

Effect of Nigerian Neem Seed Oil as Austempering Quenchant for Locally Recycled Mild Steel

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Abstract: The efficacy of locally extracted neem seed oil as a quenchant for austempered Nigerian mild steel so as to strengthen it for automotive parts production was investigated. Experimental samples were cut out of locally produced low carbon steel, prepared, raised in electric furnace to a temperature of 900°C. They were soaked for an hour and thereafter austempered by quenching them in a neem oil bath maintained at 300°C. Austempered specimens were withdrawn from oil bath at varied holding periods of 30, 60, 90 and 120 minutes. The treated specimens and an untreated sample were prepared for metallographic examination and mechanical property analyses including percentage reduction in area and elongation; hardness; tensile, impact and yield strength. Result showed that the austempering heat treatment caused changes to the microstructure of specimens with bainite being produced at shorter retention periods (30 and 60 minutes) of specimen in heated neem oil bath. Based on the microstructural changes, mechanical properties were also varied by; +5.97% to -0.53% in ultimate tensile strength; +8.44% to -0.94% in impact strength; +3.68% to -1.6% in hardness and +10.6% to -47.73% in yield strength for specimens retained for 30, 60, 90 and 120 minutes respectively. It was observed that longer austempering in neem oil quenchant increased ductility of steel samples. In comparison with ASTM 897 mechanical property limits for production of automotive shafts and gears, the result showed that though some property enhancement was achieved by austempering Nigerian steel in local neem oil quenchant, none (except based on impact strength) was suitable for production of automotive gears and shafts of high standard.

Key words: Austempering • Neem oil • Photomicrograph • Mechanical properties

INTRODUCTION

Austempering is an isothermal heat treatment that is applied to ferrous alloys, most notably steel and ductile iron primarily to improve mechanical properties. It produces lower bainite microstructure in steels and a structure of acicular ferrite and high carbon stabilized austenite known as ausferrite in cast iron. Austempered steel are used to produce high tensile, impact, fatigue strength and corrosion resistant parts for mechanical systems like ship, aircrafts, automobiles, power plant e. t. c. Property enhancement of steel by austempering is effected by such important treatment factors as quenching media, size of work piece, type/nature of steel used, cooling and holding rate and austempering temperature (Applied Process, 2005) [1]. Common austempered automobile component include; shock

absorber, transmission case, clutch housing, engine front cover, oil pan, steering column upper brackets, brake pedal brackets steering wheel cores, cylinder head cover, differential pinion, disc wheel, parking pawl, shaft, gear e.t.c. that require high tensile and impact strength Grossman and Bain (en.wikipedia.org/wiki/austempering, 2011) [2]. Their team started austempering in 1930s when working for United States steel laboratory as they evaluated the metallurgical responses of steels cooled rapidly from 788°C to intermittently high temperature and held at various time. Outcome of this pioneering work led to isothermal transformation of steel to austempered grey iron.

By 1941 Inco and Climax molybdenum collaborated on an experiment with cast iron that produced tempered bainitic microstructure with a tensile strength of 620N/m². During World War II, it was extensively used to produce

gun parts (www.applied process.com/process, 2005) [1]. It was found that the process resulted in low distortion and parts tougher than the quenched and tempered components which they replaced. However, the best equipment available for austempering was very inefficient. Then, austempering process was relatively expensive; but by 1950s austempering process was routinely applied to steel and malleable iron parts. The relative high cost of process limited its use to the highest performance parts (www.applied process.com/process, 2005) [1]. According to Rajan *et al* (1988) [3], austempering is specialized heat treatment process in which austenite is transformed into bainite. This transformation happened during conventional heat treatment process involving continuous cooling. Cooling rate for first quenching bath are the two parameters that control the process and steels for which austenite to pearlite transformation was comparatively slow but suitable for the process. Bhadeshia (2001) [4] described austempering as isothermal transformation of ferrous alloy at a temperature below that of pearlite formation and above martensite formation. Steel are austempered when heated within a range of 790°C - 900°C, quenching in a bath at constant temperature in range of 260°C - 400°C. They also specified characteristic of salt used for austempering, quenching media e. t. c. It was observed (Applied Process, 2005) [1] that application of austempering to steel provided users with a tough, high-strength component that resist embrittlement. Thus, it was recommended as appropriate for medium to high carbon stamping, forging, casting and fill density powdered metal part.

Batra (2003) [5] worked on some factors affecting austempering heat treatment of materials. He noted that amongst the factors, quenching media, quenching temperature and type of steel are the most important factors that must be seriously looked into during austempering process. Ashly *et al* (1992) [6] described it as method of hardening steel by quenching it from austenizing temperature to heat-extracting medium. He suggested salt as a suitable heat-extraction medium. During the process austenitic steel is cooled at a sufficient rate to avoid the nose of the s-curve and hold just above the start of martensite transformation (M_s point) for complete transformation to bainitic structure at constant temperature thereby, alleviating thermal and transformation stress that could cause cracking or distortion. Oil has high boiling point and transformation from start of martensite formation to finish is slow and reduces likelihood of cracks. However oil quenching results in fumes, spills and at times fire hazard.

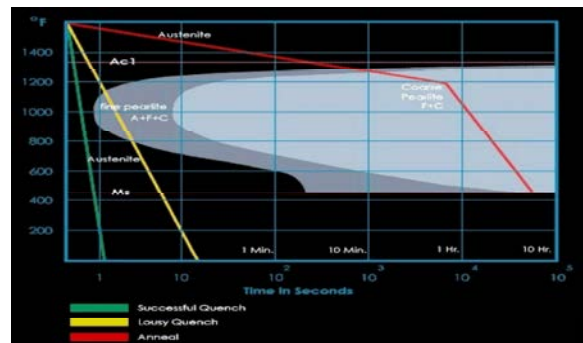


Fig. 1: Schematic I-T Diagram illustrating Austempering, Quench and Tempering Processes.

Oil cooling is much slower than water-cooling. Rate of cooling is greatest at about 600°C and is relatively slow in range of martensite formation. A reliable and tested way of increasing the cooling capacity of oil is by vigorously agitating bath or charge (Thelning, 1975) [7]. Austempering involves less heat treatment time and is preferred when fast turnaround is a prime consideration (Ajax Electric, 2002) [8]. Figure 1 below shows isothermal (I-T) diagram for steel with both austempering (green line); quench and tempering (red line) process outlined.

Austempering have been applied to thin section of certain medium/high carbon steel or alloy containing steels of thicker section. It requires high temperature quench and hold, usually in molten salt and result in low distortion with tough structure that require no tempering (Nick 1996) [9]. Austempered iron and steel offer design engineers alternatives to conventional material/process combinations. Depending on the material and application, austempering may provide benefits of ease of manufacturing, increased bending and contact fatigue strength, better wear resistance or enhanced dampening characteristics that result in lower noise in gear and shaft. Low and high carbon steel are successfully austempered along with powdered metal mix with sufficient hardenability and near full density. Bainitic microstructure produced by austempered steel is more wear resistant than tempered martensite. Also, bainitic steel is more resistant to hydrogen embrittlement and stress corrosion cracking (Lambers *et al.* 2009) [10].

The automotive industry in Nigeria is poorly developed because of non availability of locally produced high strength component parts of high standard. The situation is so precarious that even when some of the original parts of the imported vehicles that are common features on Nigerian roads fail, the owners have to resort

Table 1: ASTM 897 Property table for steel used for production of gears and shafts

Grade	Tensile Strength (N/mm ²)	Yield strength (N/mm ²)	Elongation (%)	Impact energy (Joules)	Hardness (No.)
1	850	550	10	100	269-321
2	1050	700	7	80	302-363
3	1200	850	4	60	341-444
4	1400	1100	1	35	366-477
5	1600	1300	N/A	N/A	444-555

to imported used replacement components whose reliability are unknown. Incapability of local industries have not enabled them to key in to produce and supply required high quality completely knocked down parts required by the local automotive assembly plants available in the country making actualization of the local content implementation initiative a rouse. Apart from compositional quality of automotive component production steel, the heat treatment to which steel was subjected to could correct and greatly enhance its service properties and widen its range of application.

These technologies are not readily available in Nigeria. This research is therefore aimed at investigating the efficacy of locally available austempering quenchant like neem oil as liquid bath for strengthening locally produced steel for the production of high quality automotive parts. The main objectives are to adopt known locally produced steel, subject it to austempering treatment using neem oil maintained at elevated temperature as quenchant, test specimens from the steel for mechanical property enhancement and microstructural changes and to compare the result with established standard as shown in Table 1 (Brandenberg *et al*, 2001) [11] to ascertain level suitability of such treated steel. The significance of this work is that it will add to technological effort of actualizing 100% Nigerian made automobile.

MATERIALS AND METHODS

Materials: Materials used for the research work included plain carbon steel samples obtained from steel dealers in Jos, Nigeria; quantity of well milled and filtered neem oil, metallographic etching reagents and assorted grinding and polishing papers. Experimental equipment included metal cutter, lathe machine, electric arc furnace, grinding machine, polishing machine, metallurgical microscope, izod impact test machine; Hounsfield tensile test machine, digital hardness test machine and metal analyser. Neem seeds were obtained from a fruit market along new Kano road junction, Dogarawa Zaria. Oil was milled from seed using laboratory facilities at National Research Institute for Chemical Technology, Zaria, Nigeria. The most commonly used chemical etchant for

iron-carbon steels, alloy steels and cast iron; that is nital was used for this work. It was composed of 100ml ethanol (C₂H₅OH) and 7ml nitric acid (HNO₃). Research equipment was accessed at various laboratories of Department of Metallurgical and Materials Engineering, Ahmadu Bello University and National Research Institute for Chemical Technology, both in Zaria; and Scientific Equipment Development Institute, Minna, Nigeria.

Methods: The research experiments were grouped and conducted at different stages.

Steel Specimen Analysis: The cylindrical 4.6mm diameter steel sample was cut out and prepared for compositional analysis with metal analyser at Scientific Equipment Development Institute (SEDI), Minna. The result of the chemical compositions obtained is as presented in Table 2.

Neem Oil Extraction and Analysis: About 2.0 Kg of neem seeds were purchased and sun dried such that the coat could easily be separated from the seeds. Wincrowing was done to separate and obtain clean seeds. Clean seed was then poured in a mortar and a pestle used to pound and crush seed to paste-like form. The mash was transferred to a stainless steel bowl and 0.2 litres boiling water was added into the paste and thoroughly stirred to free its oil content. It was placed in oven (Volcan laboratory furnace) and temperature was set at between 120°C and 140° C. Heating was carried out for about 10 minutes. This was aimed at enhancing oil flow during pressing. The oven heated paste was then transferred to a filter cloth of very fine sieve size. Edges of the sieving cloth were gathered and tied to prevent leakage.

Sieving cloth with the neem paste was then placed in a Bideberg ram press to squeeze out oil. Pressure was continuously applied to paste at intervals and oil was squeezed out of it until no more oil oozed out of neem seed paste. A laboratory flask was use for collection of the oil. Neem oil was taken to National Research Institute for Chemical Technology Zaria, Nigeria for proximate chemical and physiochemical analysis. Result of physicochemical analysis is as presented in

Table 2: Chemical composition of the experimental steel sample

Element	C	Si	Mn	P	S	Cr	Ni	Mo	Sn	Cu	V	Fe
Composition (%)	0.16	0.15	0.47	0.043	0.006	0.01	0.01	0.01	0.001	0.03	0.002	Balance

Table 3: Results of physicochemical analysis of Nigerian neem oil used

Properties Analyzed	Values
Specific Gravity	0.9304 (15/40°C)
Flash point	157 (°C)
Pour Point	+8 (°C)
Kinematic Viscosity	8.08 ((100°))
P H	5.6

Table 4: Results of Chemical analysis of Nigerian neem oil used

Fatty Acids Present	% weight
Oleic acid	41
Linoleic acid	20
Stearic acid	20
Palmitic	18
Sulphur	0.0293

Table 3 and that of chemical analysis is as presented in Table 4. Pour point of oil was tested according to ASTM D 97. Relative density was tested by hydrometer method (ASTM D 1298). Flash-point was determined with Pensky-martens closed cup tester (ASTM D93). Kinematic viscosity was tested in accordance with ASTM D 445 (Heat treater's Guide, 1995) [14] and sulphur content was tested with Horiba apparatus model SLFA2800 combined with X-ray machine. PH was tested using meter of serial No 7010/1886.

Test Specimen Preparation: The experimental steel specimen was cut into suitable pieces of 40mm each with a power cutting machine. The samples were surface cleaned with the grinding and polishing machines to remove surface oxide scales and grouped according to the schedule of tests prior to austempering treatment. In accordance with Vijendra Singh (2012) [12] and Kiliçli V, Erdogan M (2008) [13], samples classified as group A; cut from high carbon steel were heated to a temperature of 800°C and held for 1 hour for soaking and proper homogenization in electric arc furnace. Samples classified as group B; cut out from the low carbon steel were raised in the furnace to a temperature of 900°C and also held for 1 hour for soaking and proper homogenization. Samples were quenched in neem oil baths maintained at temperature of 300°C and held at different periods of 30 minutes, 1 hour, 1½ and 2 hours (Heat Treater's Guide, 1995) [14]. Thereafter test pieces were withdrawn from hot oil bath and air cooled to room temperature. The samples were separated and prepared for metallographic examination and mechanical property tests that included hardness, yield, impact and tensile strength.

Specimen Testing: The austempered specimens meant for metallographic tests were selected, grouped and on Bakelite power. Grinding and polishing were done manually on water lubricated grinding machine with silicon carbide abrasive paper grades 120, 180, 240, 320, 400 and 600 grit sizes. Final polishing was carried out using 0.3 microns particle size alumina to obtain mirror surface finish. Etching was done using 2% Nital for 10-20 seconds by handling with a pair of nickel crucible tongs. After etching specimens were washed with running water and dried by immersion into boiling ethanol. They are drawn from the ethanol after a few minutes and shaken to remove the surplus while it dried almost instantaneously (Thelning K. E.M, 1975) [7]. Specimens were then examined under a metallurgical microscope at a magnification of X100 and the microstructures were photographically recorded. A sample of the untreated steel was also included in the metallographic specimens for control.

A universal digital hardness machine LR300TD was used to determine hardness of specimen. A group of Bakelite mounted and polished samples was selected and subjected to hardness examination. Having known the materials to be tested, selection of ball diameter, test duration, thickness of test material and load, parameters were selected based on standard test guide (Reed-Hill R, Abbaschian R. 1991) [15].

Results were tabulated as obtained and Brinell hardness was calculated using the formula below; where D = Diameter of ball (mm); P = Load on ball (Kgf); d = Diameter of impression (mm).

$$HB = \frac{2P}{\pi D \left[D - \sqrt{D^2 - d^2} \right]}$$

Hardness number was determined and indicated as HB (dimensionless No). The tolerances of the test duration were ±2 seconds for 10 seconds period; ±4 seconds for 30 seconds period and ±6 seconds for 30 seconds test period. Environmental temperature for the control process was 30°C±5°C. The impact toughness (Impact strength) of the materials under investigation was determined with Izod test. The test employed a bench clamp type technique for holding specimens and made use of a pendulum-testing machine. A specimen was broken by a single overload event due to the pendulum impact. A stop pointer recorded how far the pendulum

swung back up after fracturing the specimen. Impact toughness of a sample was determined by measuring the energy absorbed in its fracture. This was obtained by noting the height at which the pendulum was released and the height to which the pendulum swung after it has struck the specimen. The height of the pendulum times the weight of the pendulum produced the potential energy and the difference in potential energy of the pendulum at the start and the end of the test equalled the absorbed energy which was instantaneously recorded [16].

For the tensile strength determination, the Harrison M40 (model 16K20); a precision tool room lathe was employed for production of standard test specimen. The samples were grouped and austempered with other property test specimens. The austempered pieces were thoroughly cleaned to remove all possible scales and then subjected to tensile strength tests using the universal strength testing machine. Each specimen was properly mounted and gripped in the machine this experiment and tensile load was gradually and continuously applied to it until necking and fracture occurred. The load at which failure occurred was recorded and ultimate tensile strength (UTS) was computed and tabulated. Yield strength, tensile strength, percentage elongation and percentage reduction were determined from test result [17].

RESULTS

Figure 2 presents the photomicrograph, while Table 5 presents the mechanical properties of steel sample as received before austempering for experimental control. Figure 3, 4, 5 and 6 show photomicrographs (Magnification X100) of steel specimens raised to 900°C and austempered in neem oil at 300°C for 30, 60, 90 and 120 minutes holding periods respectively. Figure 7 presents the ultimate tensile strength (N/mm²) of steel heated to 900°C and austempered 300°C in neem oil against different quench holding periods. Figure 8 present the impact strength (Joules) of steel heated to 900°C and austempered at 300°C in neem oil against quench holding period in minutes. Figure 9 shows the reduction in area (%) of steel heated to 900°C and austempered at 300°C in neem oil against quench holding time. Figure shows the Brinell hardness of steel heated to 900°C and austempered at 300°C at 300°C in neem oil against quench holding time. Figure 11 presents elongation (%) of sample heated to 900°C and austempered at 300°C in neem oil against varying quench holding periods and Figure 12 shows

Table 5: Mechanical properties of the experimental steel sample before austempering.

Brinell Hardness (No)	Tensile strength (N/mm ²)	Impact strength (Joules)	Yield ((N/mm ²)
166.85	528.90	62.73	415.14

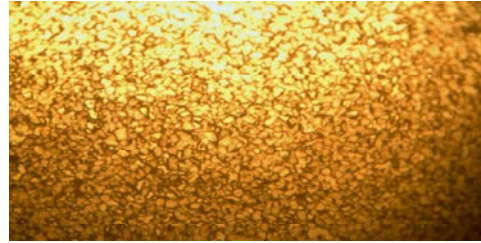


Fig. 2: Photomicrograph (magnification X100) of the steel sample before austempering.

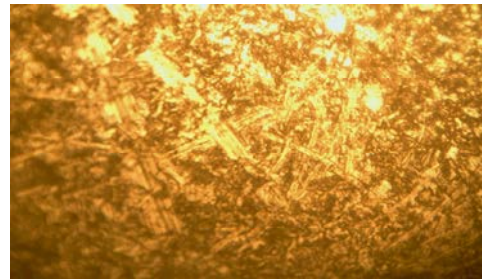


Fig. 3: Photomicrograph (Magnification X100) of the steel specimen raised to 900°C and austempered at 300°C in neem oil for 30 minutes holding time.

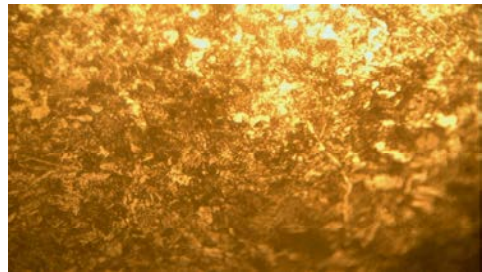


Fig. 4: Photomicrograph (Magnification X100) of steel specimen raised to 900°C and austempered at 300°C in neem oil for 60 minutes holding time.

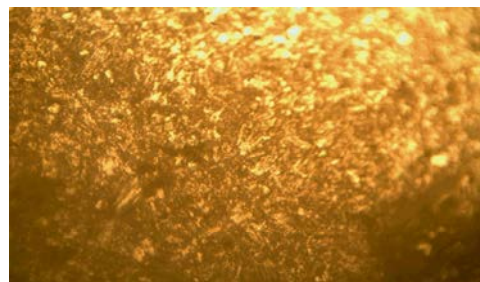


Fig. 5: Photomicrograph (Magnification X100) of the steel specimen raised to 900°C and austempered at 300°C in neem oil for 90 minutes holding time.



Fig. 6: Photomicrograph (Magnification 100) of steel specimen raised to 900°C and austempered at 300°C in neem oil for 120minutes holding time.

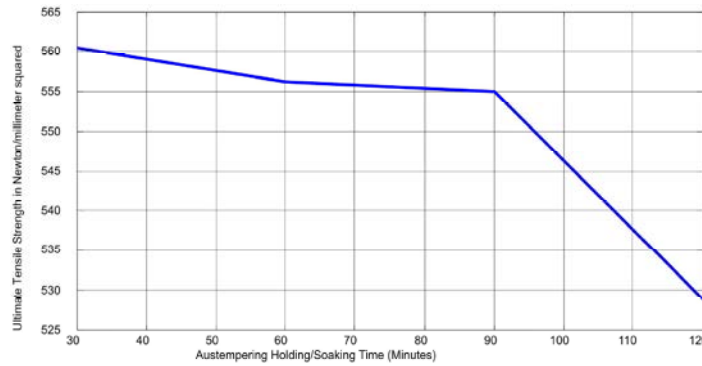


Fig. 7: Ultimate Tensile Strength of Steel heated to 900°C and Austempered 300°C in Neem oil (N/mm²) against Austempering Quench holding period in Minutes.

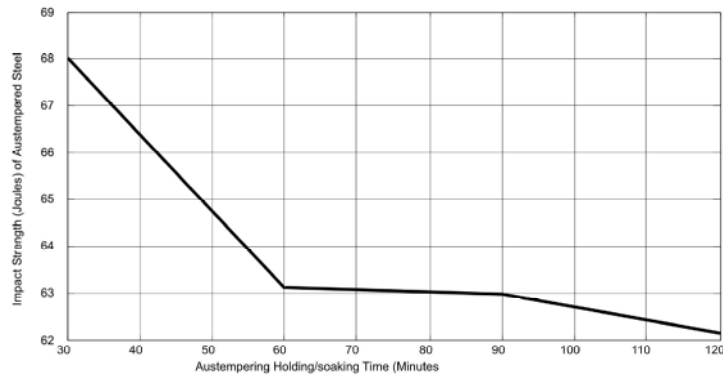


Fig. 8: Impact Strength of Steel heated to 900°C and Austempered 300°C in Neem oil (Joules) against Austempering Quench holding period in Minutes.

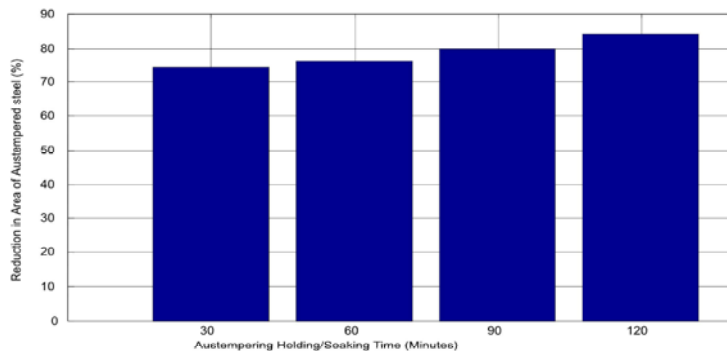


Fig. 9: Reduction in Area of Steel heated to 900°C and Austempered 300°C in Neem oil (%) against Austempering Quench holding period in Minutes.

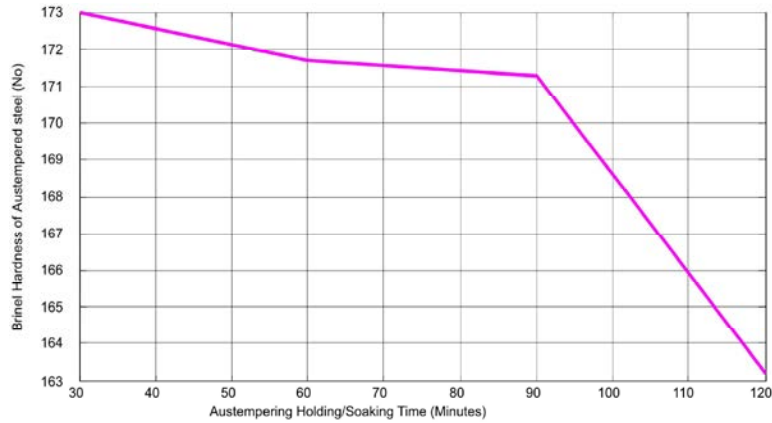


Fig. 10: Brinell Hardness of Steel heated to 900°C and Austempered 300°C at 300°C in Neem oil (No) against Austempering Quench holding period in Minutes.

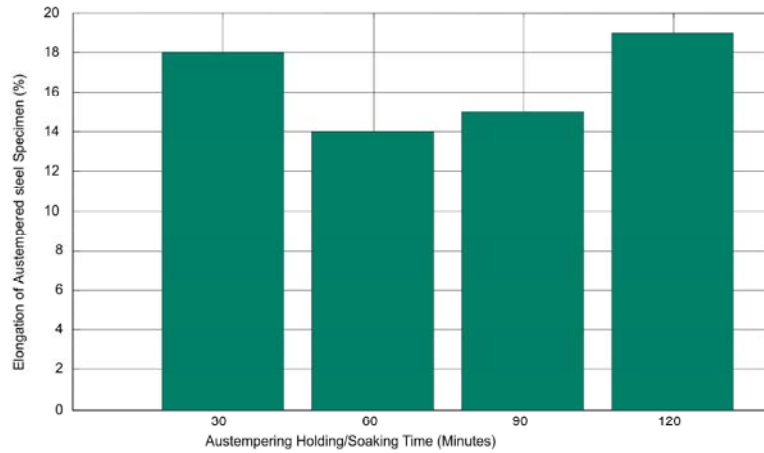


Fig. 11: Elongation of Steel Sample heated to 900°C and Austempered 300°C in Neem oil (%) against Austempering Quench holding period in Minutes.

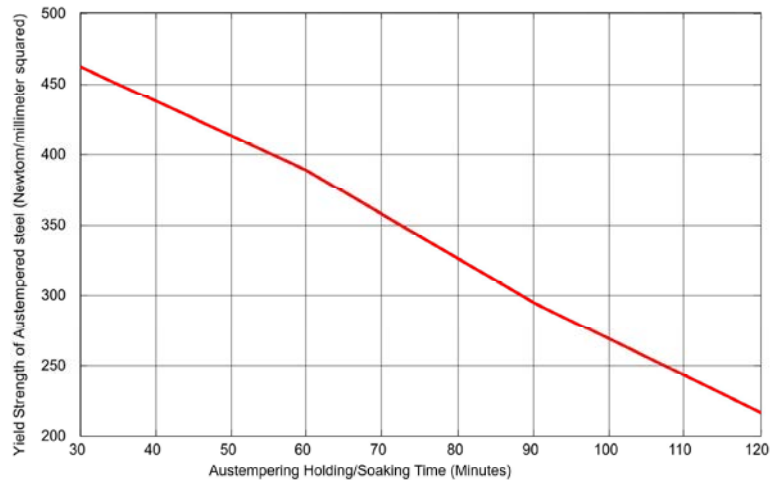


Fig. 12: Yield Strength of Steel Sample heated to 900°C and Austempered 300°C in Neem oil (N/mm²) against Austempering Quench holding period in Minutes.

yield Strength of steel samples heated to 900°C and austempered at 300°C in neem oil against varying quench holding time in minutes [18].

RESULTS AND DISCUSSION

The photomicrograph in Figure 2 shows a pearlitic structure dispersed within a ferrite matrix. This is the as-produced structure of the steel sample. The mechanical properties results are as presented in Table 5. Figures 3 to 6 present photographs of the microstructures of the steel specimens raised to a temperature of 900°C quenched into neem oil austempering bath maintained at a temperature of 300°C and held for different time period of 30, 60, 90 and 120 minutes respectively. Structures obtained at different holding time using neem oil as quenchant showed presence of pearlite distributed in ferrite with some amounts of partially transformed austenite in forms of bainite in figures 3 and 4 with figure 3 having more of bainitic structure. Figures 5 and 6 that showed microstructures of specimen austempered for 90 minutes and 120 minutes showed fine ferrite, pearlite and some untransformed austenite as conspicuous phases. The austempering treatment was like combined heat treatment that involved both hardening and tempering during which rapid cooling of austenized specimen was done in quench medium maintained at elevated temperature for short a period. The joint treatment could produce variety of microstructure phases that could have included untransformed austenite, bainite, troostite and martensite depending on the cooling rate of quenchant, tempering holding time and quenchant temperature.

The disparity in microstructure shown in figure 3 to 6 are explained by the fact that neem oil quenchant had a cooling rate that is about 31% that of water which was not sufficient to rapidly transform austenite to martensite but to bainite as observed in figures 3 and 4. Thus it produced an intermediate structure of bainite in pearlite. Quench holding periods of 30 and 60 minutes were also not sufficient to enable bath lose enough heat that could bring it down to temperature at which martensite could be formed. The 90 and 120 minutes holding period allowed more time for formation of pearlite that showed more presence in figure 6 because of longer period of soaking than steel for figure 5. The implications of these phase differences are that austempered steel corresponding to the photomicrograph in figure 3 would exhibit higher tensile, yield and impact strengths; higher elongation and hardness than those corresponding to figure 4, 5, 6 in that order. Conversely as ferrite and Pearlite were most present

in steel corresponding to microstructure in figure 6; reduction in area and elongation which showed ductility of the samples was higher than those of specimen corresponding to figures 5, 4 and 3 in that order of descent. Results of mechanical property tests presented in figures 7 to 12 showed that steel sample austempered for 30 minutes displayed the highest tensile, yield and impact strength and hardness. This was followed by specimen austempered for 60, 90 and 120 minutes in that order showing a trend of gradual reduction in the properties with increasing austempering quench holding time. However percentages of reduction in area and elongation (Figures 9, 11) showed a reversed order of this trend.

The main reasons for these are explained by differences in microstructural phases produced in samples by the heat treatment. Variation in quantities of bainite, pearlite and ferrite phases impacted different properties on the steel. Bainite improved hardness, impact, yield and tensile strength while pearlite and ferrite phases improved ductility. This is in line with previous heat treatment studies (Vijendra Singh, 2012) [12]. When the result is compared with initial mechanical properties of untreated steel presented in table 5 and figure 2, showed that steel austempered for 30 minutes (figure 3) had improvement of 5.97% ultimate tensile strength; 8.44% impact strength; 3.68% hardness and 10.6% in yield strength. Similarly, result in figure 4 for steel austempered for 60 minutes showed improvements of 5.19% ultimate tensile strength; 0.61% impact strength, 2.79% hardness and reduction of 30.25% in yield strength over untreated steel. 90 minutes austempering enhanced properties of steel by 4.93% ultimate tensile strength; 0.40% impact strength and 2.66% hardness. Its yield point wasn't clearly determinate during test. Steel austempered for 120 minutes showed property reduction of 0.53% ultimate tensile strength; 0.94% impact strength; 1.6% hardness and 47.73% yield strength. Thus longer austempering in neem oil quenchant, caused softening rather than strengthen steel due to low oil thermal conductivity.

CONCLUSION

Austempering was used in this work to strengthen locally produced Nigerian steel using neem seed oil as quenchant to ascertain its suitability for production of automotive shafts and gears. In comparison to ASTM 897 mechanical property limits for production of automotive shafts and gears presented in table 1, result of this study showed that though some improvement in property was

achieved by austempering Nigerian steel in neem oil quenchant, none (except the impact strength) was suitable for production of automotive gears and shafts of high standard. In order to achieve specified level of mechanical property requirements, carbon content of the steel must be increased to promote increased quantity of bainite formation or by introduction of acceptable percentage contents of austenite formation and stabilization alloying elements like nickel and chromium into steel at point of solidification (Bain, Edgar C. 1939).

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