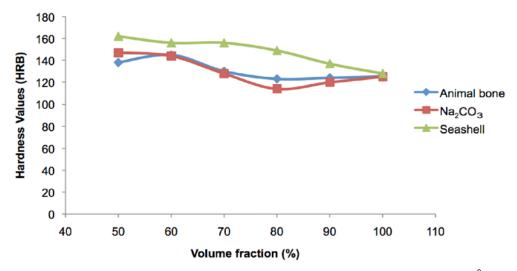


Figure 2: The relationship between hardness and volume fraction of different energizers after 4hrs at 950°C (untempered).

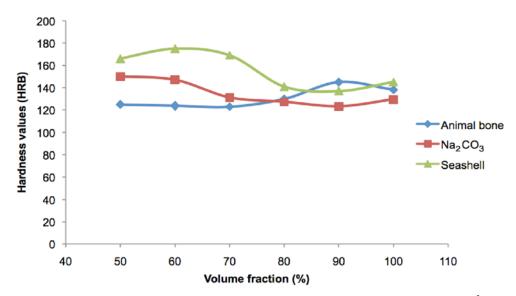


Figure 3: The relationship between hardness and volume fraction of different energizers after 6hrs at 950°C (untempered).

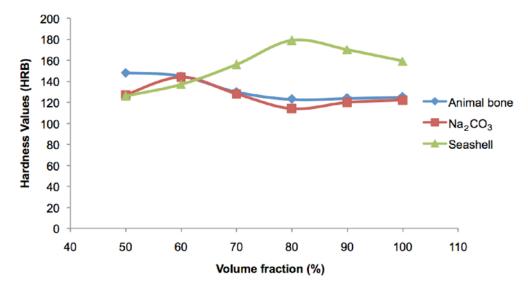


Figure 4: The relationship between hardness values and volume fraction of different energizers after 8hrs at 950⁰C (untempered).

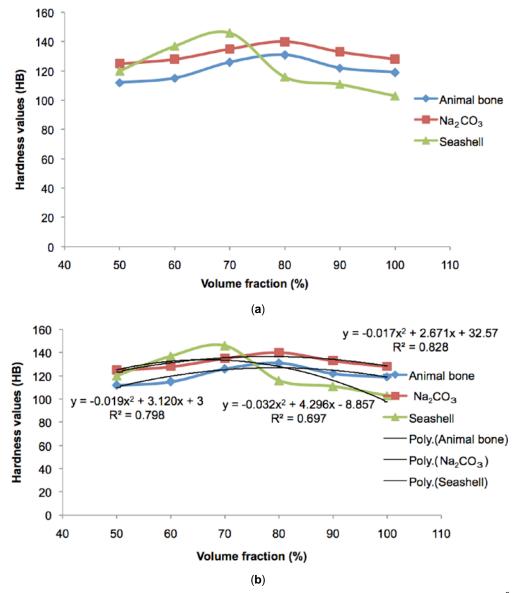


Figure 5: a: The relationship between hardness and volume fraction of different energizers after 4hrs at $950^{\circ}C$ (tempered). **b**: The Polynomial regression between hardness and volume fraction of different energizers after 4hrs at $950^{\circ}C$ (tempered).

From Figure 5, both Na_2CO_3 and animal bone maintained their steady increase with increase in volume fraction and they peaked at 80/20 with hardness values of 131HB and 138HB respectively. After 4hrs, Na₂CO₃ showed better values than animal bone at the same carburizing temperature but tempered. However, seashell maintained steady increase and peaked at 70/30 then showed significant decrease in hardness values with increase in volume fraction. Seashell recorded the lowest hardness value after 4hrs when the samples were tempered. Furthermore validation of the result shows that there exist polynomial relationships between the hardness values and the volume fraction (%) of energizers; i.e. R^2 shows 0.83, 0.80 and 0.70 respectively for Na₂CO₃, animal bone and seashell.

From Figure 6, after heat treating for 6hours at 950°C. the tempered steel samples in charcoal/seashell peaked and had the highest value of 163HRB at a composition ratio of 70%charcoal and 30% energizer. After that, it showed slight decrease at lower percentage addition of seashell. Both Animal bone and Na₂CO₃ performed at the same rate in the beginning but at 70/30 animal bone showed considerable increase more than Na₂CO₃ at subsequent volume fractions. It shows that seashell performed better than the other energizers after 6hrs. Furthermore validation of the result shows that there exist polynomial relationships between the hardness of tempered samples and the volume fraction (%) of the energizers; i.e. R² shows 0.83, 0.96 and 0.99 respectively for seashell, animal bone and Na₂CO₃.

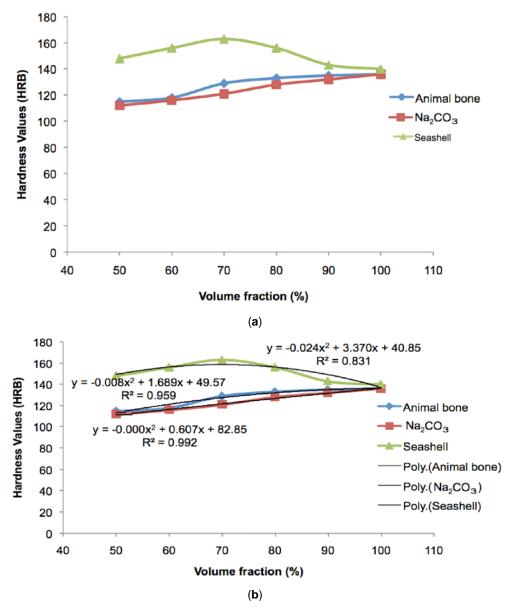


Figure 6: a: The relationship between hardness values and volume fraction of different energizers after 6hrs at 950^oC (tempered). **b**: The Polynomial regression between hardness values and volume fraction of different energizers after 6hrs at 950^oC (tempered).

From Figure 7, after heat treating for 8hrs, the three energizers showed steady rise from 50/50 volume fraction with animal bone peaking from the start till 60/40. After this, seashell rose steadily above the other energizers and peaked at 90/10 at hardness value of 163HB and later decreases slightly. Both animal bone and Na₂CO₃ maintained their steady increase with increase in volume fraction and they peaked at 80/20 with hardness values of 145HB and 135HB respectively. Comparing Figures 4 and 7, the hardness values of tempered samples reduced with increase in temperature. Through carefully controlled tempering treatment, the quenching stresses can be relieved and some of the carbon can precipitate from the supersaturated solid solution to a finely dispersed carbide phase. Furthermore, validations of the results show that there exist polynomial relationships between the hardness and the volume fraction (%) of the energizers i.e. R^2 shows 0.94, 0.83 and 0.83 respectively for seashell, animal bone and Na₂CO₃.

The Impact results shown in Figures **8**, **9** and **10** revealed that samples carburized at 950° C in seashell energizer for 8hours have the highest impact values of 184 Joules for the tempered samples which are higher than the untempered samples due to increase in toughness resulting from tempering. At soaking time of 8hours, the impact values increase with increasing volume fraction of the energizers. At 4 and 6 hours

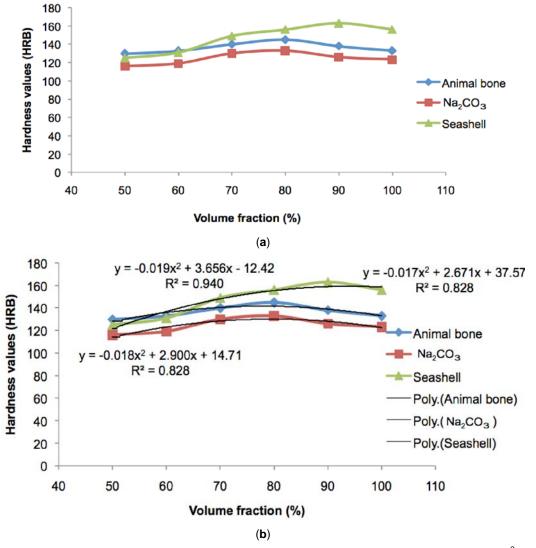


Figure 7: a: The relationship between hardness and volume fraction of different energizers after 8hrs at 950° C (tempered). **b**: The Polynomial regression between hardness and volume fraction of different energizers after 8hrs at 950° C (tempered).

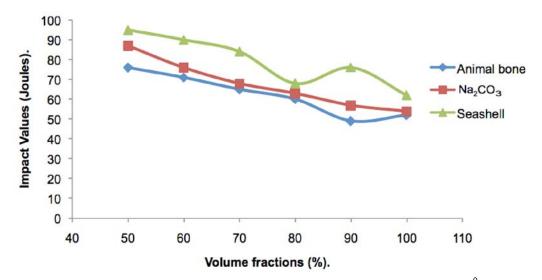


Figure 8: The relationship between impact and volume fraction of different energizers after 4hrs at 950°C (tempered).

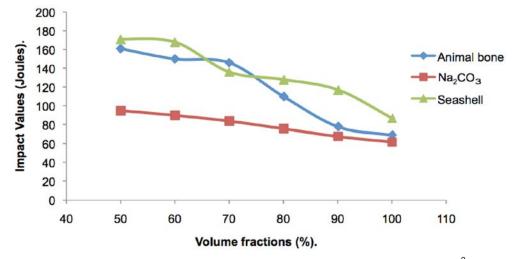


Figure 9: The relationship between impact and volume fraction of different energizers after 6hrs at 950°C (tempered).

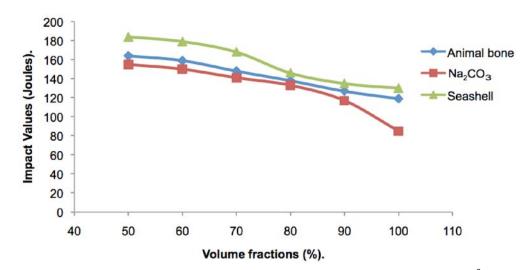


Figure 10: The relationship between impact and volume fraction of different energizers after 8hrs at 950°C (tempered).

soaking time, seashell had the highest impact values of 96 and 168Joules respectively which indicate that impact values increase with increasing soaking time as reported by Aramide *et al.* [6]. Animal bone energizer showed good quality by comparing favourably with seashell energizer at soaking time of 6 and 8hours.

From the above figures, the untempered samples have values which are superior to the tempered samples at 950°C. This is because tempering decreases hardness but increases toughness of samples as reported by Stephen and Edward [19].

4. CONCLUSION

In this study, pack carburization of mild steel using seashell, animal bone and Na₂CO₃ as energizers were examined at 950^oC. In conclusion, there is a significant increase in the carburization rate of low carbon steel by

the addition of seashell to charcoal than other energizers. This can be seen in the difference between the hardness values of carburized steel samples which had seashell, animal bone and Na_2CO_3 energizers added for equal holding time and quenching medium.

These can be deduced from the results obtained:

- 1. The hardness values of untempered samples improved moderately than the tempered samples at a temperature of 950°C and carburizing time of 4 and 8hrs.
- 2. Impact values increase with increasing soaking time.
- 3. Seashell and animal bone are potential energizers and they performed better than Na_2CO_3 at most carburizing time and temperature of 950^oC.

REFERENCES

- Higgins RA. Engineering Metallurgy. (Part 1, Applied physical metallurgy). 5th ed. Kent: ELBS; Edward Arnold Publishers Ltd. 1991.
- [2] Kanisawa HO, Koyasou YK. Development of high strength carburized steels for automobile gears. Nippon Steel Technical Report 1995; 65: 234-37.
- [3] Child HC. Surface Hardening of Steel. Oxford University Press, UK 1980.
- [4] Yang CF, Chiu LH, Wu JK. Effect of carburization and hydrogenation on the impact toughness of AISI 4118 steel. Surf Coat Technol 1995; 73: 18-22. http://dx.doi.org/10.1016/0257-8972(94)02357-3
- [5] Wang SW, Lin YC, Chen TM. A study on the behavior of hardened medium carbon steel. J Mater Process Technol 2002; 120: 126-32. http://dx.doi.org/10.1016/S0924-0136(01)01195-5
- [6] Aramide FO, Ibitoye SA, Oladele IO. Effects of carburizing time and temperature on the mechanical properties of carburized mild steel, using activated charcoal as carburizer. Mater Res 2009; 12(4): 483-87. http://dx.doi.org/10.1590/S1516-14392009000400018
- [7] Gupta RC. Effect of carburizing temperatures on the mechanical and wear behavior of mild steels. M.sc. thesis, National Institute of Technology Rourkela, India 2009.
- [8] Sarkar S. Two dimensional mathematical modeling in cylindrical coordinate for simulating the pack carburization process. ASM Int 2008; 39A: 46-48.
- [9] Muhammad HJ. Effect of various heat treatments on microstructure and mechanical properties of low carbon steel. ISIJ Int 2000; 34(3): 34-38.

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- [10] Atanda PO, Umoru LE, Adeyeye AD. Effects of carburizing variables on the case properties of C2R steel. J Miner Mater Charact Eng 2009; 8(2): 79-92.
- [11] Kumar MO. Studies on the abrasive wear of carburized mild steels. Trans Indian Inst Met 1994; 47: 417-20.
- [12] McGannon HE. The making, shaping and treating of steel, 9th ed., U.S. Steel publication, Pennsylvania, USA 1971.
- [13] Gulyaev A. Phys Metallur. Mir publishing company, Moscow, 1980.
- [14] Ohize EJ. Effects of local carbonaceous materials on the mechanical properties of mild steel. AUJ 2009; 13(2): 107-13.
- [15] Preciado MJ, Bravo PM. Effect of low temperature tempering prior cryogenic treatment on carburized steels. J Mater Process Technol 2006; 176: 41-44. http://dx.doi.org/10.1016/j.jmatprotec.2006.01.011
- [16] Ogo DUI, Ette AO. The use of river clam shell as an energizer in case carburization of mild steels. ISIJ Int 1994; 44(5): 865-68. http://dx.doi.org/10.2355/isijinternational.44.865
- [17] Ogo DUI, Ette AO, Iyorchir AI. Feasibility of sea and coconut shells as substitute to barium carbonate in small scale foundry and heat treatment shop in Nigeria. ISIJ Int 2007; 35(2): 203-209. <u>http://dx.doi.org/10.2355/isijinternational.35.203</u>
- [18] Schimizu N, Tamura I. An examination of the relation between quench-hardening behavior of steel and cooling curve in oil. Trans ISIJ Int 1978; 18: 445-50.
- [19] Steven MC, Edward LL. ASM Handbook-heattreating 1991; 4.

http://dx.doi.org/10.1590/S1516-14392009000400018