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Effect of bio-mineral oil blend quenchant on the mechanical properties of carburized mild-steel

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Abstract

In this study, the effect of bio-mineral oil blend quenchants on the mechanical properties of carburized mild steel was experimentally studied and reported. The tensile, hardness, impact, and microstructural test specimens were prepared in line with ASTM standards. Prepared specimens were then buried in a 50:50% ratio mixtures of egg-shell/date-seed particulates as carburizing medium in a sealed packed cylindrical crucible. The carburization was then carried out in a muffle furnace at 950 oC for 3 h soaking time at 5 °C/min heating rate and thereafter quenched in different percentage blends of bio-mineral oils. Before the mechanical test and microstructural examination, samples were tempered at 200 oC for 1 h.

Results from the experimental findings revealed that water and bio-mineral oil blend quenchants significantly influenced the mechanical properties and microstructure of carburized mild steel in varying degrees depending on the quenching media. Specimen quenched in 100% groundnut oil yielded the maximum yield tensile strength (805.43 MPa) and hardness at the surface edge (173.8 HV) equivalent to 106.7 and 87.66 percentage increment however, the best combination of mechanical properties (tensile strength 738.66 MPa, strain 17.12%, hardness 169.5 HV and impact strength 51.1 J) was obtained in the specimen quenched in 60/40% groundnut oil and SAE40 oil blends respectively. The enhancement in the mechanical property was due to the grain refinement in the microstructure of the bio-mineral oils quenched specimen. The 60/40 groundnut/SAE40 oil blend is therefore recommended for metallurgical heat treatment of mild steel for critical industrial applications.

Keywords: Carburization, Bio-mineral oil blend, Mild steel, Mechanical property, Quenchant

Introduction

Steel is one of the most important engineering materials in today's world with applications in construction, automobile, aviation, machinery, structural, and industrial sectors [1]. Its properties are usually modified to suit a particular critical engineering application by a series of controlled heating and quenching (heat treatment) which can alter the microstructure to enhance a desirable property. For instance, engine, aircraft, and structural components are heat treated for wear, fracture toughness, durability, and strength improvement for better performance [2]. Heat treatment is of various types and the selection of a suitable type is largely dependent on the properties required and desired service application of the materials. Notwithstanding, improvements in properties of alloys of steel such as hardness, wear, and strength are easily achievable through the carburization technique which could be a solid, liquid, or gas-fired [3].

Pack-carburization is a thermo-diffusional heat treatment process that involves the transfer of carbon from a carbon-rich environment such as coke, activated carbon, and carbonaceous agro-materials at a critical temperature onto the surfaces of the exposed mild steel specimen for a specific dwelling time followed by desirable cooling mode to modify the properties of alloy steel. The use of agro-wastes as carburizer has been found suitable in replacement of the expensive and rare conventional types (coke, coal, barium carbonate, and synthetic calcium carbonate) for properties enhancement during pack-carburization of steel alloys as evidenced in the literature [4–10]. This assertion was further substantiated by the findings of Olufemi et al. [11] where carbonized palm kernel shell was used as a carburizer. In their work, the best mechanical properties were reported at 950 °C with a dwelling time of 2 h. Similar conclusions were reached by Umunakwe et al. [7], and Ihom 2013 [4], where they all reported enhanced tensile strength and hardness properties of low carbon steel using agro-waste of palm kernel shell and coconut shell, charcoal, and cow bone as hybrid carburizer. In furtherance of these findings, Kolawole et al. [12] investigated the suitability of a hybrid mixture of date seed and snail shell as carburizer at 800, 900, and 1000 °C on the mechanical properties of water-quenched mild steel. Experimental results indicated a peak tensile strength (521 MPa) and hardness (32 HRB) at 1000 °C carburizing temperature which is equivalent to 51.45 and 31.28% increment relative to the control specimen.

The achievement of a quality and sound steel product during heat treatment such as carburization is majorly dependent on the effective control of the heating temperature, holding time, and cooling rate. The cooling rate is characterized by the types of quenching media as various quenchants have different cooling rates [1, 13]. Conventionally, water and mineral oils are the most commonly used quenchant during heat treatment. However, defects such as cracks and warpage of heat-treated samples are usually associated with the water quenchant and have been a challenge to the metallurgical industries [1]. Recently, the use of oils such as mineral oil (SAE40) as quenching media has been found suitable for the enhancement of mechanical and metallurgical properties of steel [14]. The mineral oil possesses favorable heat extraction characteristics for controlled cooling and wetting of the heat-treated samples during quenching to inhibit the formation of unwanted thermal transformational defects like distortion and cracks which can render the steel products as scrap [15–18]. Notwithstanding, the use of mineral oil as a quenchant also has its limitations such as its relatively expensive, non-renewability after use, and as well as the liberation of dangerous fumes during quenching operations which are dangerous to the environment and human health. Literature has shown that bio/vegetable oils or their equivalent blends can be an alternative and a replacement for mineral oils [19–21]. This is because, bio-oils are less expensive, renewable, and evolved less dangerous gases during quenching and have therefore attracted heat treatment industries and researchers' attention [22]. However oxidative instability, low-temperature characteristics, and short viscosity range of bio-oil quenchant are still other challenges that still need to be addressed. In lieu of these limitations of both the mineral and bio-oils as highlighted, researchers are now diverting attention to hybrid or blending of oils as quenching media for improved performance in heat treatment industries [1, 19]. The cooling effect of palm kernel oil and mineral oil (SAE40) blends as quenching media on the mechanical and microstructure of quenched medium carbon steel was investigated by Agboola et al. [19]. It was observed that the quench severity of pure palm kernel oil is greater than that of SAE40 and consequently samples quenched in pure palm kernel oil have the highest hardness value compared to the least hardness value for SAE-cooled samples. Whereas, the hardness of the treated samples in mineral and palm kernel oil blends possesses an intermediate hardness value. In another work, Rana et al. [1] reported a comparative study on the effect of various quenching media (brine, cotton-seed oil, water, oil-in-water emulsion, and hybrid polymer quenchant) on the mechanical properties of low-carbon steel. It was reported that the best combination of mechanical properties was obtained in the samples quenched in brine solution however this might be susceptible to corrosion attack.

By and large, the assessment of bio-mineral oil blends like SAE40 and groundnut oil blends on the mechanical properties of carburized mild steel is limited to the best of the author's knowledge in the literature. Therefore, this work reports the effect of bio-mineral oil blends at different ratios on the mechanical properties of date seed-egg shell carburized mild steel.

Experimental

Materials and equipment

Mild steel sourced from a local vendor in Lagos, Lagos State Nigeria with the composition presented in Table 1 corresponding to SAE/AISI1016 mild steel grade was used in this study. Date seed was obtained from the Ipata market and egg shells was collected from a snacks baker shop at Kabba, Kogi State, Nigeria. Cylindrical stainless steel crucible, clay slurry, tube furnace, tongs, quench-bath, The equipment used are; a weighing balance, hammer mill, disc mill, blender, Universal Testing Machine (UTM), hardness tester, tube furnace, and the Hounsfield balanced impact machine.

Preparation of date seed–eggshell carburizer

The date seed and eggshell particulates were prepared as per Kolawole et al. [12]. The date seed was de-shelled and washed followed by oven drying at 110 °C for 8 h (8 h) to remove moisture. The dried seed was crushed with a hammer mill, pulverized using a disc mill, and sieved down to 200 µm particle size (Fig. 1a) at the Materials Science and Engineering Laboratory, Kwara State University Malete, Nigeria. The eggshell was converted into a fine powder using a domestic kitchen blender operated for 30

Table 1 Element composition of the mild steel specimen

Elements	C	Si	Mn	S	P	Cr	A1	Fe
Percentage	0.159	0.131	0.565	0.035	0.031	0.144	0.043	Bal

Fig. 1 Pulverization of date seed and eggshell. a$_1$ Date seed with shell. a$_2$ De-shelled date seed. a$_3$ Pulverized date seed. b$_1$ Eggshell. b$_2$ Pulverized eggshell

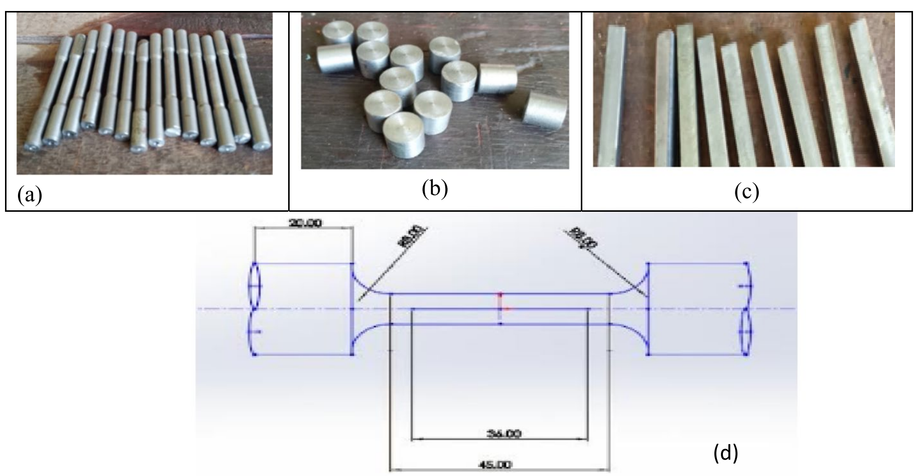


Fig. 2 Sample preparation for mechanical testing

at 15-min intervals. The powder obtained was then sieved down to 200 μm sizes as depicted in Fig. 1b.

Preparation of mild steel specimen for mechanical testing

Samples for tensile strength, impact strength, hardness, and optical examination were prepared from a 500 mm × 12 mm diameter rod of mild steel, with the chemical composition shown in Table 1. Tensile test specimens (9 mm dia) were machined out of this sample according to ASTME8/E8M-16a standard as depicted in Fig. 2a and represented in Fig. 2d, hardness (10 mm height by 12 mm diameter) Fig. 2b and impact energy specimens (10 × 10 × 75) mm with 2 mm depth V-notched shape following

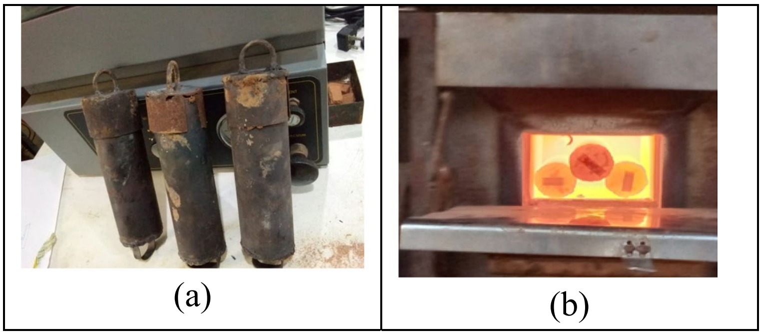


Fig. 3 Process of carburization mild steel

Table 2 Bio-oil formulation for carburized mild steel quenching

Sample	Composition
A	Control sample
B	100% water quenched (WQ)
C	100% SAE quenched (SAEQ)
D	100% groundnut oil quenched (GNQ)
E	70% Groundnut oil-30% SAE quenched (GN + SAE)Q
F	60% Groundnut oil-40% SAE quenched (GN + SAE)Q
G	50% Groundnut oil-50% SAE quenched (GN + SAE)Q

ASTM E23 specification (Fig. 2c) and following earlier report of Oluwafemi et al. [11] and Kolawole et al. [12], were adopted in this study.

Carburization process and quenching operation

The carburizer containing 750 g of the pulverized date seed and eggshell powder in a ratio of 50% each was manually mixed for 5 min to ensure homogeneity. The mixed carburizer was packed into the cylindrical crucible and the machined (tensile, impact, and hardness) samples in triplicates were buried in the mixture. The packed samples alongside the carburizer were then sealed at one end using moist clay slurry to make the crucible air-tight and eliminate the possibility of air ingress during the pack carburization process as shown in Fig. 3a, b. The crucible cover was locked and placed inside an electric furnace set to 950 °C operating at a 5 °C/min heating rate for 3 h soaking times. Afterward, the carburized samples were quenched in 100% water at room temperature and tempered in the furnace at 300 °C for 1 h before being air-cooled to room temperature.

A similar procedure was adopted for other samples quenched in 100% SAE, 100% groundnut, and other quenchant blends as presented in Table 2.

Assessment of mechanical property

The assessment of the mechanical property of the carburized mild-steel samples quenched in different media as given in Table 2 was based on tensile strength, impact energy, and hardness tests. The tensile tests were conducted using a Testometric Universal Testing Machines-UTM (Model: M500-100AT). Each of the tensile tests conducted was performed in triplicates. The load was gradually applied at a strain rate of 10^{-3} mm/min until the specimen yielded and the corresponding yield strength and strain were noted and recorded accordingly.

The impact energy test was carried out on the Hounsfield balance impact machine Model–H10-3 at the University of Ife, Osun State Nigeria to determine the impact resistance of the quenched mild steel. The sample dimensions of 10 × 10 mm square by 75-mm-long standard specimens according to ASTM E23 were used and the tests were conducted in triplicates and recorded.

Vickers pyramid method was used to evaluate the hardness of the quenched carburized and control samples. The prepared samples were mounted on the anvil and brought in contact with the pyramid indenter on a Leeb Hardness Tester (Model: SKD-552) for 15 s dwelling time. The hardness impressions were taken transversely in two perpendicular directions along the cross-section of the specimen surface.

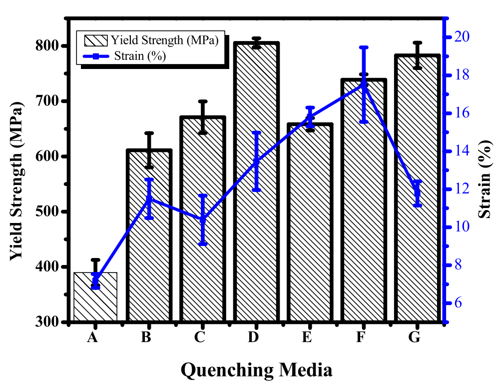
Microstructure examination of carburized quenched mild-steel

Samples for microstructural examinations were prepared by mechanical grinding in a series of sizes of emery papers in the order of 400, 600, 800, 1000, 1200, and 2000 μm grit sizes under constantly running water to avoid phase changes. The silvery shining samples were etched in 2% Nital (2% nitric acid and 98% ethyl alcohol) for 5 s and rinsed in water after which it was kept in a desiccator before the metallurgical optical examination using Accuscope optical microscope with camera (serial No.: 05240011).

Results and discussion

Tensile strength of the carburized mild-steel quenched in bio-oil

Figure 4 is the graph of tensile yield strength and strain for both the control specimen and carburized mild steel, quenched in water and different blends of bio-mineral oils. It is evident from Fig. 4 that, the quenching media has a significant influence on the tensile yield strength and strain of the carburized mild steel. It is worth noting that, the tensile yield strength and strain of the control mild-steel sample were 389.65 MPa and 7.17% respectively. This strength increases upon carburization and quenched in 100% water, SAE40 mineral oil, and ground nut oil to 611.26, 670. 97, and 805.43 MPa respectively. The corresponding strain of 7.17% for the control mild-steel sample was observed, while 11.50, 10.38, and 13.46% strain were noticed in carburized steel quenched in 100% water,



Sample	Composition of the Quenchants
A	Control Sample
B	100% Water
C	100% SAE 40 oil
D	100% Groundnut oil
E	70% Groundnut oil +30% SAE40 oil
F	60% Groundnut oil + 40% SAE40 oil
G	50% Groundnut oil +50% SAE40 oil

Fig. 4 Tensile strength and strain behavior of quenched carburized mild-steel

SAE40, and ground nut oil respectively. This increment in tensile strength is equivalent to 56.87%, 72.2%, and 106.7% with a steady enhancement of 60, 44.7, and 87.66% in tensile strain when quenched in 100% water, SAE40, and ground nut oils respectively. A similar improvement in tensile strength was noticed with a blend of ground nut and SAE 40 oils in different proportions as depicted in Table 2, though with a slight difference in the tensile strength and strain properties depending on the blend proportions. However, the best combination of tensile properties (738.66 MPa, 17.51% strain) was obtained at a bio-mineral oil blend ratio of 60% groundnut oil and 40% SAE40. In this composition, a 90% tensile strength and 120% tensile strain increment were achieved. Meanwhile, the tensile strength of samples quenched in 100% groundnut, and 50:50 SAE and ground nut oil blends are higher but possess less strain characteristics, an indicative of low plastic deformation. This implies that a high strength with better strain enhancement can be obtained using a bio-mineral oil quenchant with the blended ratio of 60:40% groundnut and SAE 40 oil respectively. The difference in the enhancement of both the tensile strength and strain of the heat-treated mild samples compared with the untreated mild steel sample as observed in this work could be attributed to the dual effects of carburizing and quenching media as used in this study. The infiltration of carbon atoms at the austenitic phase into the interstices of the grain lattice of the mild steel by diffusion and the fast cooling rate in a controlled manner of the bio-mineral quenching media acts as grain structure refiner as discernible in Fig. 7, enhances the tensile properties of the quenched carburized mild steel (Fig. 4). This result is in agreement with the report of Ndaliman [23] and Adekunle et al. [24] where they asserted that the tensile strength of the quenched specimen can be influenced by types of quenching media and heat treatment operations. Furthermore, the highest tensile strength recorded when 100% of ground nut oil was used as quenching media as compared to other formulated quenching media in this work can be arrogated to its fast cooling rate and less resistance to the motion of vapor bubbles during the nucleation boiling stage of the cooling process. The performance of groundnut oil was further affirmed by Odusote et al. [25], who reported that groundnut oils with lower viscosity characteristics and higher heat transfer coefficient will remove heat faster during quenching operation, which aids the grain refinement and thus improves the tensile strength of the samples as experimentally observed in the present study. In addition, the increasing trend of tensile strength and strain of carburized mild steel when SAE40 and Groundnut oils were blended can be attributed to the hybrid effect of the enhancing potentials of tensile strength of both the mineral and bio-oils mixtures.

Impact strength of heat-treated and untreated carburized mild-steel

The impact strength characteristics of the control and quenched carburized mild steel samples in different media were shown in Fig. 5. It was observed that the impact strength of mild steel can be influenced by the types and composition of the quenching media. The impact strength of the control mild steel sample was highest and this value slightly decreases in a fluctuating manner in different quenching media as examined in this study. The high impact strength as obtained in the control sample in this study can be arrogated to low carbon content (0.159wt%) of the mild steel as discernible in Table 1. The decrease in impact strength of the carburized specimen quenched

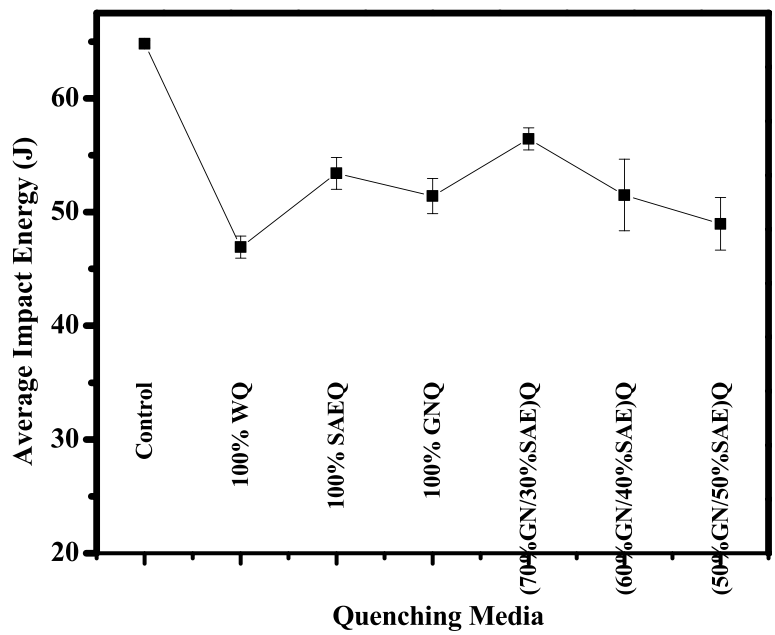


Fig.5 Impact strength of heat-treated and untreated carburized mild-steel

in different bio-mineral oils blend can be attributed to possible carbon infusion via the carburization process and the difference in physio-chemical characteristics of different quenching media with varying heat transfer coefficients as reported by Odusote et al. [25]. In all, observations from the present study as represented in Fig. 5 revealed that the least impact strength was obtained in specimens quenched in water relative to the specimen cooled in bio-mineral oil media. This could be due to higher heat transfer coefficient of water compared to other bio-mineral oil quenchants resulting in rapid cooling rate with retained martensitic phase with less absorbed impact energy as confirmed by Fig. 5. For the bio-oil quenched samples, the peak impact strength was obtained at 70% ground nut oil and 30% SAE40 quenchant followed by 60% ground nut oil and 40% SAE oil blends. The decrease in impact strength of quenched carburized mild steel was a result of the strain hardening effect of the various bio-oil quenching media, due to the fast cooling rate of the carburized sample with increased carbon compared with the control samples with lower carbon content. Hence, it resulted to loss of ductility and in turns lower absorption of energy when subjected to impact loading. Notwithstanding, the slight increase in impact energy as observed in 100% SAE and 70/30 groundnut/SAE oil blends samples could be attributed to the high viscosity and flash point of the blend composition ratio. These visco-physical characteristics of the mineral and bio-mineral oil blends can enhance thermal stability and create a little time delay in the quenching process before the reach of the flash point of the bio-oil compositions which gives room for grain coarsening and a slight increase in ductility for the slight increase in energy absorption as displayed in Fig. 5. Hence, the resulting marginal rise in impact strength. This result was similar to that reported by Dodo et al. [20] where the sample quenched in SAE40 was reported to have the highest impact strength due to the enhanced thermal stability of transesterification.

The hardness value of heat-treated and untreated carburized mild-steel

Figure 6 shows the micro-hardness distribution as measured using the Vickers micro-hardness machine for both the control and quenched carburized mild steel at different positions measured from the outer surface of the specimen. The hardness readings were taken at positions 1 mm, 3 mm, and 5 mm from the outer edge surfaces for all samples. Findings revealed that a higher hardness value at the position closer to the edges of the samples than those positions closer to the core for all samples relative to the control sample was obtained. For the control sample, the hardness is lowest at all the examined positions compared to the carburized quenched samples. The low in hardness value can be attributed to the low carbon content of the control sample. This hardness distribution for both the carburized and control samples was however regardless of the quenched media indicating a case hardening phenomenon. It will be noted that carburized sample quenched in 100% groundnut oil has peak hardness values of 173.8, 141, and 103.6 HV at positions 1 mm, 3 mm, and 5 mm respectively from the edge of the sample surfaces. Similar results were observed with 50/50% groundnut and SAE oils blend compared to others. This slight increment in both samples compared to other can be best explained with faster heat abstraction and retained martensitic phase of the quenched samples. These values indicated a hardness increase of 40.16, 21.92, and 5.71% relative to the control sample respectively at positions 1 mm, 3 mm, and 5 mm. similar increments were also noticed with other formulated quenchants though at lesser magnitude compared with that quenched in groundnut oil. This implied that bio-formulated quenchant significantly influenced the case-hardening of all samples in comparison with the control specimen. This result corroborated the observation of Sekunowo and Nwagu [26] where the increase in hardness was attributed to the fast cooling rate of the quenchant and inclusion of carbon atom via diffusion from the carburization phenomena at austenitic

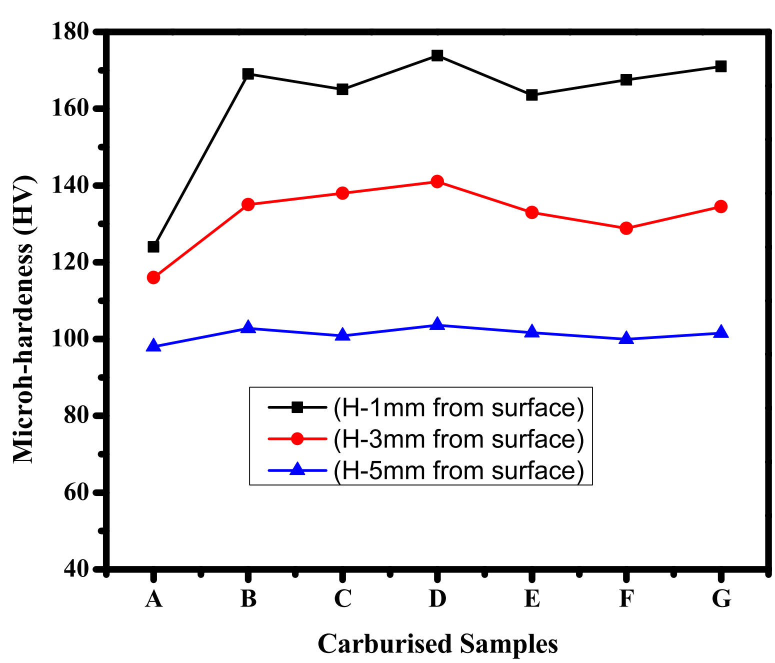


Fig. 6 Hardness value of heat-treated and untreated carburized mild-steel

temperature accelerated by the catalytic potential of the formulated quenchants. Other earlier work in tune with the present findings was that reported by Oyetunji and Adeosun [27] where the increase in hardness of carburized steel was said to be influenced by the types of carburizing media, austenitic temperature, holding time, and quenching media.

Optical microstructure of heat-treated and untreated mild-steel

Figure 7 shows the optical microstructural analysis of the control specimen, and those carburized specimens and quenched in water, SAE 40 oil, groundnut oil, and blends of 70/30, 60/40, and 50/50% groundnut oil, and SAE40 oils respectively. The microstructure of the control specimen (Fig. 7a) showed a combination of ferrite (gray) and pearlite (black) coarsely, and it can be observed that the untreated specimen possessed a higher amount of ferrite structures, as is common to mild steel. In addition, the microstructure of the specimens cooled in the bio-mineral media (Fig. 7b–g) illustrated a transformational phase change in terms of size and shape from the original austenite grains to a substantial extent as discernible in Fig. 7b–g. In the water-cooled specimen, the pearlite dispersed in a finer ferrite phase with a precipitated carbide phase (white) due to the rapid cooling rate of the specimen from the austenitic phase to room temperature. The specimen was compensated with increased brittleness and reduced ductility. Furthermore, samples quenched in SAE40 oil and groundnut oil show substantial martensite structure with well uniform dispersed pearlitic phase within the microstructure;

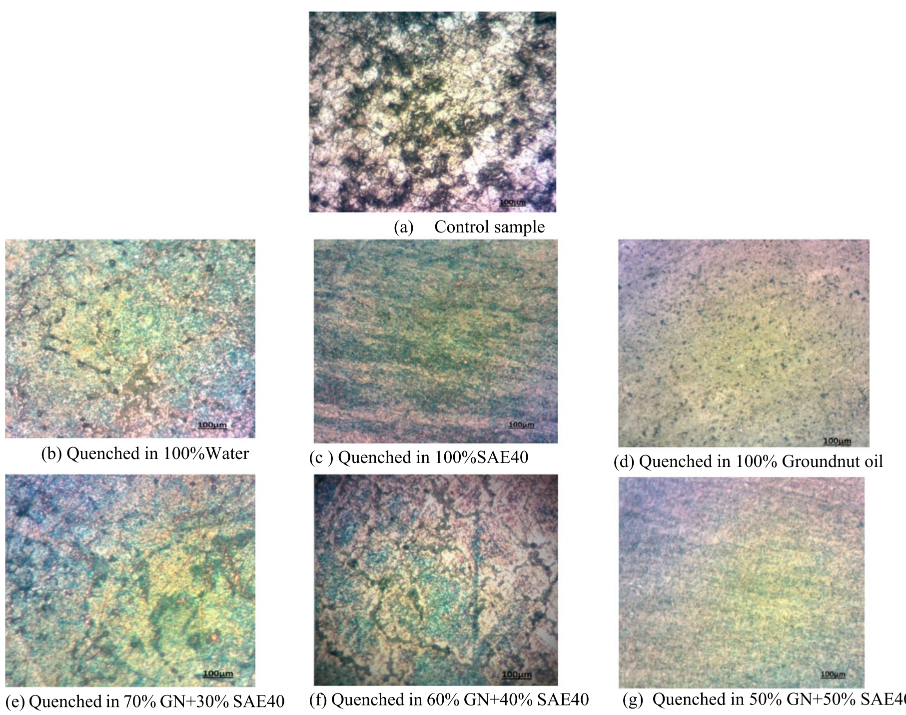


Fig.7 Optical microstructural examination of carburized mild steel quenched in different media. **a** Control sample. **b** Quenched in 100%Water. **c** Quenched in 100%SAE40. **d** Quenched in 100% Groundnut oil. **e** Quenched in 70% GN + 30% SAE40. **f** Quenched in 60% GN + 40% SAE40. **g** Quenched in 50% GN + 50% SAE40

however, a finer grains structure relative to the SAE40 cooled specimen was noticed in the groundnut oil quenched specimen. This characteristic feature of the microstructure can be linked to the fast cooling rate of the bio-mineral oil (though higher in groundnut oil than SAE40) from the austenitic temperature to room temperature which gave a mixture of hard phases/structures. Consequently, this can lead to an increase in tensile strength, hardness, and reduction in ductility of the material as observed in this study and in line with earlier reported studies [28, 29]. Meanwhile, the microstructures of specimens quenched in groundnut oil/SAE40 oils blended at 70/30%, (Fig. 7e), 60/40% (Fig. 7f), and 50/50% (Fig. 7g) respectively show uniform martensite with a well-dispersed pearlites phase in a relatively larger grain structure. It will be noted that, as the concentration of SAE40 oil increases in the quenchant formulation, the more the dispersion and grain refinement in the pearlitic phase in the microstructure as obtained in this study (Fig. 7f, g). This could be responsible for the higher tensile strength recorded in the specimen quenched in 50/50 groundnut/SAE40 relative to the two other blend formulations. The above observations were in good agreement with the literature [30] where phase dispersion and grain refinement are more at the surface edges than those closer to the core characterized by coarse grain structure.

Conclusions

In this project work, an attempt was made to study the mechanical and microstructural properties of eggshell/date seed carburized mild steel, quenched in different formulated bio-mineral oil blends of groundnut oil and or SAE 40 oil. From the results of the investigation, the following conclusions were drawn:

1. The microstructure, impact, hardness, tensile yield strength, and strain of carburized mild steel quenched in water and bio-mineral oil blends at different proportions were significantly influenced in varying degrees by the quenching media.
2. The maximum yield tensile strength (805.43 MPa) and hardness at the edge (173.8 HV) equivalent to 106.7 and 87.66% increment with a marginal decrease in impact strength was obtained in a specimen quenched in 100% groundnut oil.
3. The use of SAE40 oil, groundnut oil, and or its blends in varying proportions resulted in grain refinement of the microstructure at varying degrees with the most refinement noticed in samples quenched in 100% groundnut oil which are responsible for the enhancement of mechanical properties.
4. The 60/40% groundnut and SAE40 oil blends however yielded the best combination of mechanical properties enhancement (tensile strength 738.66 MPa, strain 17.12%, and impact strength 51.1 J) when considered altogether. This translates to 90% tensile strength and 120% tensile strain increment in comparison with the control samples. The 60/40 groundnut/SAE40 oil blend is therefore recommended for metallurgical heat treatment of mild steel for critical industrial applications.

Abbreviations

UTM	Universal Testing Machine
WQ	Water quenched

GNQ	Groundnut oil quenched
A	Control sample
B	100% Water quenched sample
C	100% SAE40 oil quenched sample
D	100% Groundnut oil quenched sample
E	70%Groundnut oil + 30%SAE40 quenched sample
F	60%Groundnut oil + 40%SAE40 quenched sample
G	50%Groundnut oil + 50%SAE40 quenched sample

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Authors' contributions
The research reported in this paper was conceptualized, and the experimental procedures were monitored by KMY and BSA. The performance of the experiment and acquisition of results was carried out by ASA and OIJ. The analysis and interpretation of the data and preparation of the manuscript were done by KMY and OKO. The authors read and approved the final manuscript.

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Availability of data and materials
The datasets are available from the corresponding author upon reasonable request.

Declarations

Competing interests
The authors declare that they have no competing interests.

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