

**SUITABILITY OF SNAIL SHELL PARTICLES AS AN ENERGIZER  
FOR CARBURIZING MILD STEEL**

**BY**

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**APRIL, 2018**

## **DECLARATION PAGE**

I declare that the work in the Dissertation entitled: suitability of snail shell particles as an energizer for carburizing mild steel has been carried out by me in the Department of Metallurgical and Materials Engineering. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this project thesis was previously presented for another degree or diploma at this or any other institution.

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**Name of Student**

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**Signature**

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**Date**

## CERTIFICATION PAGE

This dissertation entitled: SUITABILITY OF SNAIL SHELL PARTICLES AS AN ENERGIZER FOR CARBURIZING MILD STEEL by IDACHABA JAMES ABARA meets the regulations governing the award of the degree of MSc. Metallurgical and Materials Engineering of the Ahmadu Bello University, and is approved for its contribution to knowledge and literacy presentation.

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## **DEDICATION**

To God, Kauna and Achenyo.

## ACKNOWLEDGEMENTS

I would first give all glory, honour and adoration to God Almighty for making it possible for the completion of this course despite all the challenges.

After an intensive period of seven months, today is the day: writing this note of thanks is the finishing touch on my dissertation. It has been a period of intense learning for me, not only in the scientific arena, but also on a personal level. Writing this dissertation has had a big impact on me. I would like to reflect on the people who have supported and helped me so much throughout this period.

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To my wife (my sunshine) and Achenyo, you are dearly loved.

To all my sibblings, I love you all. Thank you Iganya, you were there for me.

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## ABSTRACT

The suitability of hardwood charcoal and snail shell particles for carburizing mild steel was investigated. All the mild steel specimens were carburized at constant temperature of 920°C from one (1) to five (5) hours at intervals of one hour. The mild steel specimen in the as-received and carburized conditions were all prepared for the following tests: hardness, impact and microstructure examination. The outcome of the test shows that the as-received specimen has the least hardness value (191Hv) followed by 100% charcoal which showed hardness of 493Hv for the case and 280Hv for the core. The case also showed increases in hardness as pulverized snail shell and charcoal is added in weight percentages of 5%-95%, 10%-90%, 15%-85%, 20%-80% and 25%-75%. The highest hardness value were observed for combination of weight percentage 75% charcoal and 25% pulverized snail shell which gave a case hardness value of 765Hv and core hardness value of 314Hv. The hardness of both the case and the core for all carburized steel samples increases with holding time in the furnace. The case depths also showed a trend similar to the hardness values with holding time, for the combination of weight percentage 25% snail shell the case depth increases from 0.6mm to 1.6mm with increase in holding time from 1hr to 5hrs. In the as-received condition, the impact strength obtained was 70J while in the carburized and quenched hardened condition the impact strength obtained was 78J. However on tempering the impact strength obtained was 74J. The microstructure of the steel specimen in the as-received condition is predominantly pearlite in a matrix of ferrite. However, the carburized steel specimen showed varying degrees of carbide, indicating increased carbon content at the surface.

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# CHAPTER ONE

## 1.0 INTRODUCTION

### 1.1 General introduction

Engineering materials are evolving on daily basis; however, the use of these materials is determined by availability, cost, corrosion resistance, wear resistance, durability and other functional requirements (Ihomet *al.* 2005). One of the commonly available constructional materials in the world is mild steel which is used for various construction purposes, due to its availability and cost effectiveness. This mild steel has its limitations particularly as regarding wear resistance, hardness, and strength. Mild steel finds application in engineering components such as gears, shafts, car parts, and several other areas and case hardening is normally applied to increase the wear resistance of these components. It gives the component a hard case and a tough core (Ihomet *al.*, 2012).

It was revealed that case-hardening of mild steel increases the case hardness of the mild steel resulting in an increased wear resistance of the mild steel and the more the case depth the harder the mild steel. This gives the mild steel material better performance and durability more than the untreated mild steel component (Higgins, 1983, and Ihom, *et al.*, 2011).

A clean environment contributes to healthy living; this fact has since been realized by developed countries like Japan and many other European countries. These countries have succeeded in integrating clean environment into their cultures (Gakken, 2004). The reverse is the case with most developing economies of the world. In Nigeria for instance, a visit to any of the major cities presents obnoxious situation; rubbish, garbage, polythene, plastic waste, metal scraps, and industrial wastes are seen littering

the streets. Some of these wastes form breeding places for mosquitoes and other vectors that spread sicknesses. According to Dara (2007) solid waste management comprises of purposeful and systematic control of the generation, storage, collection, transport, separation, processing, recycling, recovery, and disposal of solid waste. Most developing countries lack both the capacity and the will to manage solid waste. Ayres (1997) in analysing the problem of solid waste in cities advocated recycling, stressing that the savings in terms of reduced environmental impact are less obvious but increasingly important. Municipal waste include garbage and rubbish from households, offices, hotels, markets and also the street refuse such as street sweepings, dirt, leaves, contents of litter receptacles, large quantities of snail shell, egg shells and cow bones etc.; these can be separated and used for other industrial purposes.

Previous works have shown that egg shells and cow bones contain calcium carbonate and their efficacy as energizers in pack carburization of mild steel have equally been proven. In separate works carried out by Ihom (1991) and Ihomet *al.* (2005), the authors were able to show that carburization of mild steel is enhanced with the addition of cowbones to the carburization material. The use of municipal and agricultural solid waste in metallurgy and materials engineering is steadily increasing. Sugar cane bagasse has been used for aluminium alloy particulate composite as well as for case hardening of mild steel (Aigbodionet *al.*, 2009).

Barium carbonate ( $\text{BaCO}_3$ ), Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), Calcium carbonate ( $\text{CaCO}_3$ ) and Potassium carbonate ( $\text{K}_2\text{CO}_3$ ) have generally been added to charcoal to enhance carburization. These carbonate salts are generally referred to as energizers. In many developing countries, small scale foundries and heat treatment shops have abundant charcoal derived from the burning of fire wood. In contrast to charcoal,



energizers ( $\text{BaCO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{CO}_3$ ) are difficult to source locally and are often imported. It is therefore necessary to find an acceptable alternative energizer(s) to encourage the expansion and growth of industries in Nigeria and other developing countries

It is of interest to note that snailshell is a product from agricultural snail farms, markets and can be obtained in most of the riverine areas and equatorial forest zones.

The casehardening of a material can be accomplished by subjecting the component to high temperatures in the presence of a carbonaceous material which may be solid, liquid or gaseous. Energizers are often used to speed up the process. Ihom, *et al*, (2012) observed that the effective case depth is a good measure of carbon penetration and thus the efficacy of an energizer can be assessed by the depth of case-hardening.

## **1.2 Statement of Research problem**

There are different carburizing materials with different carburizing potentials. Generally, energizers enhance the carburizing potential of the carburizing material. Due to high demand of hard and wear resistant component in automobile, defence and other steel industries, there is a need to find a suitable locally sourced carburizer and energizer which will reduce the cost of production of machine components. Most mild steel are not suitable for engineering use at moving parts, hence, there is need for further treatment such as carburization. The conventional energizer in use e.g. Barium Carbonate are usually imported. This amounts to additional cost, hence the need to source and investigate the locally available ones to use.

## **1.3 Justification**

Low carbon steels do not have the capacity to be used as spare parts components in many engineering applications. Their inability to possess hard, wear resistant surfaces and at the same time, tough and shock resistant cores explain why their use is limited in certain engineering applications because the carbon content of mild steel is very low. However subjecting the low carbon steels to a carburising heat treatment can effectively enhance both the wear and shock resistant properties of the materials for case hardened machine parts. Energizers accelerates carbon penetration onto the surface of the mild steel to a regulated depth thereby producing a potentially hard surface. The use of the naturally occurring carbonates as an alternative reduces cost of buying commercial chemical carbonates. Snail shells is suitable and can be locally sourced therefore exploring ways of utilizing them is very crucial and good.

#### **1.4 Aim and Objectives**

The aim of this work is to study the suitability of snail shell as an energizer for carburizing mild steel. The objectives of the research are to:

- i. determine the chemical composition determination of mild steel, charcoal and snail shell.
- ii. carburize the mild steel samples using weight fractions of charcoal and snail shell particles and also determine effective case depth of the carburized sample.
- iii. investigate some mechanical properties of the as-received and carburized samples.
- iv. examine the microstructure of the as-received and carburized samples

#### **1.5 Scope**

The work involves carburization of the sourced mild steel using pulverized hardwood charcoal as the carburizer and snail shell particles as energizer at a temperature of 920°C. The samples properties were examined in the laboratory to ascertain its chemical composition using OES, its microstructural property using optical microscope, mechanical properties using impact testing machine as well as hardness testing machine.

### **1.6 Significance of the Research**

The significance of the research is to exploit the suitability of snail shell as an energizer for carburizing mild steel. Snail shell is cheap, economical and eco-friendly compared to calcium carbonate.

### **1.7 Contribution to knowledge**

- The research has found more economic value for the abundant snail shell as an alternative energizer in case hardening of mild steel.
- The optimum result obtained was at carburizing weight ratio of 75% hardwood charcoal and 25% snail shell at 920°C for 5 hours giving a case depth of 1.25mm and hardness value of 763Hv.
- The research provided information on as-received, carburized-quenched and carburized-quenched-tempered mild steel which has an impact energy of 70Joules, 78Joules and 74Joules respectively.
- The organic materials used in the casehardening media showed improvement in the hardness values of the mild steel specimens over the 191 Hv hardness value of the untreated mild steel specimen.

## **CHAPTER TWO**

### **2.0 LITERATURE REVIEW**

#### **2.1 Steel**

Generally, steel is an alloy consisting mostly of iron, with carbon content between 0.02% and 2.04%, depending on grade. Carbon is the most cost effective alloying material for iron, but various other alloying elements are used such as manganese which increases hardenability and tensile strength and silicon which also increases strength and hardness but not as effective as manganese. Carbon and other elements act as hardening agents, by preventing dislocations in the iron atom crystal lattice from sliding past one another. The amount of alloying elements and form of their presence in the steel can control the quality in terms of hardness, ductility, and tensile strength of the resulting steel. Steel with increased carbon content can be made harder and stronger than iron, but is also more brittle. Though steel has been produced by various inefficient methods long before the renaissance, its use became more common after more efficient production methods were devised in the 17<sup>th</sup> century (Mahat, 2007). Further refinements in the process, such as basic oxygen steelmaking, further lowered the cost of production while increasing the quality of the metal. Today, steel is one of the most common materials in the world and is a major component in buildings, tools, automobiles and appliances.

Steels are generally identified by various grades as defined by various standard organisations. Other than that, it is also identified by the composition, such as carbon, low-alloy or stainless steel and the product forms, such as bar, plate, sheet, strips, tubing or structural shape. The heat treatment such as annealing, quenching, and tempering also can be used to identify steel (Mahat, 2007).

## 2.2 Carbon steels

**Carbon steel**, or **plain-carbon steel**, is a [metal alloy](#). It is a combination of two elements, [iron](#) and [carbon](#). Other elements are present in quantities too small to affect its properties. The only other elements allowed in plain-carbon steel are: [manganese](#) (1.65% max), [silicon](#) (0.60% max), and [copper](#) (0.60% max). [Steel](#) with a low carbon [content](#) has the same properties as iron, soft but easily formed. As carbon content rises the metal becomes harder and stronger but less [ductile](#) and more difficult to [weld](#). Higher carbon content lowers steel's melting point and its temperature resistance in general.

Carbon steels which can successfully undergo heat-treatment have a carbon content in the range of 0.30% to 1.70% by weight. Trace impurities of various other [elements](#) can have a significant effect on the quality of the resulting steel. Trace amounts of [sulphur](#) make the steel "red-short", which is a fault: the steel is brittle and crumbly. Low alloy carbon steel, such as [A36](#) grade, contains about 0.05% sulphur and melts around 1426°C - 1538°C. [Manganese](#) is often added to improve the hardenability of low carbon steels. These additions turn the material into a [low alloy steel](#) by some definitions, but [AISI](#)'s definition of carbon steel allows up to 1.65% manganese by weight.

Typical compositions of carbon in steel are:

- (i) **Mild (low carbon) steel:** approximately 0.05% to 0.25% carbon content with up to 0.4% manganese content (e.g. AISI 1018 steel). Less strong but cheap and easy to shape; surface hardness can be increased through [carburizing](#).
- (ii) **Medium carbon steel:** approximately 0.29% to 0.54% carbon content with 0.60 to 1.65% manganese content (e.g. AISI 1040 steel). Balances ductility and strength and has good wear resistance; used for large parts, forging and car parts.
- (iii) **High carbon steel:** approximately 0.55% to 0.95% carbon content with 0.30 to 0.90% manganese content. Very strong, used for springs and high-strength wires.
- (iv) **Very high carbon steel:** approximately 0.96% to 2.1% carbon content, specially processed to produce specific atomic and molecular microstructures.

Steel can be heat-treated which allows parts to be fabricated in an easily formable soft state. If enough carbon is present, the alloy can be hardened to increase strength, wear, and impact resistance. Steels are often wrought by [cold-working](#) methods, which is the shaping of metal through deformation at a low equilibrium or metastable temperature.

### **2.3 Mild (Low Carbon) Steel**

Low carbon steel has low tensile strength, but is cheap, malleable and its hardness can be increased through carburising process. Density of mild steel is  $7861.093\text{kg/m}^3$ , the tensile strength is a maximum of 500MPa and it has a Young's modulus of 210GPa, (Mahat, 2007).

Mild steel, the most widely used in engineering materials, accounts for 85% of the annual steel production worldwide. It is a metal alloy, a combination of two major

elements which is carbon and iron. The other elements that exist in carbon steels are manganese, sulphur, phosphorus, silicon and copper. Each of these elements present are in very small quantities that may not affect the physical properties of the steel. The largest category of this class of steel is flat-rolled products (sheet or strip), usually in the cold-rolled and annealed condition. The carbon content of these high-formability steels is very low, less than 0.25% C with up to 0.4% Mn. The metal becomes harder and stronger as the carbon content rises. It is used in many applications such as in transportation, pipelines and construction (Mahat, 2007).

## **2.4 Charcoal**

Charcoal is a light, black residue, consisting of carbon and ash, obtained by removing water and other volatile constituents from animal and vegetation substances. Charcoal is usually produced by slow pyrolysis (the heating of wood or other substances in the absence of oxygen). It is usually an impure form of carbon as it contains ash (FAO, 1987).

Charcoal has been used since earliest times for a large range of purposes including art and medicine, but by far its most important use has been as a metallurgical fuel. Charcoal is the traditional fuel of a blacksmith's forge and other applications where an intense heat is required. Charcoal was also used historically as a source of carbon black by grinding it up. In this form charcoal was important to early chemists and was a constituent of formulas for mixtures such as black powder. Due to its high surface area charcoal can be used as a filter, and as a catalyst or as an adsorbent (Chris, 2012). Table 2.2 shows the typical analysis of some types of charcoal. Charcoals with carbon content over 60% are good carbonaceous material and can be used as a source of carbon or metallurgical fuel (FAO, 1987).

Wood species Production Method	Moisture content %	Ash %	Volatile matter - %	Fixed carbon %	Bulk density raw - kg/m <sup>3</sup>	Bulk density pulverised kg/m <sup>3</sup>	Gross calorific value kJ/kg Oven dry basis	Remarks	
Dakama	Earth pit	7.5	1.4	16.9	74.2	314	708	32410	Pulverised fuel for rotary kilns 1/
Wallaba	"	6.9	1.3	14.7	77.1	261	563	35580	1/
Kautaballi	"	6.6	3.0	24.8	65.6	290	596	29990	1/
Mixed Tropical Hardwood	"	5.4	8.9	17.1	68.6				Low grade charcoal fines 1/
"	"	5.4	1.2	23.6	69.8				Domestic charcoal 1/
Wallaba	Earth mound	5.9	1.3	8.5	84.2				Well burned sample 1/
"	"	5.8	0.7	46.0	47.6				Soft burned sample 1/
Oak	Portable steel kiln	3.5	2.1	13.3	81.1			32500	2/
Coconut shells	"	4.0	1.5	13.5	83.0			30140	4/
Eucalyptus Saligna	Retort	5.1	2.6	25.8	66.8				3/

**Table 2.1.** Some Typical Charcoal Analyses (FAO, 1987)

1/= Guyana. 2/= U.K. 3/= Brazil. 4/= Fiji.

#### 2.4.1 Charcoal as metallurgical fuel

Charcoal burns at intense temperatures, up to 2,700 °C. By comparison the melting point of iron is approximately 1,200°C to 1,550 °C. Due to its porosity it is sensitive to the flow of air and the heat generated can be moderated by controlling the air flow to the fire. For this reason charcoal is an ideal fuel for a forge and is still widely used by blacksmiths(FAO, 1987).Charcoal is also an excellent reducing fuel for the production of iron and has been used that way since Roman times. In the 16th century



England had to pass laws to prevent the country from becoming completely denuded of trees due to production of iron. In the 19th century charcoal was largely replaced by coke, baked coal, in steel making due to cost. Until World War II charcoal was still being used in Sweden to make ultra-high-quality steel. In steel-making, charcoal is not only a fuel, but a source for the carbon in the steel industry.

After the 2009 United Nations Climate Change Conference (COP15) in Copenhagen, Denmark, the steel industry in Brazil proposed to replace coal and coke with charcoal in their high temperature furnaces. The program "Green Steel for the Brazilian Steel Industry" converted wood from Eucalyptus plantations into charcoal that will be used in steel making (United Nations framework on climate change, 2009)

## 2.5 Snail shell

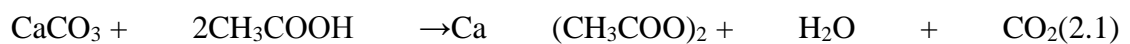
The mollusc shell is typically a calcareous exoskeleton which encloses, supports and protects the soft parts of an animal in the phylum Mollusca, which includes snails, clams, tusk shells, and several other classes. Not all shelled molluscs live in the sea; many live on the land and in freshwater.

Snail shells are indeed made up of mostly calcium carbonate which are either of two crystalline forms calcite and aragonite. They also contain small amounts of protein. Basically, the shell consists of calcium carbonate crystals organized within a matrix of protein. Calcium carbonate crystallizes in two principal forms, **aragonite** and **calcite**, and what form is present in a particular shell may depend on several factors. For example, the crystal type in the shell of the land snail *Helix pomatia* is normally aragonite and calcite. The shells of many marine snails and bivalves normally contain calcite. The fine structure of mollusks shells has been studied by using various techniques including scanning electron microscope of broken surfaces. In each of

them, blocks or stripe of calcium carbonate are separated by a thin layer of conchiolin (Asia and Oladoja, 2003). The calcium carbonates are impervious to water and this property makes it possible for snail shells and their derivatives to have very wide applications.

The formation of a shell in molluscs appears to be related to the secretion of ammonia, which originates from urea. The presence of an ammonium ion raises the pH of the extrapallial fluid, favouring the deposition of calcium carbonate. This mechanism has been proposed not only for molluscs, but also for other unrelated mineralizing lineages.

When calcium carbonate ( $\text{CaCO}_3$ ) dissolves in any common acid, carbon dioxide ( $\text{CO}_2$ ) is generated. So, for example, the dissolution of  $\text{CaCO}_3$  in acetic acid ( $\text{CH}_3\text{COOH}$ ) can be represented below



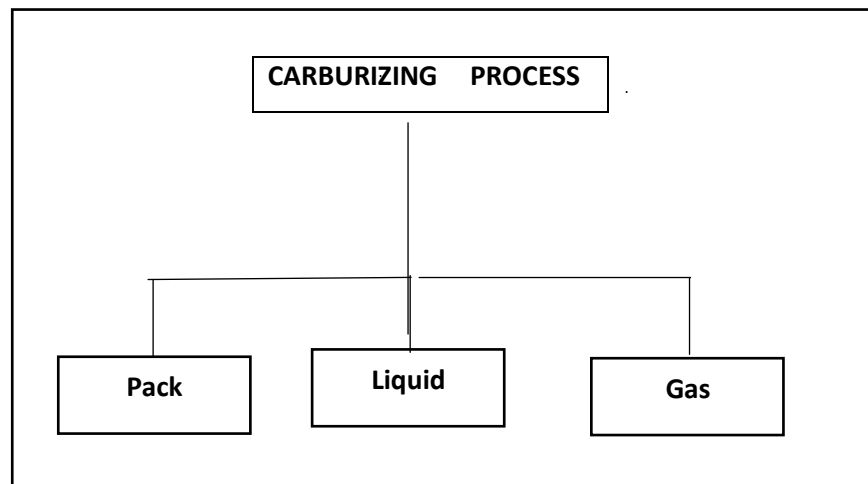
Calcium acetate ( $\text{Ca}(\text{CH}_3\text{COO})_2$ ) is soluble in water and that's why a shell placed in acetic acid will eventually completely dissolve away. Then the carbon dioxide bubbles out of the solution. And that's how one tells - by noticing the bubbles forming on the surface of the shell - that the shell is made of calcium carbonate(Vermeij, 1993).

## 2.6 Carburizing

Carburizing is a heat treatment process to increase the surface hardness and wear resistance of components which possess a fairly good impact strength in service. In the process, austenitized ferrous material is brought into contact with a carbonaceous

atmosphere or medium of sufficient carbon potential as to cause absorption of carbon at the surface by diffusion, and create a concentration gradient (Chapman *et al.*, 2004). In fact, as early as the ninth century, the Benedictine monk Theophilus Presbyter gave a precise method for hardening metallic files. A mixture of three parts of horn meal and one part of salt was used on the surface of heated files which was reheated and water quenched after the process (Prabhudev, 2000).

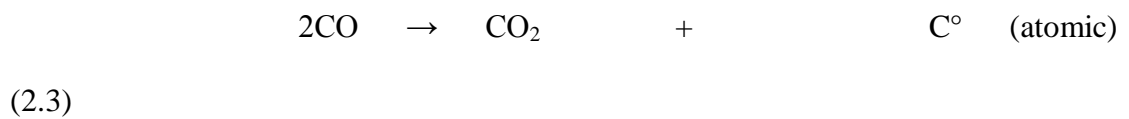
The process can increase the hardness of steel with carbon content between 0.1 and 0.3%. In this process, steel is introduced to a carbon rich environment at elevated temperatures for a certain amount of time. Because this is a diffusion controlled process, the longer the steel is held in this environment the greater the carbon penetration will be and higher the carbon content in these areas. Figure 2.1 shows the common carburizing processes (pack, gas, and liquid). Parts are then tempered to the desired hardness (Higgins, 1991).



**Figure 2.1:** Three types of carburizing process

### **2.6.1 Pack Carburizing**

In this method, the component is packed in a solid carburizing compound in a suitable container and heating it slowly in the furnace to attain a temperature of about 900°C to 950°C. At this state, the oxygen in the air reacts with the carbon present in the carburizing compound to produce carbon monoxide as shown in equation 2.2 and 2.3



In the presence of iron, the carbon monoxide dissociates as the given equation. The atomic carbon thus formed diffuses into the steel and forms the required case. In order to keep the gas generated by the compound in contact with the steel, it is necessary to pack the parts and the compound in gas-tight container (Probhudev, 2000). After certain time, the steel is then removed from the box or furnace and reheated at temperature just above its critical temperature or approximately 910°C – 925°C for fine grain steel, followed by quenching in water, brine or oil. This hardens the skin and the same time refines the core. Smaller articles and thin sections are heated to a lower temperature in order to avoid distortion. The steel is usually given a second heat treatment at about 760°C – 780°C, in order to improve the ductility and impact resistance of the core and case (Rajput, 2000).

### **2.6.2 Liquid carburizing**

Liquid carburizing is a method of case-hardening the surface of the steel to ensure higher wear resistance. This is done by holding the component in a temperature range of 880°C-930°C in a molten cyanide bath, so the atomic carbon diffuses into the

surface of the metal along with a small amount of nitrogen. The process can be performed in internally or externally heated molten salt pots. This molten cyanide bath consist of sodium cyanide and alkaline earth chloride such as barium chloride, calcium chloride, etc., which functions as catalyst to increase the rate of carbon absorption. The mixture of cyanide and chloride will set the liquid bath apart from a plain sodium cyanide bath which goes towards producing a deep case of high carbon with low nitrogen content (*Higgins, 1983a*).

Carburizing salts contain cyanide compound such as sodium cyanide (NaCN) as a source of carbon carrier and alkaline earth chlorides as activators. Barium chloride and strontium chlorides are mainly used as activators to increase the carbon contents of the skin layer on the component and to decrease the melting point and viscosity of the molten bath (ASM handbook, volume 4A, 2013).

Other than the carburizing methods above, there are other methods that can also be used in carburizing steels. The following are some examples:

### **2.6.3 Gas carburizing**

Gas Carburizing Process is a surface chemistry process, which improves the case depth hardness of a component by diffusing carbon into the surface layer to improve wear and fatigue resistance. The work pieces are pre-heated and then held for a period of time at an elevated temperature in the austenitic region of the specific alloy, typically between 820 and 940°C.

During the thermal cycle the components are subject to an enriched carbon atmosphere such that nascent species of carbon can diffuse into the surface layers of the component.

The rate of diffusion is dependent on the alloy and carbon potential of the atmosphere. Care must be taken to ensure that only sufficient carbon is available in the atmosphere at any one time to satisfy the take up rate of the alloy to accept the carbon atoms.

In practice, this is defined in a carbon potential set point profile which runs concurrently with the temperature cycle. The set point may give a boost phase where the carbon potential would be typically set above 1.0% carbon but, as the cycle progresses and the effective case depth increases, the carbon set point will be reduced to complete the diffusion stage.

Depending on the final requirement for effective case depth, the whole cycle may take many hours.

Once the heating and carbon diffusion part of the process are complete it is necessary to rapidly quench the components to a defined alloy recipe. The recipe will specify the quench method, the quenchant temperature and time.

The purpose of the quench process is to provide the required hardness of the component by completing a Martensitic phase change in the alloy.

Gas carburizing and other surface chemistry treatments can be carried out in batch or continuous furnaces dependent on the parts under treatment and the user requirements

#### **2.6.4 Vacuum carburizing**

Vacuum carburizing is usually done by using methane as the carburizing gas. But the process does not always provide the uniformity and repeatability needed to meet today's specification for precision part. Hence, the cost of disposal and high furnace maintenance is a drawback of this process. Because of that, new technologies have been developed that use propane ( $C_3H_8$ ), ethylene ( $C_2H_4$ ) or acetylene ( $C_2H_2$ ) as the carburizing gas to prevent the problem (Rajan, 1994).

Carburization process at high temperature (1000°C-1100°C) is unusual for conventional carburizing because it will cause excessive grain growth in the steel. However, to prevent this problem, the process, of high temperature carburizing is done effectively by using vacuum furnaces. Hayes Company was the first company to achieve the process of carburizing in modern furnaces. This method has shown significant saving in processing time, volume of gases and also can improve the mechanical properties of the steel (Prabhudev, 2000).

#### **2.6.5 Plasma carburizing**

Basically, in plasma carburizing, carbon is imparted to the surface of the steel by impingement of carbon ions escaping from an ionized gas or plasma. The work piece is placed in a vacuum furnace and heated within 850°C-1100°C temperature range. The reaction gas is propane (C<sub>3</sub>H<sub>8</sub>) and generating glow discharge to make the work piece a cathode. The work piece is placed near the anode of a DC circuit. A high DC voltage impressed between the work piece(s) in the anode ionizes the gas in a glowing plasma. A thin plasma envelopes all the surfaces of workpiece(s) completely. Due to ionization, this thin plasma dissociates the carburizing gas and a high carbon potential is established on the surface of the steel.

By using this method, higher carburization efficiency can be obtained compared to the gas carburizing. Also, the plasma carburizing is extremely quick. This is as the result of a combination of high temperature, higher effective carbon potential and the

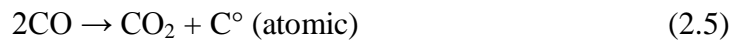
increased rate at which carbon enters the surface due to action of plasma (ASM handbook volume 4, 1990).

## 2.7 Theory of Pack Carburizing

At lower temperatures (200°C - 880°C), the atmospheric oxygen combines with the carbon in the carburizing box and produce carbon monoxide as shown in the equation.

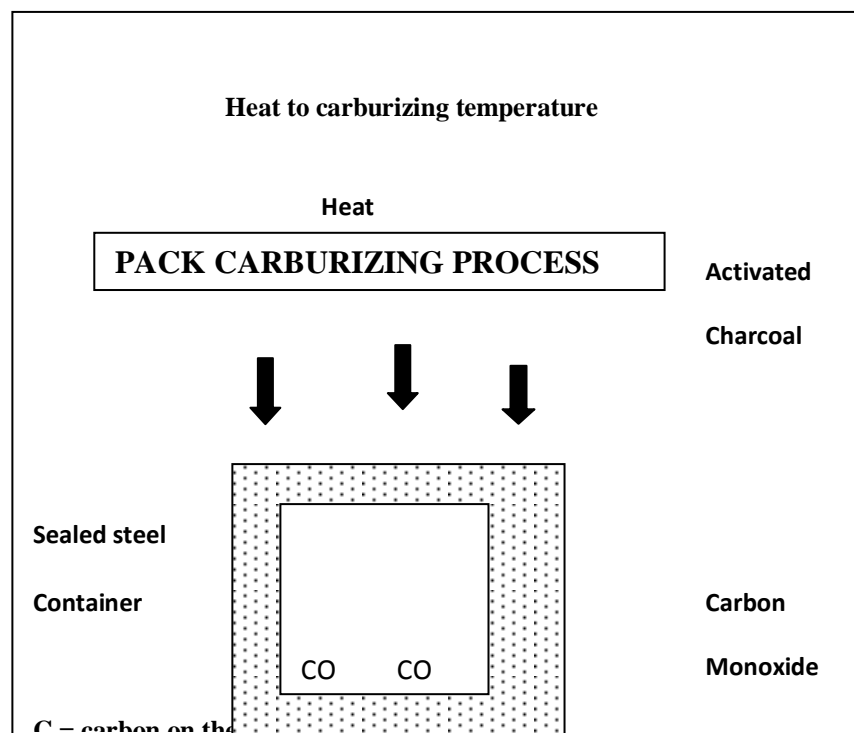


The equilibrium of the reaction is displaced to the right when the temperature is increased and as a result, carbon monoxide also increased. Carbon monoxide will break at the surface of the steel and produce carbon dioxide and atomic carbon as equation.

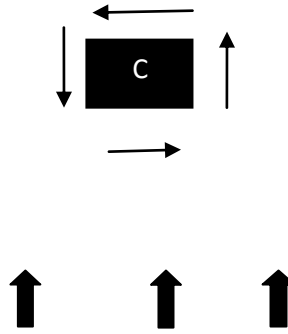


The atomic carbon produced then diffuses into the body of the steel. The carbon dioxide formed, in addition to the atomic carbon, reacts again with the carburizing compound. This cycle of reaction is repeated (Probhudev, 2000). Figure 2.2 shows the pack

carburizing process diagram.







**Figure 2.2:** Pack Carburizing process diagram

Usually, carburizing compounds used for pack carburizing should be in the form of solids. When heated, the compounds will generate gas to give its carbon to steel. The composition and structure of the carburizing steel will give effect to the speed of carbon transformation to the surface of steel. The compounds usually consist of a carbon carrier (carburizers) and energizer or activator, and a binding agent. The popular carburizers used are activated charcoal, coal, semi coke and peat coke. The advantages of using a coke-based compound are its higher heat conductivity and low dusting losses as compared to a pure charcoal-based compound.

Energizers used in carburizing compounds consist either uniquely of a carbonate or mixture of various carbonates. Carbonates of barium, sodium and calcium are used in a definite proportion. Palletized compounds from charcoal powder as energizer and a binder are more effective than compounds using larger pieces of charcoal. Such palletized compounds are suitable for direct quenching as the energizers are held intimately in contact with charcoal.

Pack carburizing is widely used as surface treatment in industries. It is an economical process especially for low volume applications. The boxes used in this process are

inexpensive compared to the other process. The carburizing can also be done in large variety of furnaces as long as the furnaces have the ability to supply a steady and uniform temperature. Other than that, there is no necessity for storing the carburizing compound in a separate room, not like the liquid carburizing, the salts necessary for the process should be stored under strict supervision. The compounds used also will give good support to parts where it can help to prevent sagging at high temperature. This will give the best surface treatment to the component.

By the way, the method has some limitation where the time needed to accomplish the process is excessively long. Apart from heating the components to be carburized, it is also necessary to heat the pots and the carburizing compound which usually weighs more than the components to be carburized. Otherwise, it is not suitable to produce light case work with close limits on case depths. Plus, handling of the compounds is dirty and dusty, (Rajan, 1994).

## **2.8 Case Depth Determination**

Methods employed for determining the depth of the case are the following:

**2.8.1 Chemical Methods:** This method is generally applicable only for carburized cases, but may be used for cyanided or carbonitrided cases. The procedure consists of determining the carbon content at various depths below the surface of a test sample. This method is considered as the most accurate for measuring the total case depth.

**2.8.2. Mechanical Methods:** In general this method is considered to be one of the most useful and accurate of the case depth measuring methods. It is the preferred method for determination for effective case depth and can be used effectively on all types of hardness cases. Table 2.1 suggests the hardness values for various nominal

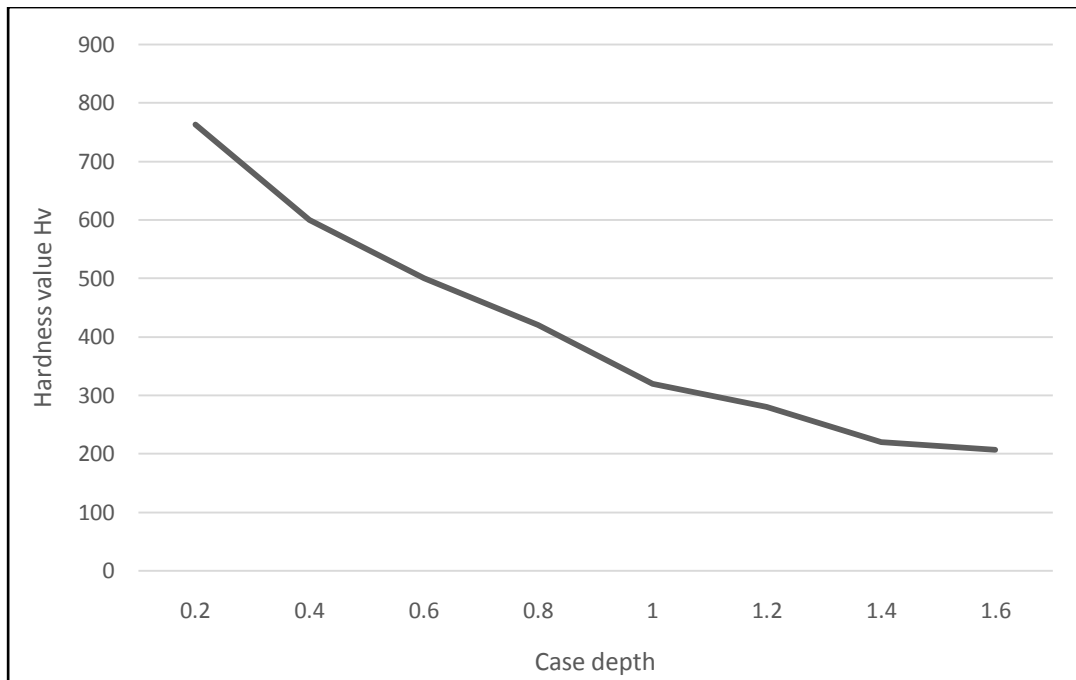
carbon levels which shows an increase in hardness value as the percentage carbon increases, (Metals Handbook, 1981).

**Table 2.2:** Hardness value for various nominal steel(DIN 50150, 2000),

Carbon content % C	Effective Case Depth Hardness	
	HRC	HV
0.28 – 0.32	35	345
0.33 – 0.42	40	392
0.43 – 0.52	45	446
0.53 and over	50	513

(SAEJ423, 1998)

The graphical determination of case depth is as shown in Figure 2.3 which is a plot of hardness values against distance at various compositions of the carburizing compound. The graphical determination of the case depth is based on 50% martensite, 50% pearlite phases commonly taken at 550Hv (Ogoet.al 1994).



**Figure 2.3:** Case hardening showing case depth with hardness value (Rajane *et al*, 1994).

**2.8.3. Visual Methods:** This method in general applies visual procedure with or without the aid of magnification for reading the depth of case procedure by any of the various processes. Samples may be prepared by combinations of fracturing, cutting, grinding and polishing methods. Etching with a suitable reagent is normally required to produce the contrast between the case and core. Nital (concentrated nitric acid in alcohol) of various strengths is frequently used for this purpose.

**2.8.4 Macroscopic Magnification:** Methods for determination of case depth measurement are recommended for routine process control, primarily because of the short time required for determinations, and the minimum of specialised equipment and trained personnel needed. They have the added advantage of being applicable to the measurement of all types of cases.

**2.8.5 Microscopic:** Microscopic methods are generally for laboratory determination and require a complete metallographic polish and an etch suitable for the material and the process. Usually the magnification is 100x, in our case the used magnification is 40x which can be extra magnified by 2 or 3 times. Microscopic method may be used for laboratory determination of total case and effective case depths in the hardened condition. The distinction between macro and micro is based on the test forces in relation to the indentation depth. Attention is drawn to the fact that the micro range has an upper limit given by the force of 2N and a lower depth limit given by the indentation of 0.2  $\mu\text{m}$ . Finally it is important to bear in mind that the method of case depth determination should be carefully selected on the basis of specific requirements in terms of economy (Metals Handbook, 1981).

Case hardness of carburized steel is primarily a function of carbon content. Most of the processes involve either enriching the surface layer with carbon or nitrogen, usually followed by quenching and tempering (Chapman *et al.*, 2004). When the carbon content of the steel exceeds about 0.50%, additional carbon has no effect on hardness but does enhance hardenability. Carbon in excess of 0.5% may not be dissolved, which will thus require temperatures high enough to ensure carbon-austenite solid solution. However, surface carbon is often limited to 0.9% because too high a carbon content can result in retained austenite and brittle martensite. In case-hardened steel, the hardenability of both case and core must be considered. The difference in carbon content, case and core have quite different hardenabilities, and this difference is much greater for some steels than for others. The relationship between the thermal gradient and carbon gradient during quenching of a carburized part can make a measurable difference in the case depth as measured by hardness. That is, an increase in base hardenability can produce a higher proportion of

martensite for a given carbon level, yielding an increased measured case depth. Therefore, a shallower carbon profile and shorter carburizing time could be used to attain the desired result in a proper chosen steel (Mahat, 2007)

## **2.9 Theory of case hardening**

The term case hardening is derived from the practicalities of the carburization process itself, which is essentially the same as the ancient process. The steel work piece is placed inside a case packed tight with a carbon-based case hardening compound. This is collectively known as a carburizing pack. The pack is put inside a hot furnace for a variable length of time. Time and temperature determines how deep into the surface the hardening extends. However, the depth of hardening is ultimately limited by the inability of carbon to diffuse deeply into solid steel, and a typical depth of surface hardening with this method is up to 1.5 mm. Other techniques are also used in modern carburizing, such as heating in a carbon-rich atmosphere. Small items may be case hardened by repeated heating with a torch and quenching in a carbon rich medium, such as the commercial products *Kasenit / Casenite* or "Cherry Red". Older formulations of these compounds contain potentially toxic cyanide compounds, while the more recent types such as Cherry Red do not (Craig, 2006)

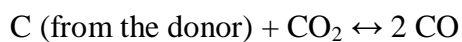
Case hardening or surface hardening is the process of hardening the surface of a metal object while allowing the metal deeper underneath to remain soft, thus forming a thin layer of harder metal (called the "case") at the surface. For iron or steel with low carbon content, which has poor to no hardenability of its own, the case hardening process involves infusing additional carbon into the case. Case hardening is usually done after the part has been formed into its final shape, but can also be done to increase the hardening element content of bars to be used in a pattern welding or

similar process. The term face hardening is also used to describe this technique, when discussing modern armour (Craig, 2006).

Hardened metal is usually more brittle than softer metal, through-hardening (that is, hardening the metal uniformly throughout the piece) is not always a suitable choice for applications where the metal part is subject to certain kinds of stress. In such applications, case hardening can provide a part that will not fracture (because of the soft core that can absorb stresses without cracking) but also provides adequate wear resistance on the surface.

Carbon itself is solid at case-hardening temperatures and so is immobile. Transport to the surface of the steel was as gaseous carbon monoxide, generated by the breakdown of the carburizing compound and the oxygen packed into the sealed box. This takes place with pure carbon, but unworkably slowly. Although oxygen is required for this process it is re-circulated through the CO cycle and so can be carried out inside a sealed box. The sealing is necessary to stop the CO either leaking out, or being oxidized to CO<sub>2</sub> by excess outside air.

Adding an easily decomposed carbonate "energizer" such as barium carbonate breaks down to BaO + CO<sub>2</sub> and this encourages the reaction increasing the overall abundance of CO and the activity of the carburizing compound.



It is a common knowledge fallacy that case-hardening was done with bone, but this is misleading. Although bone was used, the main carbon donor was hoof and horn. Bone contains some carbonates, but is mainly calcium phosphate (as hydroxylapatite). This

does not have the beneficial effect of encouraging CO production and it can also introduce phosphorus as an impurity into the steel alloy.

Both carbon and alloy steels are suitable for case-hardening; typically mild steels are used, with low carbon content, usually less than 0.3%. These mild steels are not normally hardenable due to the low quantity of carbon, so the surface of the steel is chemically altered to increase the hardenability. Case hardened steel is formed by diffusing carbon (carburization), nitrogen (nitriding) and/or boron (boriding) into the outer layer of the steel at high temperature, and then heat treating the surface layer to the desired hardness (Craig, 2006).

### **2.9.1 Diffusion in solid materials**

Diffusion is the net movement of a substance (e.g., an atom, ion or molecule) from a region of high concentration to a region of low concentration. This is also referred to as the movement of a substance down a concentration gradient. A gradient is the change in the value of a quantity (e.g., concentration, pressure, temperature) with the change in another variable (e.g., distance). For example, a change in concentration over a distance is called a concentration gradient, a change in pressure over a distance is called a pressure gradient, and a change in temperature over a distance is called a temperature gradient (Smith, 1993).

### **2.9.2 Fick's Laws**

Fick's laws of diffusion describe diffusion and were derived by Adolf Fick in 1855. They can be used to solve for the diffusion coefficient,  $D$ . Fick's first law can be used to derive his second law which in turn is identical to the diffusion equation (Smith, 1993).



Fick's first law relates the diffusive flux to the concentration under the assumption of steady state. It postulates that the flux goes from regions of high concentration to regions of low concentration, with a magnitude that is proportional to the concentration gradient (spatial derivative), or in simplistic terms the concept that a solute will move from a region of high concentration to a region of low concentration across a concentration gradient.

The second law predicts how diffusion causes the concentration to change with time.

In one (spatial) dimension, the first law is:

$$J = - S \cdot D \cdot \frac{\delta c}{\delta x} \quad (2.7)$$

Where

$J$  = the amount of carbon which passes in unit time across area  $S$  in a plane normal to the 'x' axis;

$\frac{\delta c}{\delta x}$  = variation of carbon content with depth below the surface (concentration gradient of

the carbon);

$D$  = the diffusion coefficient of carbon in  $\gamma$ -iron

$D$  is very dependent upon temperature but in this instance is of the order of  $10^{-7}$  cm<sup>2</sup>/s at the carburizing temperature (920°C).

From the Diffusion Equation **2.7**

$$\frac{\delta c}{\delta x} = D \frac{\delta^2 c}{\delta x^2}. \quad (2.8)$$

The *depth* of carbon could be derived using one of the standard methods for solving differential equations from Fick's second law. However a simple formulae derived by Einstein from mathematical studies of diffusion, can be applied:

$$x = \sqrt{(2Dt)}. \quad (2.9)$$

Where  $D$  = the diffusion coefficient ( $\text{cm}^2/\text{s}$ )

And  $t$  = time of diffusion (s).

This can be written:

$$\text{Case depth (d)} = k\sqrt{t} \quad (2.10)$$

Where  $k = \sqrt{2D}$  and gives a rough estimate of the time required to produce a case of given depth (Bokshteinet *al*, 2005).

### 2.9.3 Applications of Fick's laws

Equations based on Fick's law have been commonly used to model transport processes in foods, neurons, biopolymers, pharmaceuticals, poroussoils, population dynamics, nuclear materials, semiconductor doping process, etc. Theory of all voltammetric methods is based on solutions of Fick's equation. A large amount of experimental research in polymer science and food science has shown that a more general approach is required to describe transport of components in materials undergoing glass transition. In the vicinity of glass transition the flow behavior becomes "non-Fickian". It can be shown that the Fick's law can be obtained from the

Maxwell-Stefan equations of multi-component mass transfer. The Fick's law is limiting case of the Maxwell-Stefan equations, when the mixture is extremely dilute and every chemical species is interacting only with the bulk mixture and not with other species. To account for the presence of multiple species in a non-dilute mixture, several variations of the Maxwell-Stefan equations are used (*Taylor, et al 1993*).

## **2.10 Surface treatments**

**Treatment of metals can be of essential importance in many industries. It is not a new process, but a process that dates back as early as mankind started using gold decoratively before 4000 BC.**

Today there can be several reasons why it is necessary to change the surface properties of metals. For example surface treatment of metals are used for:

- Decoration and/or reflectivity
- Improved hardness (e.g. for resistance to damage and wear)
- Prevention of corrosion

Treatment of metal surfaces plays an enormous role in extending the life of metals, such as in automotive bodies and construction materials, an often seen application is cleaning of stainless steel bodies for windows, etc.

Almost every industry will have a need for metal surface treatment equipment. Among the industries who today use metal surface treatment include the following; the [automotive industry](#), the construction industry, the Container industry, the electrical industry, the [medical industry](#), industrial equipment, industries using laboratory equipment, aerospace, and several other industries. The range of

components treated varies from: spectacle frames, components for automotive, screws, nuts, bolts, different tools and many others (Craig 2006).

### **2.10.1 Induction Hardening**

Induction hardening is a form of [heat treatment](#) in which a metal part is heated by [induction heating](#) and then [quenched](#). The quenched metal undergoes a [martensitic transformation](#), increasing the [hardness](#) and brittleness of the part. Induction hardening is used to selectively harden areas of a part or assembly without affecting the properties of the part as a whole

Induction heating is a non-contact heating process which uses the principle of [electromagnetic](#) induction to produce heat inside the surface layer of a work-piece. By placing a [conductive](#) material into a strong alternating [magnetic field](#), electric current can be made to flow in the material thereby creating heat due to the  $I^2R$  losses in the material. In magnetic materials, further heat is generated below the [Curie point](#) due to [hysteresis](#) losses. The current generated flows predominantly in the surface layer, the depth of this layer being dictated by the frequency of the alternating field, the surface power density, the [permeability](#) of the material, the heat time and the diameter of the bar or material thickness. By [quenching](#) this heated layer in water, oil, or a [polymer](#) based quench, the surface layer is altered to form a [martensitic](#) structure which is harder than the base metal (Rudnevet.al, 2002).

### **2.10.2 Flame Hardening**

Flame hardening is similar to induction hardening, in that it is a surface hardening process. Heat is applied to the part being hardened, using an oxy- acetylene (or similar gas) flame on the surface of the steel being hardened and heating the surface above the upper critical temperature before quenching the steel in a spray of water. The result is a hard surface layer ranging from 0.127cm to 0.635cm deep. As with induction hardening, the steel component must have sufficient carbon (greater than 0.35%). The composition of the steel is not changed; therefore core mechanical properties are unaffected. Flame hardening produces results similar to conventional hardening processes but with less hardness penetration. Applications for flame hardening are similar to those for induction hardening, although an advantage of flame hardening is the ability to harden flat surfaces. Flat wear plates, and knives can be selectively hardened using this process (Craig, 2006)

## **2.11 Heat treatment after Carburization**

Heat treating is a group of industrial and metalworking processes used to alter the physical, and sometimes chemical, properties of a material. The most common application is metallurgical. Heat treatments are also used in the manufacture of many other materials, such as glass. Heat treatment involves the use of heating or chilling, normally to extreme temperatures, to achieve a desired result such as hardening or softening of a material. Heat treatment techniques include annealing, case hardening, precipitation strengthening, tempering and quenching. It is noteworthy that while the term *heat treatment* applies only to processes where the heating and cooling are done for the specific purpose of altering properties intentionally, heating and cooling often occur incidentally during other manufacturing processes such as hot forming or welding (Smith, 1993). After carburizing, the work is either slow cooled for later quench hardening, or quenched directly into oil. Quench selection is made to achieve

the optimum properties with acceptable levels of dimensional change. Hot oil quenching may be used for minimal distortion, but may be limited in application by the strength requirements for the product. Alternatively, bearing races may be press quenched to maintain their dimensional tolerances, minimizing the need for excessive post heat treatment grinding. In some cases, product is tempered, then cryogenically processed to convert retained austenite to martensite, and then retempered (Smith, 1993).

## **2.12 Review of related works:**

Rashmi *et al* (2012), reported on the mechanical and wear properties of carburized low carbon steel samples. The mechanical and wear properties of mild steels were found to be strongly influenced by the process of carburization. The experimental results also showed that the mild steels carburized under different temperatures (850°C, 900°C and 950°C) out of which the mild steels carburized at the temperature of 950°C gave the best results for the different kinds of mechanical and wear properties.

Ihom (2013), studied various carburizing compounds used to pack carburized mild steel. Various weight percentages of cow bone were used as energizer in the carburizing compounds. The experiments were carried out using a muffle furnace at 900°C for 8 h. The result showed that 60 wt% charcoal / 40 wt% cow bone had the best result with an effective case depth of 2.32 mm produced on the case of the carburized steel. The work showed that cow bone can be used as energizer in pack carburization of mild steel.

Atanda *et al* (2009), reported on the investigation of the effect of carburizing variables – temperature, time and percentage of energizer – on the case properties of C2R steel

obtained from HMT Ltd. India. A carburizer consisting of hardwood charcoal and coke respectively in the ratio of 2:1 was used for the research with sodium carbonate as the energizer. The carburizing box was filled with 20 mm thick carburizer compound prior to fixing the steel samples in place.

Aramideet *al*(2010a), reported on the mechanical properties of mild steel subjected to packed carburization treatment using pulverized bone as the carburizer, carburized at 850°C, 900°C and 950°C, soaked at the carburizing temperature for 15 minutes and 30 minutes, quenched in oil and tempered at 250°C. They concluded that the sample carburized at 900°C soaked for 15 minutes and the one carburized at 850°C soaked for 30 minutes followed by oil quenching and tempering at 250°C were better because they showed a trend of hard case with softer core.

Ogoet *al* (1994), studied Feasibility of Sea and Coconut Shells as Substitute to Barium Carbonate ( $\text{BaCO}_3$ ) in small scale foundry and heat treatment shop in Nigeria. Samples were carburized at 950°C in carburization compounds containing charcoal and energizing materials at predetermined proportions. The results obtained showed that sea shell was a more effective energizer than the coconut shell and compared favourably with barium carbonate. Sea shell showed 82.5% efficiency compared with  $\text{BaCO}_3$  and 134.70% with limestone ( $\text{CaCO}_3$ ) at 950°C carburizing temperature. It was found that 30% sea shell addition to charcoal gave optimal carburization case depth which was 2.8 times the values obtained from 100% charcoal. However, there was no significant difference between 100% charcoal and compounds containing various proportions of coconut shell in the carburizing kinetics.

Ihomet *al* (2013) Investigation of Egg Shell Waste as an Enhancer in the Carburization of mild Steel reported the result to show that egg shell containing carburizing material produced higher case hardness on the steel than those carburizing materials without egg shell. Sugar cane waste produced a case hardness of 45.1HRC, with egg shell the case hardness became 45.5HRC. Melon shell waste produced a case hardness of 47.8HRC, and with egg shell the case hardness became 52.6 HRC. This hardness corresponded to an effective case depth of 0.65mm. Aracaceae flower wastes produced a case hardness of 56 HRC on the mild steel this corresponded to an effective case depth of 0.7mm, with egg shell the case hardness became 56.6 HRC with an effective case depth of 0.75mm. The calculated total case depth based on carburizing parameters was 1.34mm. The effect of egg shell waste as an enhancer in the carburization of mild steel was established. The result of the work clearly showed that egg shell waste can be used in place of calcium carbonate to improve the hardness value of mild steel during carburization.



## **CHAPTER THREE**

### **3.0 MATERIALS AND METHODS**

#### **3.1 Materials**

The steel material of 16mm diameter for the work was collected from the Metallurgical and Materials Engineering departmental Laboratory of the Ahmadu Bello University, Zaria. The snail shells were collected from the Terminus market in Jos, Plateau State, while the charcoal was collected from Nigerian Metallurgical Development Center, NMDC, Jos, Plateau State

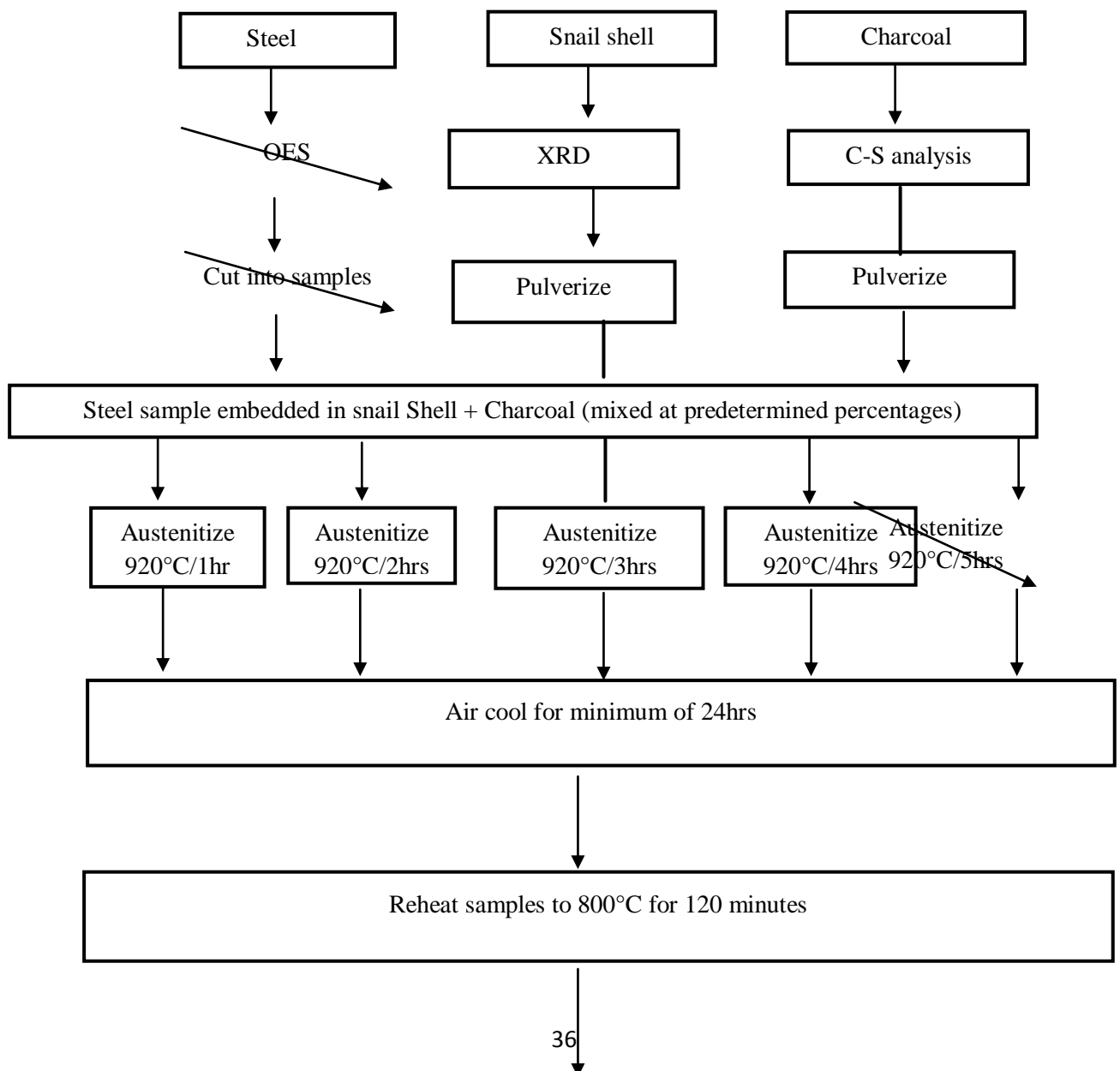
#### **3.2 Equipment**

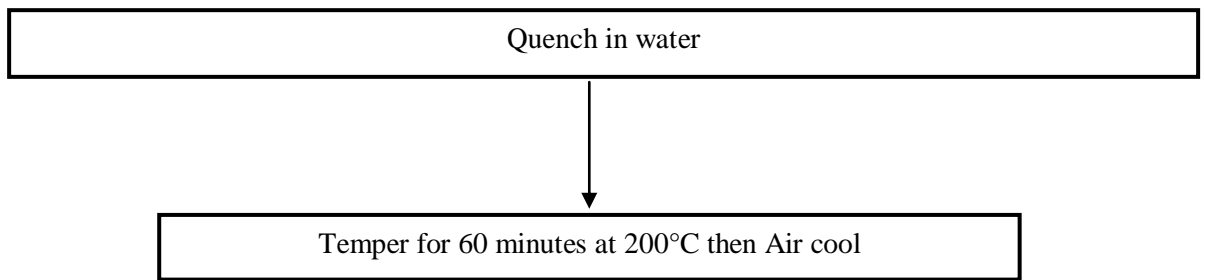
The equipment/apparatus that were used for the research included the following: Tubular Furnace of maximum operating temperature of 1200°C, hand blower, Sieving Machine (Model S750), Lathe Machine (made by H.ErnaultSomua model j350), Shuttle Buffalo Hammer Mill (Model WA8H), quenching baths, hacksaw, heat resisting steel carburization boxes, Grinding Machine Shambhari Index, Polishing Machine (made by Fritz Werner of Germany model-wp2000), and Vickers Micro hardness Testing Machine (Model MV1-PC with serial No: 07/2012-1329 made by Fuel Instruments and Engineers PVT. Ltd., of India), weighing balance, Optical

Metallurgical Microscope (model NJF-1), W&T Avery Izod impact testing machine (type 6701, serial number 5142519), X-ray fluorescence(MiniPAL4 model© 2005, PALalytical B.V, IJlyweg 1, 7602 EA, Almelo, The Netherlands. PP6.1-9.14 (ED-XRFS)), X-ray Diffractometer(made by PAN Analytical BV of Netherlands), TSA Carbon-Sulphur Analyzer, and Optical Emission Spectrometer (model PDA: 7000).

### 3.3 Method

#### 3.3.1 Block diagram of the methodology





**Figure 3.1:** Schematic block diagram of the heat treatment of mild steel

### 3.3.2 Materials Preparation

The Snail shells were pulverized using a grinding mill. The hardwood charcoal was reduced to small sizes by pounding in a mortar with a pestle. The pulverized charcoal and snail shell were then sieved to 0.25mm sieve size. A small quantity of both pulverized snail shell and charcoal were set aside for characterization and further grinding was carried out in preparation for characterization. The pulverized charcoal and the shells were then mixed at the predetermined proportions as shown below in Tables 3.1.

**Table 3.1: Composition of the carburizing compounds used in this research**

Sample	Composition
1	100% Hardwood Charcoal + 0% Snail Shell
2	95% Hardwood Charcoal + 5% Snail Shell
3	90% Hardwood Charcoal + 10% Snail Shell
4	85% Hardwood Charcoal + 15% Snail Shell
5	80% Hardwood Charcoal + 20% Snail Shell

The steel material was cut and machined into 30 samples of length and diameter of 50mm and 16mm and all the surfaces were polished. The bottom of the carburizing box was covered with a layer of the carburizing compound. A single specimen was placed in each box and the remaining space was filled with the carburizing mixture. The lids to the carburizing boxes were then replaced and sealed using clay paste to avoid air ingress so that oxidation will not take place. The box was then placed in the central zone of the furnace at a constant temperature of 920°C was maintained. Carburizing time of 5hrs at an interval of one (1) hour were observed. At the end of each treatment, the specimen was removed from the furnace and air cooled. The surfaces of the samples was polished after the carburization process. The samples were then prepared for hardness, impact and microstructural analysis.

### **3.3.3 Optical Emission Spectrometer (OES) examination of steel sample**

The steel sample was analysed using Optical Emission Spectrometer model PDA: 7000. The steel samples were machined to desired sizes and surfaces of the sample was grinded and polished to a mirror-like surface. The steel samples were then taken to the spark unit on the OES machine for analysis. The results which are in elemental form are then displayed on the computer screen.

### **3.3.4 X-ray Diffractometer (XRD) analysis of the snail shell**

The snail shell powder was analysed using Bruker D80-θ X-ray diffractometer equipped with  $\text{CoK}\alpha$  monochromating multilayered mirrors. A Rietveld refinement software, TOPAS<sup>TM</sup>, was used for quantitative analysis. The pulverized snail shell was ground

further to obtain a fine powder. The powder was now placed in the sample holder and smear uniformly onto a glass slide assuring a flat upper surface then packed into a sample container and sprinkle on double sticky tape. This is done typically to avoid interference because the substrate is amorphous. The intensity of the diffracted rays is continuously recorded and results obtained as the sample and detector rotates through their respective angles.

### **3.3.5 X-ray fluorescence (XRF) analysis of the snail sample**

The snail shell powder was also analysed using XRF MiniPAL4 model© 2005, PALalytical B.V, lelyweg 1, 7602 EA, Almelo, The Netherland. PP6.1-9.14 (ED-XRFS). The sample was prepared by dissolving a fully oxidized sample at a high temperature in a solvent in a graphite crucible. The melted mixture is then agitated and poured into a mould to create a glass disk for the analysis. The glass disc is then excited with x-ray radiation, normally generated by the x-ray tube operated at a potential of between 10 and 100kV. Interactions of this primary radiation with atoms of the sample causes ionization of discrete orbital electrons. During the subsequent electronic re-arrangement by which the atom de-excites back to the ground state, fluorescence x-rays of the energy characteristic of the elements are emitted. The emission intensity of this characteristic radiation is measured with a suitable x-ray spectrometer and compared with that from a standard sample.

### **3.3.6 Carbon-sulphur (CS-i) analysis of the charcoal**

The charcoal was analysed using the TSA Carbon-Sulphur Analyzer. After weighing the sample in a ceramic crucible, the weight is transferred from the interfaced to the

PC. Then an accelerator (iron) is then added and, after having placed the crucible on the pedestal, the analysis starts. The analysis time is 45 seconds. The detector signals and the instrument parameters are displayed during analysis. Evaluation of the signals and display of results are done automatically.

### **3.3.7 Carburizing Process**

The prepared steel material of dimension 50mm by 16mm were proportioned into six (6) groups with each group having five(5) samples. The first 5 were placed into the pack carburizing box and a 100% (100gm) of the wood charcoal was used. The 5 (five) samples were each heated in the central part of the tubular furnace at a temperature of 920°C at a holding time of 1hr to 5hrs. This was designated as the control sample since no energizer was used in this case. The pack boxes was introduced into the tubular furnace within the uniform temperature zone which has already attained the carburizing temperature. The heating-up time needed to make up for the sudden temperature drop which follows the introduction of the packs into the furnace was less than 10 min and is therefore negligible compared to the carburizing time. At the end of the carburization process, the specimen was taken out and air cooled. The same was repeated for another set of 5 samples which were placed in the pack boxes and of 95% (95gm) wood charcoal and 5% (5gm) snail shell. They were heated in the tubular furnace for holding times of 1hr to 5hrs after which the specimen was taken out and air cooled. This process was repeated for different composition of charcoal and snail shell.

### **3.3.8 Heat treatment after carburization**

The carburized samples were removed from the container after cooling, placed back into the furnace and reheated to 800°C and allowed to soak for 120 minutes.

Thereafter the samples were removed and quenched in water in a process known as quench hardening and then tempered at 200°C for 60 minutes.

### 3.3.9 Hardness Profile

Steel rod of 50mm long was used for the experiment. The carburized steel rods were prepared and polished for hardness measurements on a Vickers Hardness Tester. Hardness measurements on all the specimens were carried out on Micro Vickers Hardness Tester model - MVI-PC. In order to determine the Vickers hardness (Hv) according to ISO 6507, the diamond pyramid-shaped indenter (with interfacial angle of 136°) was pressed onto the specimen with a defined test load of 0.3 Kgf. The Vickers hardness (Hv) results from the quotient of the applied test force, F and the surface area of the residual indent on the specimen. To calculate the surface area of the residual pyramidal indentation, the average of the two diagonals (d<sub>1</sub> and d<sub>2</sub> in mm) was used. This was repeated for each of the specimen.

$$\begin{aligned} \text{Vickers hardness (Hv)} &= k \left( \frac{\text{Test force}}{\text{Surface area of indentation}} \right) \\ &= 0.102 \left( \frac{2F \sin \frac{136^\circ}{2}}{d^2} \right) = 0.1891 \left( \frac{F}{d^2} \right) \end{aligned}$$

From the hardness values obtained for each specimen, hardness profiles were plotted and effective case depths at various intervals were extracted. The case depth of the samples was determined using Grossman hardness method. For each of the sample, test was conducted thrice and the average of the samples was taken as the observed values in each case.

### 3.3.10 Impact test

The impact energy test was carried out using W&T Avery Izod impact testing machine type 6701. The specimen was a cantilever, clamped upright in an anvil, with a V-notch at the level of the top of the clamp. The specimen was hit by a striker carried on a pendulum which was allowed to fall freely from a fixed height, and gave a blow of 160 Joules ( $\approx$  120 ft lb). After fracturing the specimen, the height to which the pendulum rose was recorded by a slave friction pointer mounted on the dial, from which the absorbed energy amount was read.

### **3.3.11 Optical Microscopy Examination**

The samples were cut from the as-received, carburized and tempered specimen. The cut samples were mounted in Bakelite, and mechanically ground progressively on grades of Silicon carbide (SiC) impregnated emery paper (80-600 grits) sizes using water as the coolant. The ground samples were then polished using one-micron size alumina polishing powder suspended in distilled water. Final polishing was achieved using 0.5-micron alumina polishing powder suspended in distilled water. After the polishing operation, etching of the polished sample was done using Nital reagent and the specimen were viewed under a metallurgical microscope with an in-built camera.



## CHAPTER FOUR

### 4.0 RESULTS AND DISCUSSION

#### 4.1 Chemical composition of the as-received steel

The Elemental composition of the steel sample as obtained using the Optical Emission Spectrometer (OES), is shown in Table 4.1.

**Table 4.1:** Chemical composition of the as-received steel

Element	C	Si	Mn	P	S	Ni	Cr	Mo	Cu	Ti	Al	Co	V	Fe
%	0.155	0.224	0.967	0.025	0.010	0.03	0.036	0.017	0.040	0.030	0.012	0.035	0.039	98.368

#### 4.2 Analysis of Snail Shell

##### 4.2.1 Characterization of snail shell

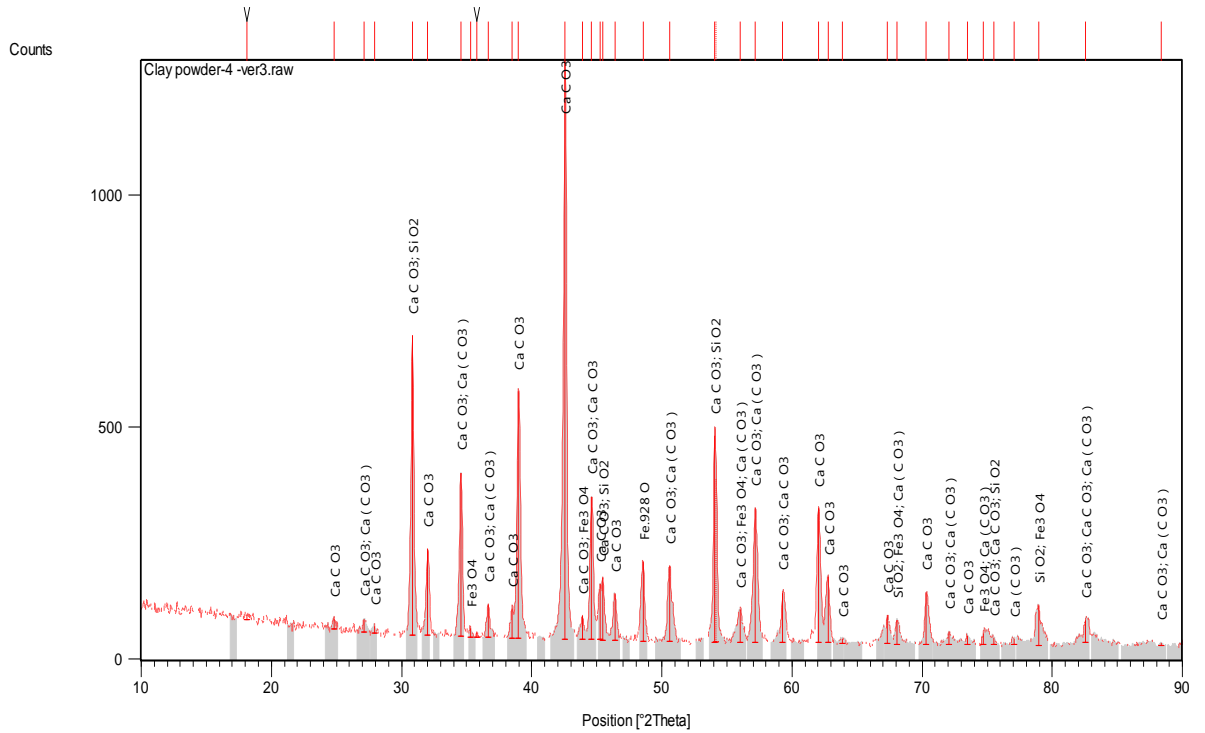
The XRF composition of the snail shell is represented in Table 4.2. The XRF analysis confirmed that CaO was found to be major constituent of the snail shell. With the presence of high percentage of CaO mean that snail shell can be used as an energizer. This result of XRF is in agreement with the result of XRD obtained.

**Table 4.2:** Chemical composition of Snail Shell obtained using XRF

Compound	SO <sub>3</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	V <sub>2</sub> O <sub>6</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	CuO	SrO	ZrO <sub>2</sub>	BaO
Conc unit %	0.05	0.20	88.36	0.19	0.01	0.02	0.60	0.03	0.83	0.007	0.06

The XRD pattern of Snail shell is given in Figure 4.1. The Snail shell showed the presence of CaCO<sub>3</sub> phase while the identified crystalline phases is given in Table 4.3.

The XRD pattern of the snailshell powder obtained has major diffraction peaks at: 26.96, 34.06 and 9.01 and phases at these peaks are: Calcite, CaCO<sub>3</sub>, Quartz, syn (SiO<sub>2</sub>) and Tilleyite (Ca<sub>5</sub> Si<sub>2</sub> O<sub>7</sub> (CO<sub>3</sub>)<sub>2</sub>), while each of these phases have a score of 94, 25 and 13 (Figure 4.1 and Table 4.3). Complete analysis confirmed that the Snail shell powder contains these (C, O, Si, Ca), with Calcite having higher score of 94 as well as aragonite and CaCO<sub>3</sub>. This confirms that Snail shell powder is a calcium carbonate based materials as also reported in the previous work done by Ogoet *al*, (1994).



**Figure 4.1:** XRD pattern of the snail shell powder

**Table 4.3:** Identified crystalline phases of snail shell powder

Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
70-0095	36	Calcium Carbonate	0.000	0.322	CaCO <sub>3</sub>
03-1067	40	Aragonite	0.000	0.206	CaCO <sub>3</sub>
81-0065	20	Silicon Oxide	0.000	0.248	SiO <sub>2</sub>
75-0449	15	Magnetite	0.000	0.025	Fe <sub>3</sub> O <sub>4</sub>
86-2334	21	Calcite	0.000	0.077	CaCO <sub>3</sub>
79-2175	17	WPIstite, syn	0.000	0.093	Fe <sub>928</sub> O

The presence of Calcite as well as  $\text{CaCO}_3$  and Aragonite in the snail shell shows that the snail shell can be used as an energizer, which is in agreement with Bolewski *et al.*, (1974). The  $\text{CaCO}_3$  on dissociation assists in supplying the nascent carbon for the carburization (Okongwu, 1989; Ihomet *et al.*, 2011; Aramide *et al.*, 2010; Ihomet *et al.*, 2012).

### 4.3 Proximate and elemental analysis of Charcoal

The proximate analysis of the charcoal is as shown in Table 4.4. The result obtained from the analysis showed that fixed carbon contains 83.64% which shows that it is a good carbonaceous material and the effect of the volatile matter (14.64%) as well as the ash (1.74%) is not causing any significant impact on the sample FAO, (1987).

**Table 4.4:** Proximate analysis of the charcoal

Fixed Carbon %	Volatile matter %	Ash %
83.64	14.62	1.74

Table 4.5 is an analysis of the charcoal using the carbon-sulphur analyser. The result obtained from using TSA carbon sulphur analyzer showed that 58.76% C and 0.53% S were present in the charcoal. It was observed that the charcoal powder has acceptable quality to be used as a metallurgical fuel since the recommended value of above 60% C content is high in percentage fixed carbon (Antalet *et al.*, 1996). With this high amount of carbon and low sulphur content in the charcoal confirms that the charcoal can be used for carburization.

**Table 4.5:** Analysis of Carbon and Sulphur of the charcoal using carbon-sulphur analyser

No.	Carbon %	Sulphur %	Weight %
1	58.75538	0.53012	0.0150

#### 4.4 Hardness Test Value (HV) for the Carburized steel

The hardness test results are as presented in Tables 4.6- 4.12.

As observed in Table 4.6, the hardness profile for the as-received steel sample showed a trend of an equally spread hardness profile across the steel sample indicating equal hardness for the case as well as the core of the steel. However as the steel sample is being carburized across varying time and temperature with various predetermined mixture of energizer and charcoal, the hardness of the steel sample began to vary, increasing from core to case showing an impregnation of carbon on the surface (case) of the steel sample.

**Table 4.6:** Hardness values for the as-received mild steel sample

Distance in mm from the case to the core	0.5	1.0	1.5	2.0	2.5	3.0	3.5
Hardness value	191	191	191	191	191	191	191

**Table 4.7:** Hardness value (HV) of carburized mild steel using 100% hardwood charcoal

Distance (mm) from the case to the core	1hr	2hrs	3hrs	4hrs	5hrs
0.5	400	429	449	470	493
1.0	370	398	394	389	404
1.5	348	357	358	360	391
2.0	312	318	320	323	328
2.5	258	275	293	307	325
3.0	218	266	269	278	294
3.5	211	216	266	269	280

**Table 4.8:** Hardness value (HV) of carburized mild steel using 5% pulverized snail shell and 95% hardwood charcoal.

Distance (mm) from the case to the core	1hr	2hrs	3hrs	4hrs	5hrs
0.5	411	454	506	520	554
1.0	394	418	458	490	530
1.5	385	391	394	396	441
2.0	310	318	321	357	373
2.5	293	297	300	301	328
3.0	280	284	286	287	307
3.5	277	280	284	286	287

**Table 4.9:** Hardness value (HV) of carburized mild steel using 10% pulverized snail shell and 90% hardwood charcoal.

Distance (mm) from the case to the core	1hr	2hrs	3hrs	4hrs	5hrs
---	-----	------	------	------	------

0.5	427	505	532	543	583
1.0	422	459	526	537	550
1.5	413	429	431	434	457
2.0	413	348	342	343	366
2.5	339	331	333	323	351
3.0	315	322	321	323	325
3.5	310	315	320	321	322

**Table 4.10:** Hardness value (HV) of carburized mild steel using 15% pulverized snail shell and 85% hardwood charcoal.

Distance (mm) from the case to the core	1hr	2hrs	3hrs	4hrs	5hrs
0.5	422	496	501	506	622
1.0	409	422	473	485	591
1.5	365	408	417	434	441
2.0	325	377	374	377	435
2.5	320	337	342	350	370
3.0	310	320	335	337	351
3.5	303	310	320	325	337

**Table 4.11:** Hardness value (HV) of carburized mild steel using 20% pulverized snail shell and 80% hardwood charcoal.

Distance (mm) from the case to the core	1hr	2hrs	3hrs	4hrs	5hrs
0.5	444	594	612	644	675
1.0	441	493	462	568	594
1.5	420	437	444	517	476
2.0	400	425	428	413	429
2.5	387	383	401	385	409
3.0	317	323	326	385	393
3.5	311	317	323	365	370

**Table 4.12:** Hardness value (HV) of carburized mild steel using 25% pulverized snail shell and 75% hardwood charcoal.

Distance (mm) from the case to the core	1hr	2hrs	3hrs	4hrs	5hrs
0.5	575	627	638	690	763
1.0	554	572	557	570	643
1.5	476	499	505	550	587
2.0	473	442	443	432	467
2.5	418	383	383	415	441
3.0	368	355	365	314	349
3.5	350	353	357	318	314



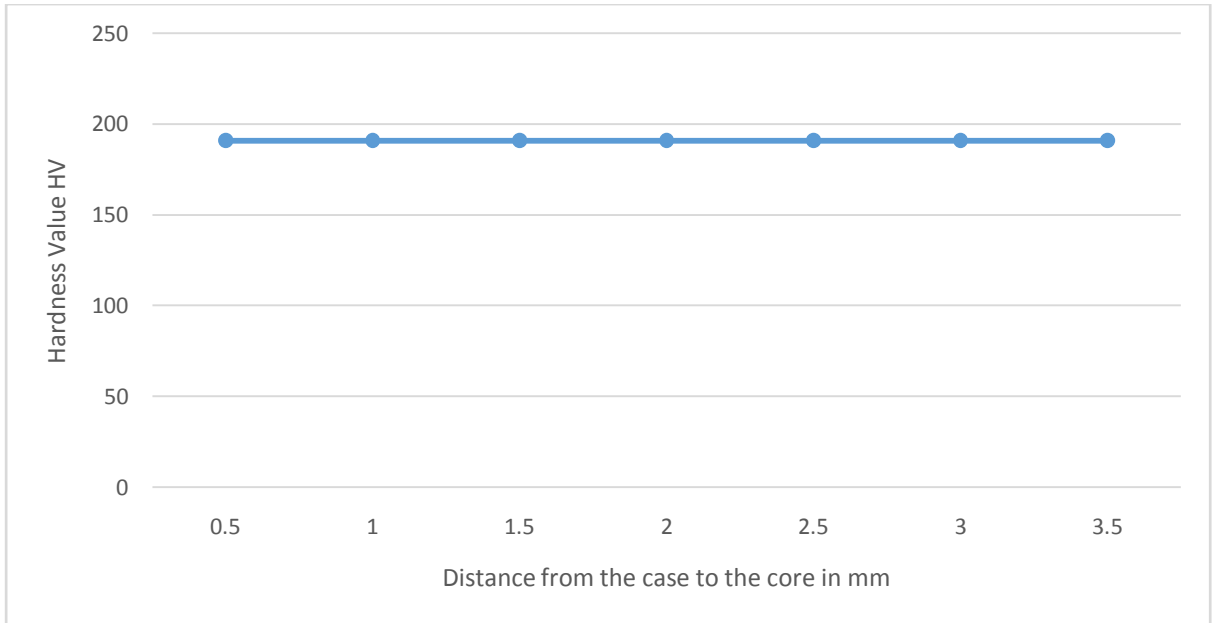
The as-received (control sample) mild steel had the least hardness value of 191HV followed by the carburized steel sample with no pulverized Snail Shell (100% hardwood Charcoal) then that with 5%, 10%, 15%, 20% and then 25% pulverized snail shell. The holding time in the furnace also influenced the carburization characteristics of the steel. Displayed in Tables 4.7 - 4.12 is the result obtained by holding the samples at 1, 2, 3, 4 and 5 hrs in the furnace. At a fixed concentration of additives, the hardness value increased with the holding time.

The sample carburized for 5 hrs with the concentration of 25% pulverized snail shell and 75% hardwood Charcoal had the highest hardness value of 763HV which meant that the hardness increased with increase in soaking time.

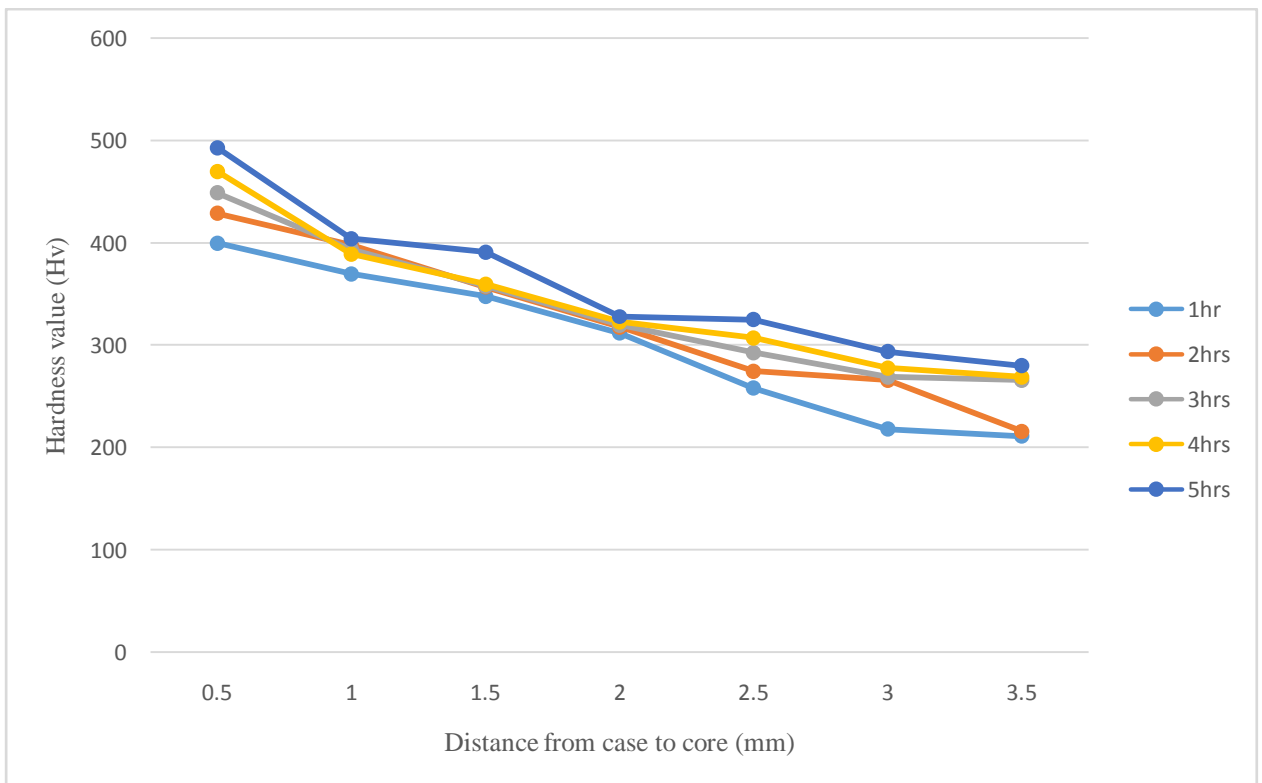
Hence, the above results show that snail shell enhances the carburization of mild steel.

#### **4.5. Case Depth Determination**

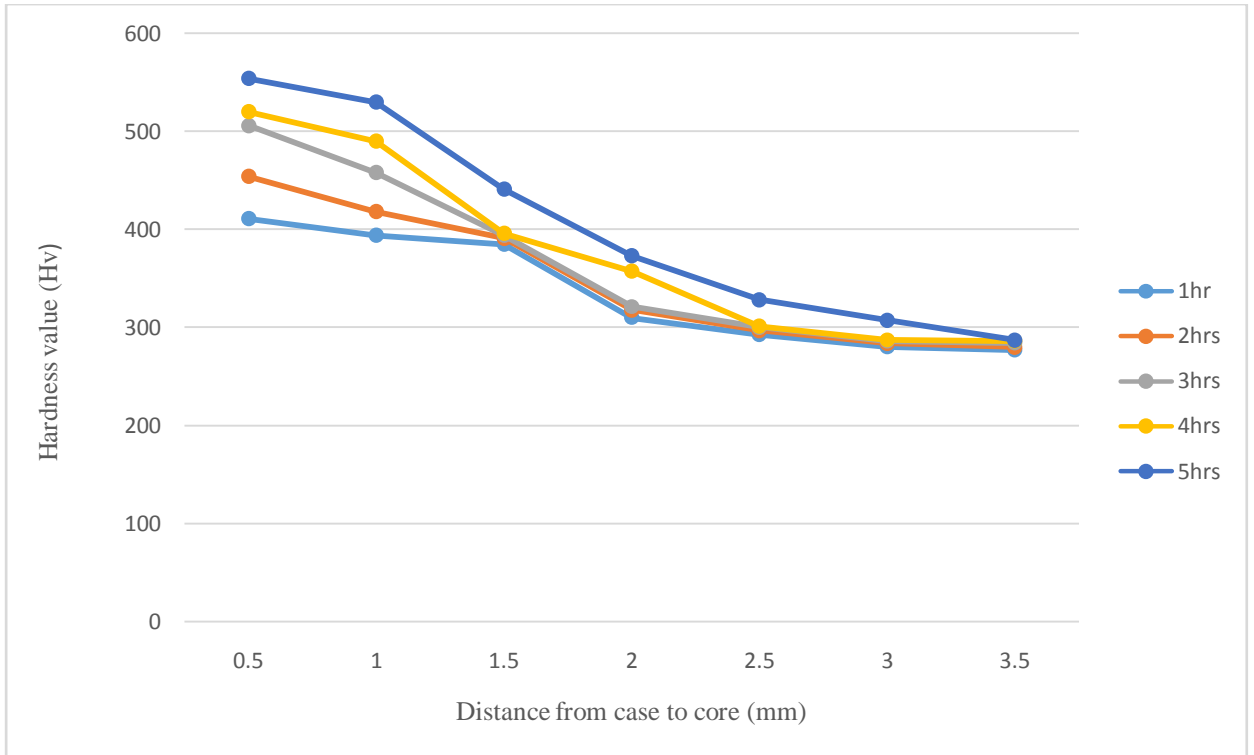
Figure 4.2– 4.8 is the graphical representation of the data presented in Table 4.6 – 4.12 which is a plot of hardness profiles against distance at various compositions of the carburizing compound. The graphical determination of the case depth was based on 50% martensite, 50% pearlite phases commonly taken at 550Hv (Rajan *et al.* 1994). Values of case depth at 1, 2, 3, 4, and 5 hrs for a given composition are displayed in Tables 4.13 to Table 4.17.



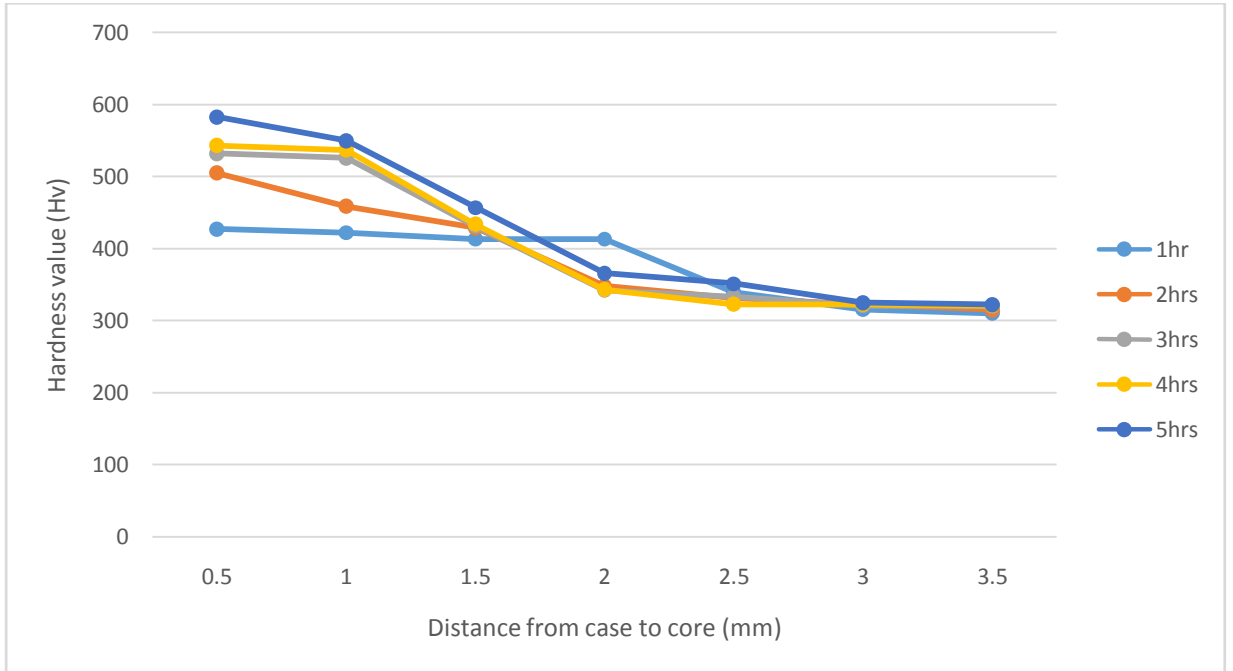
**Figure 4.2:**Hardness profile (Hv) of the untreated steel



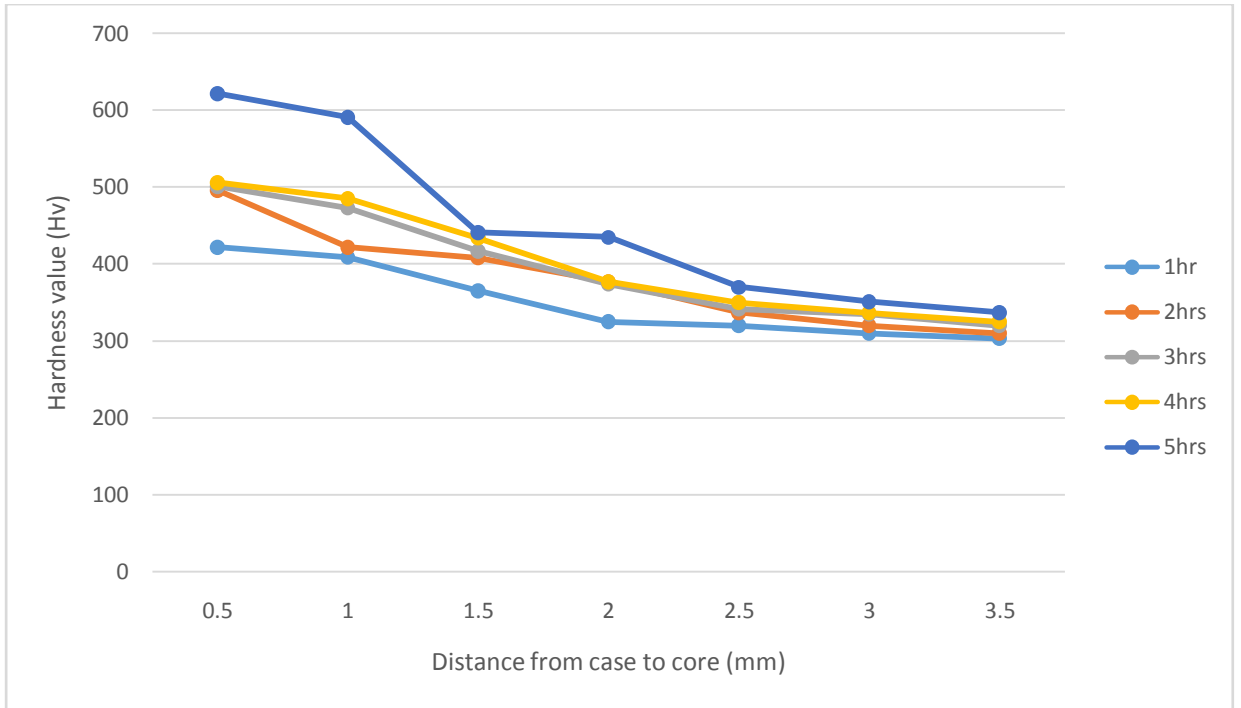
**Figure 4.3:**Hardness profile (Hv) against distance from case to core for the 100% charcoal and 0% snail shell carburizing from 1hr to 5 hrs of carburizing



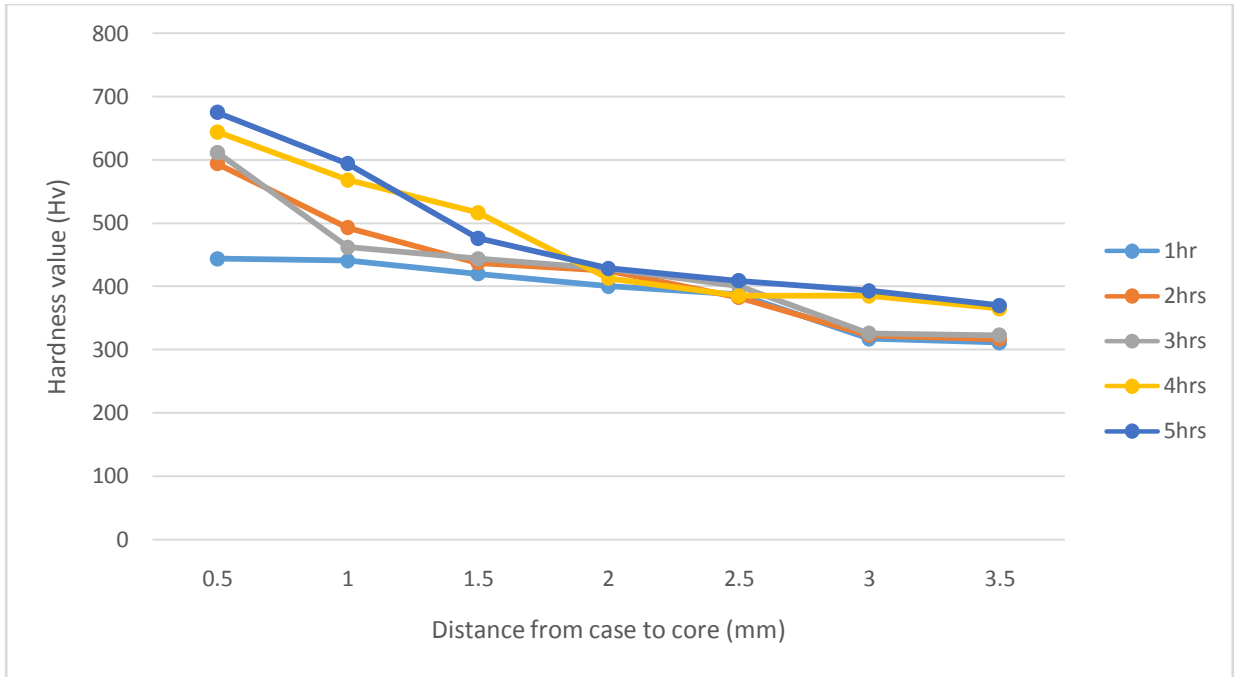
**Figure 4.4:** Hardness profile (Hv) against distance from case to core for the 95% charcoal and 5% snail shell carburizing from 1hr to 5 hrs of carburizing



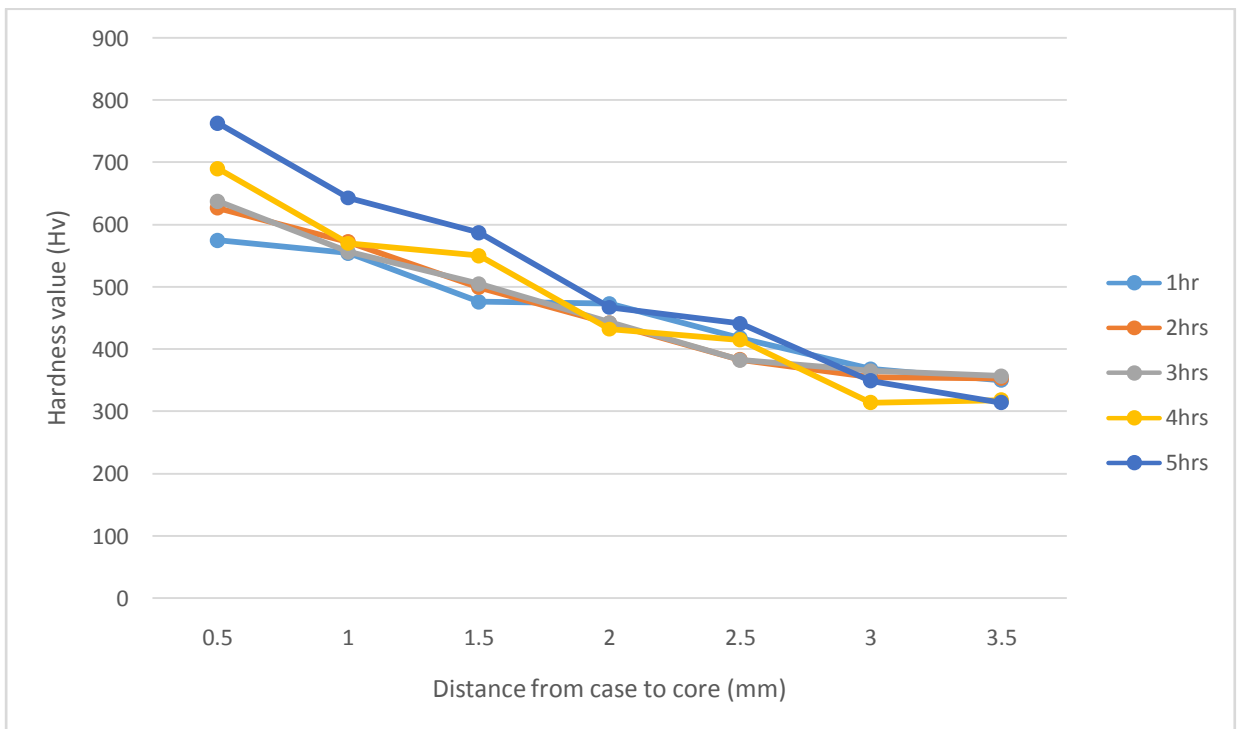
**Figure 4.5:** Hardness profile (Hv) against distance from case to core for the 90% charcoal and 10% snail shell carburizing from 1hr to 5 hrs of carburizing



**Figure 4.6:** Hardness profile (Hv) against distance from case to core for the 85% charcoal and 15% snail shell carburizing from 1hr to 5 hrs of carburizing



**Figure 4.7:** Hardness profile (Hv) against distance from case to core for the 80% charcoal and 20% snail shell carburizing from 1hr to 5 hrs of carburizing.



**Figure 4.8:** Hardness profile (Hv) against distance from case to core for the 75% charcoal and 25% snail shell carburizing from 1hr to 5 hrs of carburizing.

Figure 4.2 – 4.8 showed that with the increase in carburizing time, the effective case depth increases from 0.5mm to 1.5mm. This was also influenced by the increase in the percentage of pulverized snail shell which serves as the energizer to the carburizer. Also from the graphs, the sample treated with 25% snail shell for 5 hours had the highest case depth. This is in line with the report given by Ihom, (2013).

**Table 4.13:**Case depth (mm) measured at 550Hv and 920°C carburization temperature with composition of 95% charcoal and 5% snail shell

Time (Hrs)	1	2	3	4	5
Case depth (mm)	Nil	Nil	Nil	Nil	1

**Table 4.14:**Case depth (mm) measured at 550Hv and 920°C carburization temperature with composition of 90% charcoal and 10% snail shell

Time (Hrs)	1	2	3	4	5
Case depth (mm)	Nil	Nil	Nil	0	1.1

**Table 4.15:**Case depth (mm) measured at 550Hv and 920°C carburization temperature with composition of 85% charcoal and 15% snail shell

Time (Hrs)	1	2	3	4	5
Case depth (mm)	Nil	Nil	Nil	Nil	1.5

**Table 4.16:**Case depth (mm) measured at 550Hv and 920°C carburization temperature with composition of 80% charcoal and 20% snail shell

Time (Hrs)	1	2	3	4	5
Case depth (mm)	Nil	Nil	Nil	Nil	Nil

Case depth (mm)	Nil	0.5	0.75	1.5	1.5
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**Table 4.17:**Case depth (mm) measured at 550Hv and 920°C carburization temperature with composition of 75% charcoal and 25% snail shell

Time (Hrs)	1	2	3	4	5
Case depth (mm)	0.6	1.1	1.2	1.5	1.6

The case depth increases with holding time and the concentration of the snail shell and the maximum was attained at 25% snail shell.

Figures 4.2 to 4.8 shows the hardness profiles obtained with the various carburizing materials. It was from these profiles that the effective case depths were extracted. All the hardness profiles have the same trend rising on the left and descending on the right indicating that the hardness of the carburized steel specimens decreases from the case to the core of the steel. This is typical of all carburized steels; a hard case and a soft core (Ihomet *al.*, 2012; Ihom, 2013). The plots showed that Figure 4.3 at 0.5 mm had hardness value of 493Hv and at 3.5 mm a hardness value of 280 Hv. Figure 4.4 at 0.5 mm had hardness value of 554Hv and at 3.5 mm a hardness value of 287Hv. Figure 4.5 at 0.5 mm had hardness value of 583Hv and at 3.5 mm a hardness value of 322. Figure 4.6 at 0.5mm had hardness value of 622 and at 3.5mm a hardness value of 337..Figure 4.7 at 0.5mm had hardness value of 675 and at 3.5mm a hardness value of 370.Figure 4.8 at 0.5mm had hardness value of 763 and at 3.5mm a hardness value of 314. This profile is higher than the other four profiles. All the hardness values showed a drop in hardness as measurement was taken towards the core of the steel. 75wt% charcoal/ 25wt% snail shell had the highest hardness value for both the 0.5 and 3.5

mm measurement. This is an indication that 25wt % snail shell is the best composition to use when using snail shell as an energizer. It is better than all the other percentages. From Table 4.13 to Table 4.17 it was observed that as the weight percent of carbon increases the case depth of the samples also increases. The presence of carbon increases the hardness of the steel, hence increasing the case depth. This is in line with earlier observation that the higher the surface carbon after carburizing, the higher the hardness values.

From the theoretical case depth obtained using Ficks'law (equation 2.10), the case depth was estimated to be:

$$\text{Case depth (d)} = K\sqrt{t} \quad (4.1)$$

Where, d = total case depth,

t = carburizing time in hours = 5hours

k = depth factor in mm / = 0.6 at 920°C

Inserting the above data in Equation above, the theoretical total case depth:

$$\begin{aligned} \text{Case depth (d)} &= 0.6\sqrt{5} \\ &= 1.34\text{mm} \end{aligned}$$

The diffusion of carbon proceeds from the higher concentration at the workpiece surface to the lower concentration at the core (Ihomet *al*, 2005; Okongwu, 1989).

When consistent concentrations (surface and core) are maintained, the depth of case may be predicted for any constant temperature of operation, for this work the total case depth was predicted to be 1.34mm based on the input data provided above.

#### 4.6 Comparison of snail shell with other energizers

**Table 4.18:** Comparative values of different energizers.

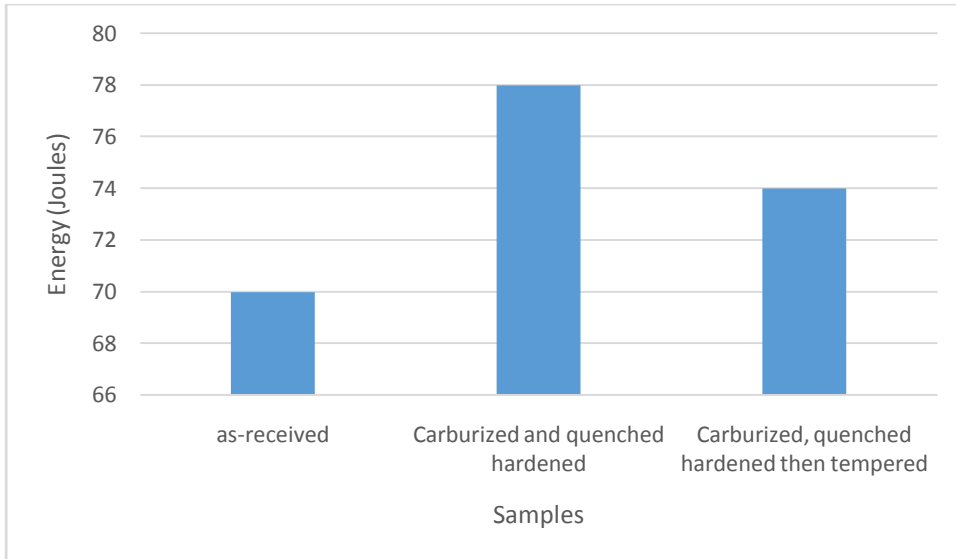
	<b>Optimum Hardness values(Hv)</b>	<b>Case depth(mm)</b>	<b>Impact Energy(J)</b>	<b>Steel Carburized with Materials</b>



<b>Aramideet <i>al</i>(2010)</b>	343.2	---	60.4	Activated carbon
<b>Ihom(2013)</b>	750	2.32	----	Cowbone and wood charcoal
<b>ASM handbook</b>	620-772	125 $\mu$ m-1.5 mm		Carbon
<b>Priyadarshiniet <i>al</i>(2014)</b>	294	----	-----	Carbon
<b>HeshamElzanaty(2014)</b>	551 – 694	--	-----	Activated carbon
<b>Emmanuel Jose Ohize(2014)</b>	724.75-865	---	7.0-14.0	Wood, bone charcoal and coal
<b>Ihom and Offiong(2014)</b>	830-900	1.8	-----	
<b>Present work</b>	<b>763</b>	<b>1.5</b>	<b>74</b>	<b>snail shell and Hardwood charcoal</b>

#### **4.7 Impact Energy of as-received, carburized and quenched hardened and carburized, quenched hardened and tempered**

W&T Avery Izod impact testing machine (type 6701) s/no E51425/9) was used to conduct impact energy test energy on the as-received, carburized and quenched hardened and carburized, quenched hardened then tempered mild steel samples carburized using carburizing compound with ratio 75% pulverized charcoal to 25% pulverized snail shell at 920°C for 5 hours. The result obtained is shown in Figure 4.9 and compared favourably with the result obtained by Atandaet *al* (2009). This shows increase in the impact energy of the mild steel when subjected to carburization and heat treatment.

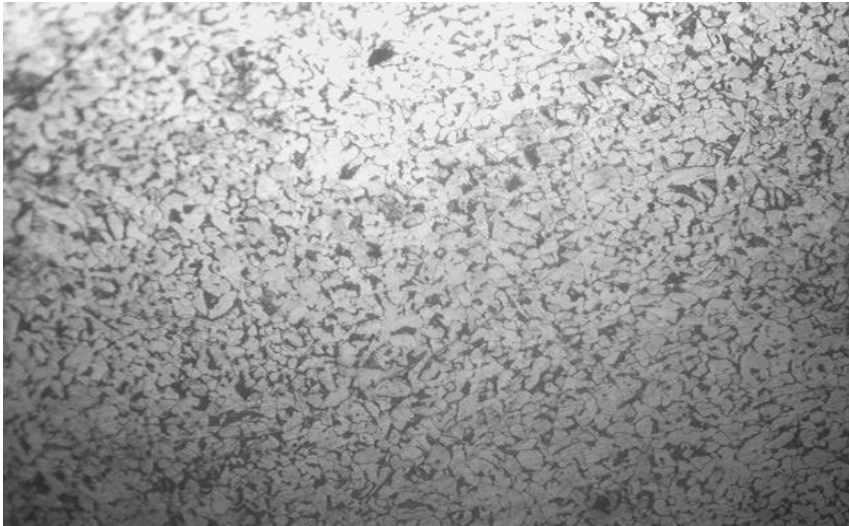


**Figure 4.9:** Impact strength for the as-received, carburized and quenched hardened and Carburized, quenched hardened and tempered steel sample using 75% charcoal and 25% snail shell.

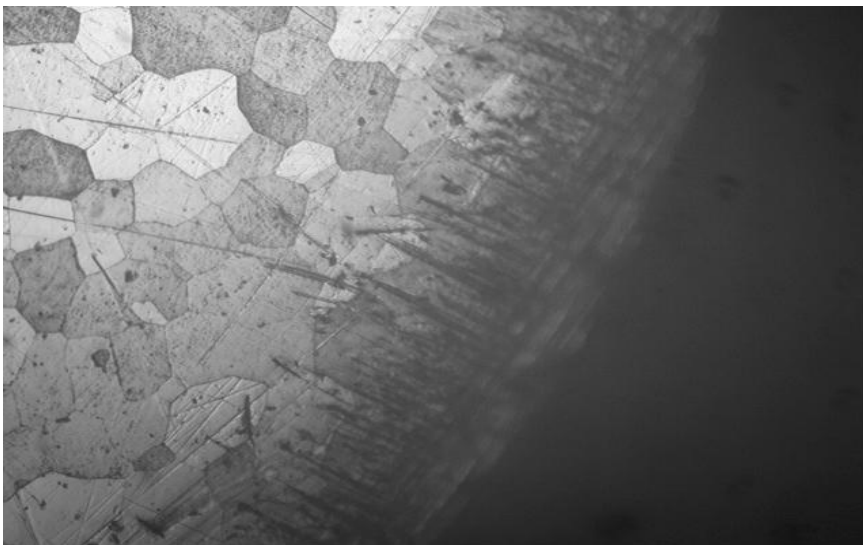
Impact strength test was conducted for the as-received, carburized and quenched hardened and Carburized, quenched hardened and tempered mild steel sample which was carburized using carburizing compound of 25% snail shell to 75% charcoal at 920°C. The result as shown in Figure 4.8 shows that there is an increase in the impact energy of the steel when subjected to carburization then quenched hardened which means that the specimen became more brittle and less ductile as a result of the presence of martensite. But as the sample was subsequently tempered there is a decrease in the impact energy (increased toughness and ductility) of the steel and the specimen with lowest absorbed energy means it's ductile and has highest toughness which can withstand the sudden high loads which is in this test that was carburized.

#### 4.8 Microstructure

The microstructure of the as – received mild steel is shown in micrograph 4.1. The micrograph showed the presence of pearlite (dark) in large ferrite structure (white). This is another confirmation that the steel used in the research is a mild carbon steel (Singh, 2004).



**Micrograph4.1.** Microstructure of the as-received (untreated)mild steel with a pearlite structure in a Ferritic Matrix,(nital etched, 100x).



**Micrograph4.2:** Microstructure of pack carburized bright mild steel, 0.155% carbon treated with snail shell as energizer, carburized to produce a satisfactory case with a cementite network obviously indicating increased carbon content at the surface, 100x, (Etchant: Nital).

Micrograph 4.2 is a micrograph of carburized sample. The sample is selected from the many samples treated and this particular sample has composition of 75% pulverized charcoal and 25% pulverized snail shell at 950°C for five hours. The area captured in

the micrographs is within the compound layer and the diffusion layer, precipitates can be seen in the structure. Micrograph 4.2 shows the presence of pearlite and ferrite with a coarse grain size, it also clearly indicated that there was indeed a change in the case area of the mild steel which is reflected in the increased hardness of the carburized specimen.

## **CHAPTER FIVE**

### **5.0 CONCLUSION AND RECOMMENDATIONS**

#### **5.1 Conclusion**

The suitability of snail shell as energizers in pack carburization has been examined, based on the research, the following conclusions made:

- i. The addition of snail shell to charcoal has produced a significant increase in the case depth of mild steel to 1.5mm which shows that snail shell acted as an energizer during carburization therefore snail shell contains  $\text{CaCO}_3$  and can be used as an energizer, and it's readily available.
- ii. Carburizing compound made up of 75% charcoal and 25% snail shell produced an optimal carburizing effect and gave a high effective case depth of 0.6, 1.1, 1.2, 1.5 and 1.6 mm at 1, 2, 3, 4 and 5 hours of carburizing time at  $920^\circ\text{C}$ .
- iii. Impact test for the carburized quenched - tempered mild steel samples using carburizing compound ratio 75% pulverized hardwood charcoal to 25% pulverized snail shell at  $920^\circ\text{C}$  gave impact strength of 74 Joules. Also the

hardness test conducted on the same sample gave a hardness value of 763Hv at the case and 314Hv at the core which was better than that conducted for the as-received sample with 191Hv.

- iv. Microstructural examination reveals the presence of pearlite in ferrite matrix in the as-received state while the carburized showed a satisfactory case with cementite network indicating increased carbon content at the surface.

## **5.2 Recommendation**

Based on the findings of this work, the following recommendations are offered:

- i. Pulverized hardwood charcoal and pulverized snail shell powder can be effectively used as carburizing materials in the ratio of 75% pulverized hardwood charcoal to 25% pulverized snail shell at 920°C for 5 hours carburizing conditions.
- ii. Studies should be carried out on similar work by varying the particle size of the pulverized snail shell and that of the charcoal.
- iii. Other energizers can be mixed together with the pulverized snail shell in order to improve/enhance its effectiveness.
- iv. Further studies can be carried out by varying the temperature as well as increasing the soaking time of the mild steel.



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