

THE USE OF SELECTED BIOMATERIALS AS ENERGISER IN SURFACE HARDENING OF MILD STEEL

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ABSTRACT

Surface hardening is an essential heat treatment given to gears, shafts, cutting tools and other machine components, to impart on them a good wear resistance surface. The kinetics of the depth of diffusion of carbon in mild steel is usually enhanced by the addition of an energizer such as barium carbonate (BaCO_3) in the hardening mix. Barium carbonate is not locally available in Nigeria. Investigation were therefore carried out to appraise some agricultural waste: cow bones, coconut shells and sea shells for use as energizers in surface hardening of mild steel. Packed-carburization technique was used and various compositions of the carburizing mixture were investigated. At 900°C , optimal values of surface hardness were obtained at 20% cow bone and 30% sea shell additions. Coconut shell did not seem to significantly influence the hardening process at any level of addition. The results showed that cow bone was 65.5% as efficient as BaCO_3 , while sea shell was 82.5% efficient. This paper presents the effects of energizer concentration, chemical compositions and holding time on the kinetics and quality of the hardened products.

INTRODUCTION

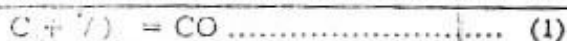
The production of agricultural wastes is an integral part of any agro-industry, such as food, breweries, tobacco, etc. It is desirable that use be found for such wastes, or they be properly disposed of. Some of the wastes include coconut shells, sea shells and cow bones.

Barium carbonate (BaCO_3), sodium carbonate (Na_2CO_3), and

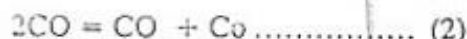
potassium carbonate (K_2CO_3) have been used as energizers to enhance the kinetics of carburization (McGannon, 1971; Gulyeav, 1980). Hardwood charcoal, which is a major component of the carburization compound, is abundant and can be sourced locally from burning of firewood. Unlike charcoal, the energizers such as BaCO_3 , is difficult to source locally and is therefore imported. The foreign exchange requirement for importation of the raw material (energizers) hinder the development of small scale foundry industries and heat treatment shops in Nigeria. It is therefore necessary to find alternative energizer(s) to encourage the growth of the foundry industries and heat treatment shops.

Some of the possible energizers include limestone (CaCO_3), and other materials of high carbonate content such as sea shells, coconut shells and cow bones. Cow bone can be sourced almost free of charge in many parts of the country at slaughter houses (abattoir). Sea and coconut shells can be sourced at riverine areas and equatorial forest zones. They are renewable sources unlike rock mineral energizers. Research was therefore carried out with a focus on cow bone, coconut shell and sea shell for possible use as energizers in small foundries. The work covers the characterization, the effect of energizer concentration, chemical composition and holding times on the kinetics and quality of the hardened products.

Generally, carburization compound consists of a mixture of hardwood charcoal and an energizer such as BaCO_3 . At carburization temperatures of about 900°C and 950°C , entrapped air within the charcoal lumps reacts to form carbon monoxide:



Carbon monoxide is unstable at the process temperature and thus decomposes according to the equation:



The atomic carbon is quickly absorbed at the metal surface and diffuses into the metal. The cycle is repeated with more of the charcoal reacting to produce more carbon monoxide:



The process results in the increase of the surface concentration of absorbed atomic carbon.

The amount of atmospheric oxygen in a charcoal packing varies and may be insufficient to produce enough carburizing gas (CO). It is, therefore, necessary to provide more oxygen to enable the production of sufficient CO, in accordance with equation (1). Generating sufficient amount of CO, as shown in equation (3), also enhance the production of CO. However, a mole of C gives rise to two moles of CO according to equation (3).

The physical chemistry of carbonates reveals that most carbonates decompose to metal oxides and carbon dioxide on heating at high temperatures. From available data, the minimum decomposition temperature of the commonly available carbonates fall below the process temperature of carburization (McGannon, 1971; Weast, 1976).

It is, therefore, possible to generate CO by decomposing carbonate salts in the carburizing compound. This gas will react with charcoal according to equation (1). The simplicity of the decomposition process and the relative abundance of carbonate salts favour the use of carbonates as energizers.

MATERIALS AND EXPERIMENTAL PROCEDURES

2.1 CHEMICAL CHARACTERISTICS OF THE RAW MATERIALS

(a) Coconut Shell

Coconut shell is classified as a polysaccharide with molecular formula $C_6H_{10}O_5$ and chemical composition on dry basis, consisting of approximately 36.51% lignin, 33.61% cellulose, 29.27% pentosan and 0.61% ash (woodroof, 1979). Coconut shell is

similar to hardwood and contains little or no oil and resinous matter.

(b) Sea Shell

Sea shells are the calcified covering of most mollusks and composed of conchiolin which is mainly a mineral salt of calcium carbonate ($CaCO_3$) with traces of magnesium carbonate. Analysis of sample collected from Calabar, Cross River State, Nigeria gave the following composition: 4.07% SiO_2 , Al_2O_3 , NO_3 , 0.08% Fe_2O_3 , 88.46% $CaCO_3$ and 0.96% MgO (Ekeshili, 1991).

(c) Cow Bone

Cow bone, on the other hand, consists of collagen fibres which are organised in layers upon which mineral salts especially calcium phosphate and carbonates are impregnated. The chemical composition of bones are given as follows: organic component (collagen, mucopolysaccharides, elastin and fat) 30%, inorganic calcium phosphate 38.25%, calcium carbonate and trace ions 6.75% and water 25% (Parker, 1989). Hence cow bone consists of both organic and inorganic compounds.

(d) Mild Steel

Mild steel RST37 was obtained from Jos Steel Rolling Company, Jos with the following composition:

C	Si	Mn	P	S	Cr	Ni	Cu	Fe
0.15	0.21	0.51	0.022	0.006	0.02	0.02	0.07	Balance

The above composition is closely approximated by SAE 1015 specification. The sea and coconut shells were obtained from Cross River State. The cow bone was obtained from the slaughter houses at the Makurdi Modern Market, Benue State.

2.2 EQUIPMENT

The carburizing boxes with dimensions 80 x 80 x 70mm were made of heat resistant steel. An electric furnace of type OH85TR, made in Hungary was used for the heat treatment. It has a temperature sensitivity of $\pm 0.5^\circ C$. A micro-hardness (Vickers) testing machine, Model MHT-1 manufactured by Matsuzawa Seiki Company of Japan was used. Olympus Metallurgical microscope also made in Japan was used for the microstructural observation.

2.3 PROCEDURE

Hardwood charcoal, the shells and the cow bone were ground and sieved to particle size of 80% finer than

425 microns. They were then mixed at predetermined proportions as shown in Tables I, II and III. The mild steel rods were cut into 30mm lengths, thoroughly washed and dried.

The bottom of the carburizing box was covered with a layer of the carburizing compound chosen from Tables I, II, and III. A single specimen was placed in each box and the remaining space was filled with the carburizing compound. The box was then covered with a lid and sealed with fire clay to prevent infiltrate air from entering the box during the carburizing process. The box was then placed in the central zone of the furnace which was found to have a constant temperature.

Carburizing times of 2, 4, 6 and 8 hours were used. At the end of each carburizing time, the box was removed from the furnace and quenched in water at room temperature (about 27°C). Each set of test was repeated at least three times. The samples were prepared for microhardness and microstructural analysis.

3. RESULTS AND DISCUSSION

A. Effect of Energizer Concentration on Carburization Time.

At constant temperature, carburizing time, and at a fixed distance from the surface of the specimen, the carburizing kinetics depends almost entirely on the concentration of the carburizing medium. The effect of varying the amount of the coconut shell, sea shell and cow bone is presented in the following section.

Sea Shell

The hardness of the uncarburized steel was taken at 0.2mm interval and the mean value was HV 223.95 with a standard deviation of 0.60. The profile was taken from the edge to 4mm depth. Table IV is a summary of the results of hardness measurements on carburized samples, using charcoal and sea shell. The results show that as the amount of sea shell increases, the rate of carburization also increases. It is however observed that an optimal value occurred at 30% sea shell and 70% charcoal. Above 30% sea shell addition, the hardness value dropped.

Cow Bone

Table V is a hardness profile on carburized samples using charcoal and cow bone. The same trend is observed except that the optimal value occurred at 20%

cow bone and 80% charcoal. Above 20% cow bone addition the hardness value dropped.

Coconut Shell

Table VI summarises the results using various concentrations and coconut shell and charcoal. It can be observed that there is no significant increase in the hardness values at any given depth, due to increase in the coconut shell concentration. At 100% coconut shell, the hardness profile was not different from those obtained at 100% charcoal. The result shows that the coconut shell was not an effective energizer, but behaved as charcoal. This confirms the observations by Woodroof (1979) that coconut shell is similar to hardwood.

B. The Effect of Holding Time on Carburization

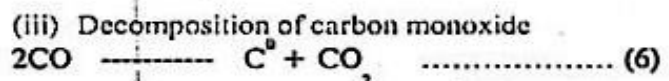
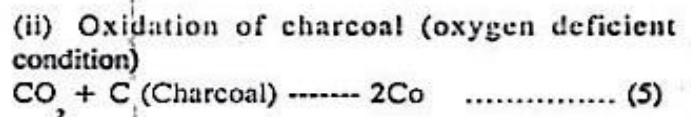
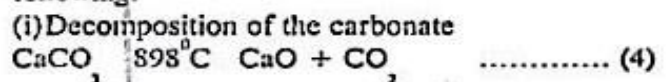
Displayed in Tables VII and VIII are the results obtained by holding the sample at 2, 4, 6 and 8 hours for sea shell and cow bone respectively. At 20% cow bone concentration and fixed holding time, the hardness decreases with increase of distance from the surface towards the centre of the specimen as shown in Table IX. Hardness also increases with holding time at constant concentration and constant depth. Similar observation were made with respect to sea shell.

C. Reaction Kinetics

The above results show that sea shell and cow bone enhanced carburization kinetics of mild steel while coconut shell was not effective as an energizer. The difference in behaviour could be due to the reaction mechanism which favour equations (2) and (3) resulting in increased carburization rate.

Sea Shell Reactions

The major reactions from the sea shell include the following:



The ease of generation of CO in equation (4) will result in the increase of the concentration of atomic

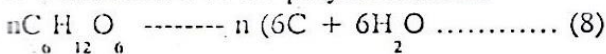
carbon (C²) as shown in equation (6). The more the decomposition of CaCO₃, the higher the degree of carburization. However, it was observed that an optimum carburization occurred at 30% sea shell addition.

A plot of the average case depth against the concentration of sea shell is presented in Figure 1. The hardness values are observed to decrease as the concentration of the sea shell exceeds 30%. This could mean that the furnace atmosphere, was richer in CO from increased decomposition of CaCO₃ giving rise to decarburization. On the other hand, below 30% addition the furnace atmosphere could be deficient in CO and hence slower carburization kinetics. Figure 1 also shows that longer holding times gave higher values of cases depths at all compositions of the carburization compound.

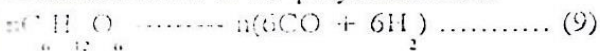
Coconut Shell

From the composition of the coconut shell, two possible reactions can be presented at the reaction temperature.

i. Dissociation of the polysaccharides



ii. Gassification of the polysaccharides



Equation (8) will merely increase the overall carbon content of the carburizing compound and generate water vapour. Since the carbon concentration in charcoal is between 85% - 98% (McGraw Hill, 1989), it can be considered to be supersaturated. Hence additional carbon produced due to equation (8) above will not significantly influence the reaction rate, if other conditions remain constant. This analysis confirms the result shown in Table (V).

Equation (9) would support increased carburization kinetics but was not thermodynamically favoured at the process temperature. It was also observed that the tests using coconut shell generated severe scaling problem on the specimen and the carburization box. This observation supports the production of H₂ instead of H₂ in the decomposition.

Cow Bone

Since cow bone consists mainly of polysaccharides 30%, inorganic phosphate 38.25%, calcium

carbonate and trace ions 6.75%; the following reactions are possible:

- (i) The dissociation and gassification of the polysaccharides as shown in equation (8) and (9).
- (ii) The decomposition of the phosphates. This is impossible at the process temperature (950°C) since the orthophosphate will only melt at 167°C.
- (iii) Decomposition of the carbonates shown in equation (4).

Reaction (i) above will not significantly increase the kinetics of carburization, as discussed in the section under coconut shell. The phosphates will also not influence the kinetics of carburization since they are not dissociated at the process temperature (950°C).

The decomposition of the carbonates as shown in equation (4) will increase the carburization kinetics, similar to values shown in Table (V) and Figure (2). The explanation given for the sea shell will also be valid in this case.

The effect of holding time is almost the same as that observed for sea shell. However a second peak was observed at 35% cow bone. This second peak has a lower hardness value than the peak at 20% cow bone.

Case Depth:

The American Society for Metal (ASM) defines effective case depth as HRC:50 or HV 550 on Rockwell and Vickers Hardness Scales respectively (Boyer, 1985). Values of case depth at 2, 4, 6 and 8 hours are displayed in Tables (X) and (XI).

Table (X) - Case depth (mm) measured at HV 550 and at 950°C carburization temperature.

Time = 2 hours

% Sea Shell	0	10	20	30	50
Case Depth (mm)	0.55	0.90	1.00	1.45	1.30

Table (XI) - Case depth measured at HV 550, 950°C carburizing temperature at different holding times.

Composition of Carburizer	30% Sea Shell				20% Cow Bone				100% Charcoal	100% Coconut Shell
	2	4	6	8	2	4	6	8	8	8
Holding time (hour)	2	4	6	8	2	4	6	8	8	8
Case depth	1.45	1.95	2.60	2.80	1.82	2.09	2.28	2.60	1.10	1.10

It is of interest to note that the case depth obtained at 100% charcoal coincided with that of 100% coconut shell after eight hours of carburization. The hardness profile in both cases were identical within the limits of experimental error.

The Diffusion coefficient of carbon in steel (D_c) is also given by the equation below (Shackelford; Geiger 1973):

$$D = D_0 e^{-\frac{Q}{RT}} \dots\dots\dots (10)$$

where Q is the activation energy and R is the universal gas constant. Diffusion coefficient (D_c) of carbon in austenite is given as 1.8×10^{-7} cm²/sec and 1.3×10^{-7} cm²/sec, by Geiger and Poirier (1973) and West (1976), respectively.

The average value of D_c was determined to be 2.1×10^{-7} cm²/sec using data and case depth for 100% charcoal samples. This value of D_c for carbon diffusion in austenite is in agreement with published values. Using the case depth at two hours and 30% sea shell, the corresponding diffusivity value was 1.46×10^{-6} cm²/sec, which compares with 1.7×10^{-6} cm²/sec and 3.6×10^{-6} cm²/sec reported by Okongwu and Paranthaman (1987) and Read-Hill (1973), respectively. The value reported by Okongwu and co-worker was based on use of energizers. For the cow bone, the diffusivity was found to be 2.3×10^{-6} cm²/sec which compares with the above results.

Comparison of Sea Shell, Cow bone with other energizers.

The effective case depth obtained using other energizers is given in table (XII) for 2 hours carburization.

Table (XII): Comparative values of different energizers.

Energizer	30%	20%	20%	20%	20%
	Sea Shell	Cow bone	Lime Stone	Dolomite	BaCo3
Case Depth	1.45	1.82	1.08	0.36	1.76
Efficacy	2.64	2.10	1.96	0.65	3.20
Efficiency	82.40	65.60	61.40	20.45	100

The efficiency was determined using BaCO₃, as the reference since it has the highest efficacy.

The table above indicates that BaCO₃ is the best energizer followed by Sea Shell which is 82.40% as

efficient as BaCO₃. This is followed by cow bone. Coconut shell is not included in the above table because it showed similar behaviour like charcoal, i.e. not an effective energizer.

Efficacy = Case depth with energizer / Case depth at 100% charcoal.
(Time and temperature are kept constant).

CONCLUSIONS

The kinetics of selected Agricultural wastes in surface hardening of parts has been examined and the following conclusions are made:

1. The addition of sea shell, and cow bone to charcoal can produce a significant increase in the carburization rate of mild steel.
2. The carburization compound made up of 30% sea shell and 70% charcoal produced an optimal carburizing effect at constant temperature and holding time.
3. At constant temperatures and holding time 20% cow bone and 80% charcoal will produce optimal carburizing effect.
4. Case depth increase with holding time at all compositions of the carburizing compound.
5. The hardness profile of coconut shell is identical with that of charcoal, which implies that coconut shell was not an effective energizer.
6. Effective average case depth 1.45, 1.95, 2.60 and 2.80mm were observed at 2, 4, 6 and 8 hours of carburization with 30% sea shell mixture at 9000C. At 20% cow bone, the depth were 1.82, 2.09, 2.28 and 2.60mm observed at 2, 4, 6 and 8 hours respectively.
7. Severe scaling and corrosion problems occurred with coconut shell and cow bone specimen.
8. Sea shell compares favourably with BaCO₃ as energizer giving a relative efficiency of 82.5% when compared with BaCO₃.
9. Cow bone can also be used as an energizer and has a relative efficiency of 65.60%. But care must be taken because of the associated corrosion problem.
10. Sea shell is the best substitute for BaCO₃.

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Table I: Carburization Compound using Hardwood Charcoal and Sea Shell

Compound	Composition
1.	100% Charcoal + 0% Sea Shell
2.	90% Charcoal + 10% Sea Shell
3.	80% Charcoal + 20% Sea Shell
4.	70% Charcoal + 30% Sea Shell
5.	60% Charcoal + 40% Sea Shell
6.	50% Charcoal + 50% Sea Shell

Table II: Carburizing Compound using Hardwood Charcoal and Coconut Shell

Compound	Composition
1.	100% Charcoal + 0% Coconut Shell
2.	90% Charcoal + 10% Coconut Shell
3.	80% Charcoal + 20% Coconut Shell
4.	70% Charcoal + 30% Coconut Shell
5.	60% Charcoal + 40% Coconut Shell
6.	50% Charcoal + 50% Coconut Shell
7.	0% Charcoal + 100% Coconut Shell

Table III: Carburizing Compound using Hardwood Charcoal and Cow bone

Compound	Composition
1.	100% Charcoal + 0% Cow bone
2.	90% Charcoal + 10% Cow bone
3.	85% Charcoal + 15% Cow bone
4.	80% Charcoal + 20% Cow bone
5.	75% Charcoal + 25% Cow bone
6.	70% Charcoal + 30% Cow bone
7.	65% Charcoal + 35% Cow bone
8.	60% Charcoal + 40% Cow bone

Table IV: Effect of Concentration of Sea Shell on the Hardness Profile of Carburized Mild Steel, RST37 at 9500c for 2 hours

Distance (mm)	Hardness Values (HV) at Concentration of Sea Shell (%)					
	0.0	10.0	20.0	30.0	40.0	50.0
0.2	618	705	763	804	798	775
0.4	575	644	725	755	763	757
0.6	543	598	657	746	700	675
0.8	523	547	583	661	652	622
1.0	490	523	547	639	606	579
1.2	481	505	540	606	547	543
1.4	478	490	520	554	533	530
1.6	457	481	502	520	520	517
1.8	422	478	490	514	502	496
2.0	371	457	483	505	487	484
2.2	341	427	476	490	484	481
2.4	312	371	465	481	478	470
2.6	294	341	444	470	457	451
2.8	270	312	406	465	418	400
3.0	244	294	373	444	390	431
3.2	242	260	341	406	364	351
3.4	240	244	312	371	341	328
3.6	240	242	269	323	315	270
3.8	238	240	255	270	252	250
4.0	238	240	242	251	244	240

Hardness value of Carburized Specimens Using Charcoal and Cow Bones.

Table V: Average Hardness Values in HV of Specimens in various carburizing compounds (1 to 8) heated for 2 hours.

S. No	Distance (mm)	90%	85%	80%	75%	70%	65%	60%	
		Charcoal + 100% Charcoal	Charcoal + 10% Cowbone	Charcoal + 15% Cowbone	Charcoal + 20% Cowbone	Charcoal + 25% Cowbone	Charcoal + 30% Cowbone	Charcoal + 35% Cowbone	Charcoal + 40% Cowbone
		A	B	C	D	E	F	G	H
1	0.2	660	705	712	850	614	575	830	771
2	0.4	627	644	649	808	546	546	789	732
3	0.6	596	548	644	767	554	519	749	696
4	0.8	566	524	535	729	526	493	712	661
5	1.0	538	492	507	692	500	468	676	628
6	1.2	511	490	507	658	475	445	643	597
7	1.4	485	470	502	625	451	423	610	567
8	1.6	461	457	502	594	429	402	579	538
9	1.9	438	427	487	564	407	381	551	512
10	2.0	416	406	473	536	387	362	523	460
11	2.2	392	351	461	509	367	344	497	437
12	2.4	362	341	419	483	349	327	472	415
13	2.6	334	301	420	430	332	310	449	394
14	2.8	309	231	383	383	315	295	426	355
15	3.0	208	251	315	341	299	280	384	301
16	3.2	258	244	273	303	369	366	300	257
17	3.4	236	242	244	270	242	230	260	233
18	3.6	214	224	249	240	218	216	230	220
19	3.8	213	222	246	214	218	216	230	220
20	4.0	212	221	245	214	218	216	228	220

Table VI: Effect of Concentration of Coconut Shell on the Hardness Profile of Carburized Mild Steel, RST37 at 950°C for 2 hours

Distance (mm)	Hardness Values (HV) at Concentration of Coconut Shell (%)						
	0.0	10.0	20.0	30.0	40.0	50.0	100.0
0.2	618	618	618	622	622	618	631
0.4	575	579	575	579	575	579	583
0.6	543	547	543	547	543	547	547
0.8	523	523	527	523	627	523	523
1.0	490	490	493	490	493	490	502
1.2	481	484	481	484	481	484	490
1.4	478	478	476	478	476	478	478
1.6	457	459	454	457	459	457	457
1.8	427	427	425	427	425	427	441
2.0	371	373	371	372	371	373	427
2.2	341	344	343	344	343	344	400
2.4	312	312	314	312	314	312	375
2.6	294	294	291	293	291	294	353
2.8	260	270	260	270	260	270	341
3.0	244	245	247	245	247	245	320
3.2	240	244	242	244	242	244	282
3.6	240	242	242	242	242	244	269
3.8	238	242	240	242	240	242	256
4.0	238	240	240	242	240	242	242

Table IX: Hardness value (HV) at optimum addition (20% Cow bone) at different holding times.

Distance (mm)	Hardness (HV) at 20% Cow Bone			
	2 HOURS	4 HOURS	6 HOURS	8 HOURS
0.2	850	900	960	1040
0.4	808	855	912	988
0.6	767	812	866	939
0.8	729	762	823	892
1.0	692	733	782	847
1.2	658	696	753	805
1.4	625	662	706	765
1.6	594	629	670	726
1.8	564	597	636	690
2.0	536	568	605	650
2.2	509	539	575	622
2.4	483	500	546	591
2.6	430	487	518	562
2.8	383	463	492	533
3.0	341	439	468	507
3.2	303	417	448	482
3.4	270	396	422	458
3.6	240	376	401	435
3.8	214	357	381	413
4.0	214	339	362	392

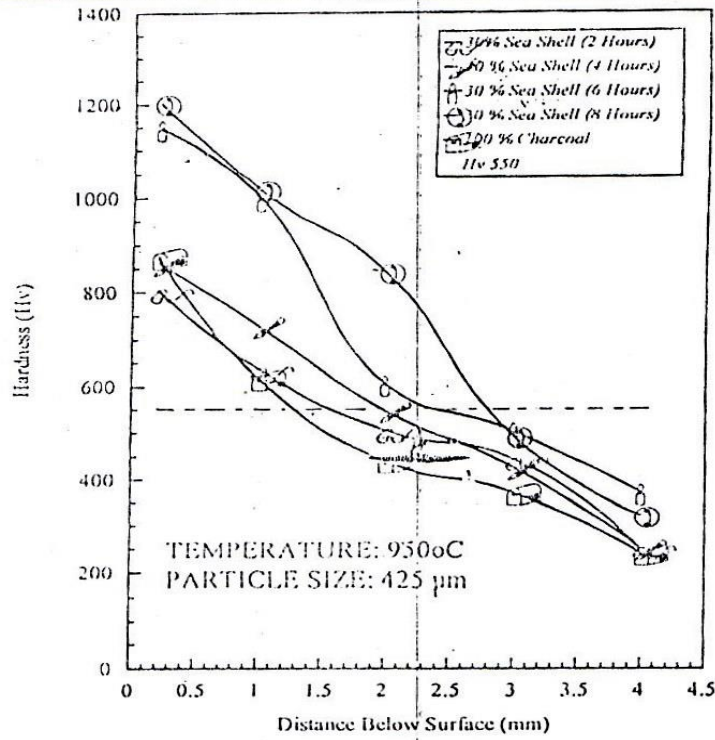


Figure 2: Effect of Holding Time on the Carburizing Kinetics Using 30 % Sea Shell at 950 oC

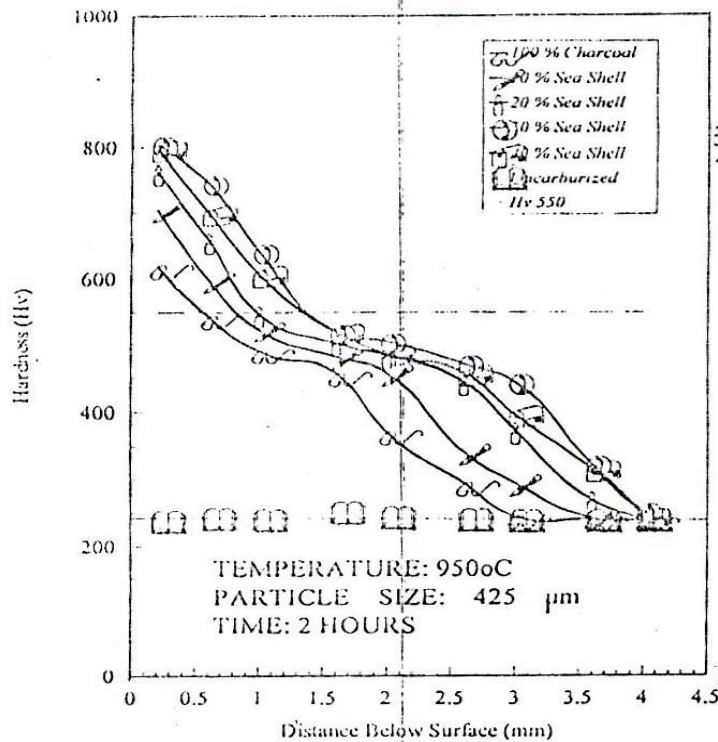


Figure 1: Graphical - Determination of Case Depth using Hardness Profiles and distance data for Different Carburizing Compounds after 2 Hours at 950 oC