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Evaluation of Nigeria's Local Fluids as Industrial Quenchants for the Heat Treatment of Tool

K.K. Ugwuagbo[1], O.P. Elenwo[2], and I. Uchegbulam[3]
Department of Physics, University of Port Harcourt, P.M. B5323, Choba, Rivers State, Nigeria. Corresponding
E-Mail: *onyinyechi.elenwo@uniport.edu.ng*

Abstract— The study examined the impact of quenching heat treatment on the mechanical, physical and microstructural properties of AISI 8250 Alloy Steel using vegetable oils as quenchants. Palm oil, Groundnut oil and Shea butter were compared to water-quenched and the annealed sample used as control. Heat treatment was run up to 850oC austinitic temperature and fast-cooled in these quenching media before tensile, hardness and metallographic analyses were carried out. The Ultimate Tensile Strength (UTS) of the samples were increased when quenched in Water, Palm oil, Ground nut oil and Shea butter by 69.01, 57.75, 42.25 and 38.73% respectively. Also, the surface and core hardness of the samples increased in the order of Water>Palm oil>Groundnut oil>Shea butter, while the surface to core hardness gradients of 85.29, 81.25, 90 and 100% was recorded for Water, Palm oil, Groundnut oil and Shea butter respectively. The results suggest that water quenching may only be recommended due to its very high strength and hardness potentials provided a post-quenching heat treatment such as tempering will be used to improve its adverse effect of cracking tendencies. However, Palm oil was the best quenchant when a good balance between mechanical strength, hardness and crack resistance was desired in addition to its sustainability and environmental friendliness. Study has shown Palm oil's additional value apart from its food demands and its potency as a favorable replacement for water in quenching heat treatment in any industrial, commercial and domestic processing of materials.

Keywords— Quenching; Tool Steel; Heat treatment; Sustainability; Environmentally Friendly.

I. INTRODUCTION

Quenching is a heat treatment process that rapidly cools a work piece to enhance hardness by refining grain structures and prevents undesired phase transformations by limiting the time for thermodynamically favourable reactions to occur. In metallurgy, quenching is mainly used to harden steel by transforming austenite into martensite, while the choice of quenchant affects cooling rates and material properties. Material Scientists plays very crucial role in modern life, developing new materials and improving on existing ones are fundamental to material science development and advancement.

Furthermore, the study of materials focuses on how their atomic, micro, and macro structures affect properties and performance. This property-performance correlation has been one of the reasons for the breakthroughs celebrated in the research and development of novel materials.

In recent times, the need for green production where due consideration is given to safeguarding the environment has become paramount and even a legal issue in modern science and technology. The need to develop sustainable materials and environmentally friendly ways of material development is yet to be fully imbibed into the material science culture. In this light, this study joins the research community to engage more biodegradable quenchants into the industrial heat treatment technology. Rejects, scraps and defective products are highly prohibited in a sustainable production system, (Elenwo et al., 2024). One way of waste reduction is through enhance property processing.

Hence, as a property enhancement process through controlled solid-state heating and cooling cycles that introduce desirable properties (Agboola, et al., 2020), heat treatment has been deployed in metals, ceramics and composites.

In so doing, the primary focus becomes how to integrate microstructural evolutions into material property enhancements in a sustainable and environmentally friendly manner.

Based on this, the present study investigates the effects of quenching with different vegetable oils to determine the most suitable quenchant for strength and hardness improvement.

Several authors have attempted this before using different materials and methods. For instance, Okwonna, et al., 2017, explored the use of palm kernel oil esters for austempering high and medium carbon steels. Their results showed that the Palm oil quenched samples had better hardness values than the as-received samples.

Similarly, Adekunle, et al., (2020), blended vegetable and Jatropha oils in the ratio of 7:3 for heat treatment of Aluminum alloys with resourceful results obtained in terms of cooling rate, the heat transfer coefficient, the Grossman quench severity.

Similarly, Isamotu, et al., (2020), compared the potentials of palm oil and palm kernel oil as quenchants for low carbon steels. The results showed that palm kernel oil had superior yield strength while palm oil made significant advancement in tensile strength, impact strength and Hardness values.

Also, Agboola, et al., (2015) made a comparative assessment of SAE40 engine oil and tap water to the potentials of Nigerian vegetable oils like cotton seed oil, palm kernel oil, neem seed oil and palm oil.

Their results show that the vegetable oils were suitable as alternative quenchant to the conventional engine oil and tap water for medium carbon steels with the best being Palm kernel oil.

In all the literature that studies Nigerian local oils as quenching media for heat treatment, none have considered AISI 8260 alloy steel which is widely used for making crankshafts, gears and other tough components. Hence, this study explores the potentials of Nigerian local oils as quenching media for AISI 8260 Alloy Steel.

Aim of study: The aim of the study was to evaluate the effects of quenching with different vegetable oils in Nigeria to determine the most suitable quenchants for strength and hardness improvement in materials.

II. MATERIALS AND METHODS

Material preparation

The steel sample chosen for this study was AISI 8620 machined from a Tricycle crankshaft. This sample is a low carbon alloy steel specially designed for case hardening by carburizing for a tough core with hard surface.

To understand the composition of the alloy steel sample, elemental composition evaluation was conducted by energy dispersive X-Ray Fluorescence analysis using Oxford Instrument X-Met 7000 XRF Spectrometer (Oxford Instruments plc, England, UK).

Five positions were scanned to compare the results and it was observed that the precision was high leading to the result presented in table 1:

Table 1: *Elemental Composition of alloy steel Sample*

Element	**C**	**Cr**	**Mo**	**Si**	**Mn**	**Ni**	**P**	**S**	**Fe**
Wt %	0.21	0.54	0.20	0.16	0.81	0.57	0.03	0.04	Balance

Five samples were machined from the sample using a center lathe to a gage length and diameter of 28 mm and 5 mm respectively to enable a firm grip on the chuck of the Mosanto tensometer, a cylindrical grip section of 8 mm in length and diameter was also fabricated.

Three Vegetable oil quenchants comprising of groundnut oil, shea butter oil and palm oil were compared to deionized water quenching. These vegetable-based quenchants were procured from a food market at Choba, Port Harcourt Nigeria while the deionized water was purchased from the Biotechnology laboratory at the university of Port Harcourt, Nigeria.

The samples were given specific coding according to the quenching media used as shown in table 2.

Table 2: *AISI 8260 Sample Codes*

Sc	**Control sample tested without heat treatment process**
SW	Austinitized to 850ºC, held at that temperature for 60 minutes and water-quenched.
SP	Austinitized to 850ºC, held at that temperature for 60 minutes and quenched in palm oil.
SS	Austinitized to 850ºC, held at that temperature for 60 minutes and quenched in shea butter oil.
SG	Austinitized to 850ºC, held at that temperature for 60 minutes and quenched in groundnut oil.

III. METHODS

The procedure followed in carrying out the experiments are shown in the flow chart in figure 1.

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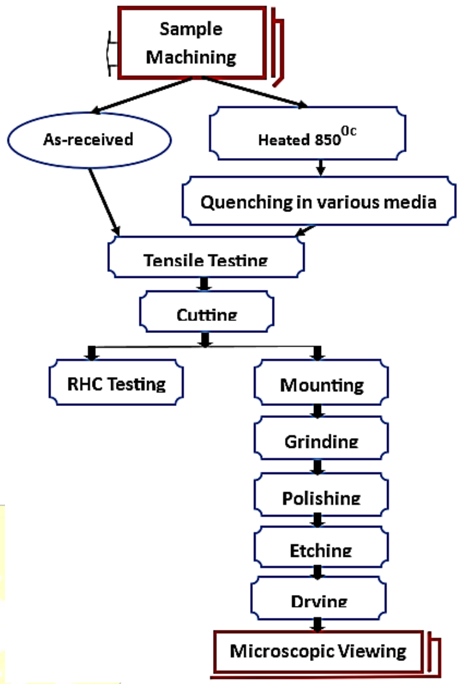


Figure 1: *Flow Chart of the Experimental Procedure*

After machining out five samples from the Tricycle crankshaft, one sample was reserved as a control in the As-received condition while the others were loaded into the electric furnace for the heat treatment process. The furnace was gradually heated up to 850ºC and held at that temperature for 60 minutes to thermally homogenize the heat in the samples. after this soaking time, the door of the furnace was opened and the samples were quickly transferred into the respective quenchants. The media were gently agitated to ensure controlled and uniform cooling to avoid development of soft spots. This also minimized the formation of bubble and steam pockets to form. After the samples cooled to room temperature, they were retrieved and cleaned for physical and mechanical analysis.

Tensile testing: To evaluate plastic deformation, the tensile test was conducted to study the response of the samples to axial loading. The standard ASTM E8 procedure was followed as reported in Uchegbulam et al., 2019. The test started by first measuring the initial diameter and gage length of each sample.

The graph paper was mounted on the autographic drum and the cross arm (cursor) placed on the zero-force zero-displacement position. The five test samples were loaded into the chuck, locked and the hand wheel turned steadily so that a controlled loading impacts on the test pieces. The reaction of the tricycle crankshaft steel samples to this loading was recorded on the graph paper attached on the data drum which was later studied after the samples finally got fractured.

Cutting and mounting of samples: After the samples passed through the tensile testing, their metallographic microstructures were studied by first cutting off a small piece about 5mm2 from both the As-received and the heat-treated samples using the Hacksaw and Bench vise.

The cut sample was impregnated with a thermosetting phenol powder using a hot mounting press. The die of the mounting press was first warmed, the sample dipped into it and finally filled with the phenolic powder. The top die was used to cover the powder before mounting into the mounting compaction chamber and compressed when the electric power was heated. After an auto-regulator was off, the compact was removed for grinding.

Grinding and Polishing: Different abrasive papers were used ranging from grit size 220, 320, 400, 600, 800, 1000 and finally 1000 grit size. These abrasives were used to grind the specimen into a scratch-free sample surface.

Next, the ground specimens were polished on an emery cloth on which diamond paste mixed with water was poured on as a polishing reagent. After a mirror like shiny surface was obtained free from surface scratches, the sample was deemed as being ready for etching.

Etching: An etching reagent consisting of 2% HNO3 (Hydrogen trioxonitric (VI) acid) and 98% Alcohol was used to etch the sample surface for two (2) seconds to prickle the polished surface. The aim of this process was to eat off dirt from the sample surface so that the microstructural components can be clearly exposed for viewing. After dipping the polished sample into the etchant, the wet surface needs to be rinsed off to avoid corrosive attack from the acidic deposit of the etchant (acid). This was done by dipping it into distilled water and inserting the rinsed sample into a blower chamber for drying making the samples ready for viewing on the metallographic microscope.

Viewing: The polished samples were placed on the stage of the metallographic microscope carefully while avoiding contact of anything with the etched surface to avoid contamination. The magnification was x100 (10 objective lens and 10 eyepieces). After a clear resolution was observed at the eyepiece, the image was revealed on a computer screen and was printed for analysis.

Hardness Testing: A portion of the fractured tensile sample was also cut to be subjected to Rockwell hardness testing. The core hardness of the five samples was carried out by making a flat surface from the hardness test samples in line with ASTM E18 Standard. The flat surface was ground with abrasive papers of different grit sizes to get rid of dirt and artifacts that may interfere with the expected hardness results. The ground surfaces were also polished to reveal a mirror-like surface before mounting them on the jig of the Rockwell hardness tester. As a hardened surface, Rockwell C scale (HRC) suitable to the ASTM E18 requirements for hard surfaces. This involved using a diamond cone with 120° cone angle where 10 kgf and 150 kgf minor and major loads were applied. To establish a reference loading, the minor laod of 10kgf was first applied before the 150 kgf was applied and allowed to stay for a 15 seconds dwell time. At the end of the 15 minutes' dwell time, the major load was removed leaving the minor load still on the sample surface. To achieve precision, Rockwell hardness number (HRC) measurement process was repeated three times and the average was taken.

Similarly, to measure the surface microhardness of the AISI 8620 alloy steels samples were measured following the procedure outline above for core hardness measurement. This was done by placing the horizontal surface of the samples on the jig of the hardness tester according to ASTM E92 standard.

IV. RESULTS

Ultimate Tensile Strength

From the result in figure 2, it could be seen that the water-quenched sample had the highest ultimate tensile strength. This was followed by that of the palm oil quenching and groundnut oil while the shear butter offered the least ultimate tensile strength. Based on this, with reference to the annealed AISI 8260 alloy steel used as received, the ultimate tensile strength of the sample was increased when quenched in water, Palm oil, Ground nut oil and Shea butter by 69.01, 57.75, 42.25 and 38.73% respectively. This result was different from that obtained in Adeyemi and Adebayo, (2009) where Groundnut oil quenched carbon steel was higher than the Palm oil quenched sample.

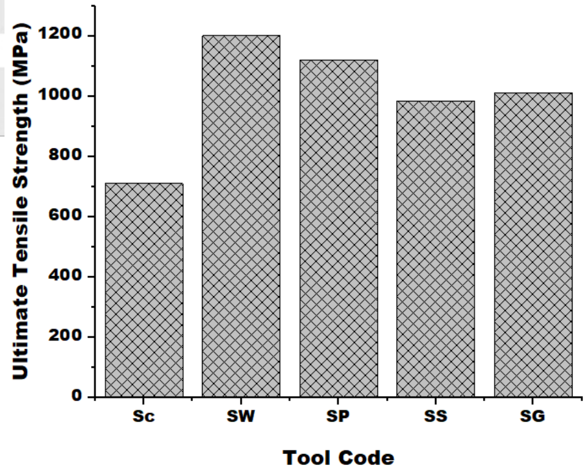


Figure 2: *Ultimate Tensile Strength of Annealed and Quenched AISI 8260*

Surface and Core Rockwell Hardness

From the result in figure 3, it can be seen that the sample had uniform hardness from the surface to the core. However, by subjecting it to quenching heat treatment, it can be noticed that the water-quenched sample had the highest surface and core hardness values. This was followed by that of the palm oil quenching and groundnut oil while the shea butter offered the least hardness.

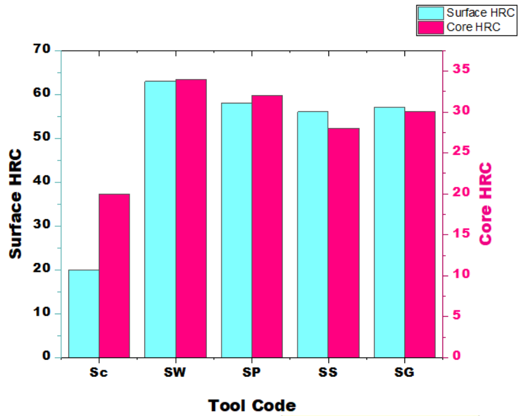
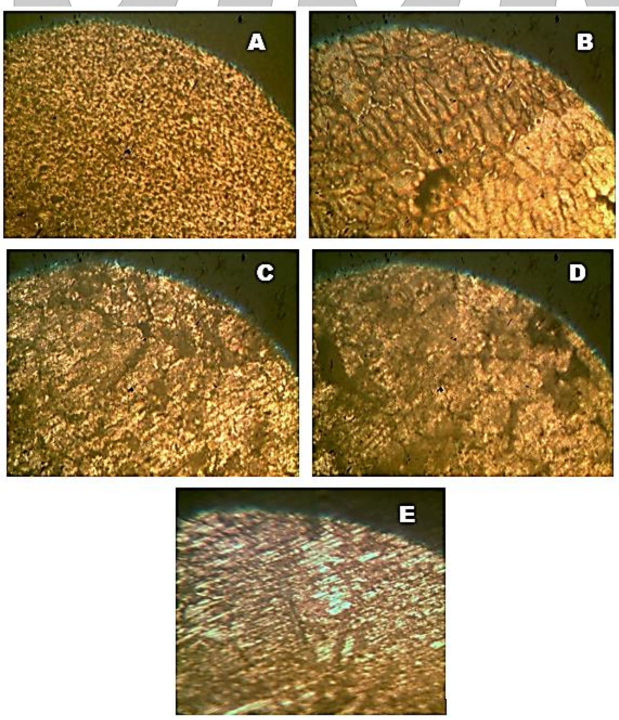


Figure 3: *Surface and Core Rockwell Hardness of Annealed and Quenched AISI 8260*

Based on this, with reference to the annealed AISI 8260 alloy steel used as received, the surface and core hardness of the sample was increased when quenched in the order of water, Palm oil, Ground nut oil and Shea butter. Remarkably, while the annealed sample had zero hardness gradient between the surface and the core, the samples quenched in water, Palm oil, Groundnut oil and Shea butter had surface to core hardness gradients of 85.29, 81.25, 90 and 100% respectively. This shows that the sample quenched in shea butter will be prone to cracks due to hardness difference between the core and surface while the sample quenched in palm oil is the least.

V. METALLOGRAPHIC RESULTS

Metallographic micrographs of the samples are presented in figure 4. It can be seen that figure 4a had a fine dispersion of a black phase evenly distributed with a white matrix. It is believed that this black phase was martensite. This result suggests full transformation of retained austinite into a martensitic phase. In comparison to the mechanical and physical tests, Martensite is known for its hard and strong properties and this can be linked to the high degree of ultimate tensile strength and very high HRC values obtained with the water quenched sample. Also, there was a demonstration of grain growth in micrograph B where the sample was quenched in Palm oil which was slightly similar to the martensite microstructure in micrograph A. In this Palm oil quenched sample, it can be noticed that the larger grainsize relates to the longer cooling rate where the thermal conductivity of Palm oil was relatively lower than that of water leading to longer cooling rates and larger grainsize with lesser Ultimate tensile strength and hardness.



Plates; A,B,C,D, and E: Shows Optical Micrographs of different quenching of AISI 8260 alloy steel in different quenching media (a) Quenched in water (b) Quenched in Palm Oil (c) Quenched in Groundnut Oil (d) Quenched in Shea Butter (e) Annealed sample used as received.

Remarkably, the micrographs C and D are similar and had coarser grain size. These were the samples quenched in Groundnut oil and Shea butter respectively. The very large grain sizes depict very long cooling rates resulting in less hardening and less strength. Finally, micrograph E is the control sample which was used as received in its annealed form. The very highly coarse grain sizes are strong indications of no prior heat treatment. The very coarse microstructure is responsible for the low levels of both strength and Rockwell hardness values.

These result shows that quench heat treatment increases the ultimate tensile strength and hardness of AISI 8260 Tool Steel but the quenching media determines the strength and hardness levels.

VI. CONCLUSION

In this study, three Nigerian local oils were used as quenching media for heat treated AISI 9260 tool steel in comparison to water-quenched and the annealed samples. Microscopic evaluations linked the strength and hardness values recorded in the study to the transformation of retained austenite phase into a martensitic phase in eutectic carbide matrix. This martensitic phase appeared in different grain sizes and dispersion in the different vegetable oils and water as quenching media. Experimental results also revealed that that the water-quenched samples recorded highest ultimate tensile and Rockwell Hardness values compared to the vegetable oils. In summary:

- Water quenching offered the highest tensile strength, core and surface hardness values but demonstrated the highest risk of cracking.
- Palm oil provided a good balance between moderate strength and hardness with reduced distortion
- Groundnut oil demonstrated an average strength and hardness but increased risk of cracking.
- Shea butter resulted in the lowest strength and hardness in addition to increased risk of cracking.

Based on this, it worth concluding that Palm oil quenching is the best quenchant when a good balance between mechanical strength, hardness and crack resistance are desired.

However, water quenching will be recommendable due to its very high strength and hardness potentials provided a post-quenching heat treatment such as tempering will be used to improve the adverse effect of cracking tendencies of water quenching.

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