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Effects of Bath Temperature on Cooling Rate, Mechanical Properties and Microstructure of Medium Carbon Steel during Quenching Operations

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Abstract
The effects of variations of bath temperatures of Selected Nigerian Vegetable oils on cooling rates, mechanical properties and microstructure of the quenched steel samples were investigated in this work. Cooling curves at different bath temperatures have been experimentally determined. From the cooling curves, the cooling ability variations were then analysed. The results obtained show that increasing bath temperatures increased the cooling rates of the quenched steel. These cooling rates are found to enhance mechanical strength with limited ductility of the quenched carbon steel. The hardness and tensile strength for palm kernel quenched sample increased from 40.8 HRC to 43 HRC. However, percentage elongation and impact values decreased from 0.28% to 0.21% and 9.5 N/mm^2 to 7.5 N/mm^2 respectively as bath temperature increases from 35 °C to 100 ºC. The results also showed that the microstructure of the quenched steel samples can be changed and significantly improved by varying the bath temperature. Reasons for variation in mechanical properties and microstructure are discussed.
Keywords: Quenching, temperature, vegetable oil, steel, cooling rate, mechanical properties.

1. Introduction

Quenching is a heat treatment method that has been used for many thousands of years and is still of importance today [1]. The process is performed to change the microstructure of metals and in turn their mechanical properties. The cooling rate must be fast enough so that a phase transformation occurs to obtain a required microstructure. The effectiveness of quenching depends on the cooling characteristics of the quenching medium and the ability of steel to harden [2]**. T**herefore the choice of effective quenching medium during heat treatment is very critical in ensuring the achievement of desired mechanical properties. The choice of quenching medium is dependent on the desired properties, structures, amount of distortion to be tolerated and carbon composition of the quenched steel [3].

Quenching occurs in three stages [4]. Stage A, is called the "vapour blanket" stage and is characterized by the formation of a vapour film around the component. It forms when the supply of heat from the surface of the part exceeds the amount of heat that can be carried away by the cooling medium. This is a period of relatively slow cooling during which heat transfer occurs by conduction through the vapor blanket. Upon further cooling, Stage B, the nucleate boiling stage begins during which the vapour film collapses and cooled quenchant comes into contact with the hot metal surface resulting in violent boiling. The highest heat transfer rates occur in this stage due to the heat of vapourization. The duration of the vapour phase and the temperature at which the maximum cooling rate occurs depends on different properties of the quenchant and has a critical influence on the ability of the steel to harden fully [5]**.**

As cooling continues, Stage C, which is the third stage called the convection stage is entered. This final stage begins when the surface temperature of the steel sample is reduced to the boiling point of the quenchant and the metal surface is completely wetted by the fluid. Below this temperature, boiling stops and slow cooling takes place by conduction and convection. The difference in temperature between the boiling point of the quenchant and the bath temperature is a major factor influencing the rate of heat transfer in liquid quenching [6]. Cooling rate in this stage is slower than in the second stage and is highly influenced by the viscosity of the quenchant.

Oils are classified by their ability to transfer heat as fast - medium - or slow - speed oils [7]. Speed oils are used for low hardenability alloys, and large cross-sections that require high cooling rates to produce maximum properties. Medium oils are used to quench medium to high hardenability steels and slow or marquench oils are used where hardenability of steel is high enough to compensate for the slow cooling aspects of this medium.

Over the years, a great variety of fluids have been used for quenching, including water, brine solutions, mineral oils and polymer quenchant. They all have different kinds of cooling ability and are used for different kinds of products [8]. Although water quenching is faster and less costly than oil quenching, the degree of distortion particularly in casting of complicated shape that accompany water quenching can be very high and therefore in such cases oil quenching, which is less severe than water quenching is generally preferred [9]. Petroleum oil derived quenchants are used when lower cooling rates and more uniform cooling is desired for better distortion control and crack prevention [10]. However, because of environmental concerns and growing regulations over contamination and pollution, associated with mineral based oils , vegetable oils has been identified as safer, renewable and biodegradable alternative quenchant to petroleum based oil .Palm kernel oil has been established as a viable alternative quenchant for medium carbon steel [11] .

Mechanical properties of steels are strongly connected to their microstructure [12]. Achieving desired mechanical properties and minimizing the possibility of occurrence of quenching cracks are the key indicators of successful hardening process. Apart from the hardenability of the alloy, the geometry of the part, and the quenchant used, the effectiveness of quenching depends on a number of other external factors such as temperature, the effective heat Transfer Coefficient between the metal piece and the quenchant, agitation and concentration of additives in the quenchant [13]. It is important to understand how these parameters affect the quenching procedure to get control over the mechanical properties, distortions and risk of cracking. In the present investigation, the effects of variations of bath temperatures of Selected Nigerian Vegetable oils on cooling rates and mechanical properties of the quenched carbon steel samples were investigated.

One way to evaluate a quenchant is to relate it to the quenchant's ability to extract heat from the work piece. The other way is to relate it to the results given when hardening a metal work piece in the quenchant [13]. The relative quenching performance of the vegetable oils was conducted at quenchant bath temperatures of 35, 50, 70, and 100°C using cooling curve analysis. Since the hardness pattern formed within the steel sample indirectly indicate the quenching ability of the oil, hardness of the steel samples was measured using Rockwell hardness tests. The Microstructure obtained was analyzed by using Philips XL-30 metallurgical microscope at 400× magnification

Heat transfer coefficients are used for calculating the convection heat transfer when cooling a solid in a fluid. It is a measure of a material's ability to release heat.

The heat transfer coefficients are calculated from the cooling curve by using Equation (1) (F.P. Incropera et al., 2007).

$$\frac{q}{A*\Delta T} \qquad [W/m^2k] \qquad (1)$$

where q= the heat flow input per second,

A= heat transfer surface area

ΔT= difference in temperature between the solid and fluid.

Hardening power is related to the ability of a quenchant to harden steel. The hardening power for unalloyed steel when quenched in oil is calculated according to the following equation:

Hardening Power = 91,5 + 1,34 * Tvp + 10,88 * CR550 – 3,85 * Tcp (2)

where Tvp= transition temperature between the vapour and boiling phase,

CR_{550}= cooling rate at 550°C

Tcp = transition temperature between the boiling and convection phase.

Equation (2) is experimentally determined based on measurements according to the ISO standard 9950 for oils. Oils with high hardening power give a faster quenching rate.

2. Materials and equipment

2.1 Materials

The materials used for this study was medium carbon steel obtained from Ajaokuta steel company limited, Ajaokuta, Kogi State, Nigeria. The average chemical composition of the steel is given in Table 1. The quenchant under investigation (Cotton seed oil, Neem seed oil, palm kernel oil and palm oil) are typical vegetable oils produced in Nigeria. Previous investigations [1] have shown these oils to be of various levels of saturated oil with the main contributors' being lauric, myristic, palmitic and stearic acid. Previous investigations have also shown the oils as possible alternative quenchants to petroleum based oil.

Table 1. Chemical composition (wt %) of the medium carbon steel used.

Element	C	Si	Mn	P	S	Cr	Fe
% Composition	0.357	0.16	0.75	0.032	0.041	0.1	98.2

2.2 Equipment

An electric furnace Model HAS 1508-0611NH, with a maximum temperature of 1200°C was used to austenite the steel sample for the purpose of quenching. The tensile tests on the specimens were conducted using an electron universal testing machine (Model: INSTRON 3369). A digital micro hardness testing machine of 490.3 MN load sensitivity (Model: Leco LM 700 AT) was used for hardness measurements across the cross-section of the quenched specimens. An Izod impact testing machine was used for the impact test done on the quenched specimens. A metallurgical microscope with an in-built camera was used to examine and photographically record the microstructure of the quenched steel samples.

3. Methods

3.1 Cooling Rate Determination Procedure

The experimental investigation was conducted using a standard probe made of a cylindrical medium carbon steel specimen of 12.5 mm diameter × 60 mm long machined from a 16 mm rod obtained from Ajaokuta steel company limited, Ajaokuta, Nigeria. The composition of the carbon steel used is shown in Table 1. A type K thermocouple was inserted in a hole of 3mm diameter drilled through the specimen centre and care was taken for the thermocouple to be rigidly fitted to make good contact with the bottom of the specimen's drilled hole. A standard tensile test specimen of 5mm diameter and 20mm long were machined from the same steel rod. In all, four tensile specimens were machined for tensile test. Four standard impact test specimens of 10mm square by 55mm long were also machined for the impact test [7].All the test specimens were heated in an electric furnace at a heating rate of 25ºC/sec to a temperature of 850º C and then allowed to soak at this temperature for about 10 min. At the same time the beaker containing the oil was heated on a hotplate to the required temperature, after which two each of the austenized tensile specimen, one impact test specimen and the standard specimen were quenched in each of the oil. Quenching of these samples was conducted at quenchant bath temperatures of 35, 50, 70, and 100°C. The effect of bath temperature of the quenching medium on cooling rate and mechanical properties of the quenched steel samples were investigated. The temperature versus time curves at various temperatures was recorded using a card data logger Model RD 8900 connected through a cold junction, maintained at a temperature of 0°C. Critical cooling parameters [8] were determined.

3.2 Measurement of Mechanical properties

The hardness values determinations were carried out on test – pieces cut from the middle portion of the quenched specimens. The hardness impressions were taken transversely in two perpendicular directions along the cross-section of the quenched specimens by a Digital Micro Hardness testing machine (Model: Leco LM 700 AT) under applied load of 490.3 MN and dwell time of 10 seconds using a' C' scale (HRC). Hardness numbers taken at different points were automatically read from the digital counter and the average value was taken. Two repeat tests were performed on each specimen and the average taken as representative of the hardness obtained for the corresponding treatment.

The tensile tests on the specimens were conducted using an electron universal testing machine (Model: INSTRON 3369). The cylindrical test specimens used were of 5.0mm in diameter and gauge length of 20 mm (Figure 3.3). In carrying out the test, one end of the specimen was gripped in the jaws attached to the adjustable crosshead and then after lifting this crosshead to the appropriate height, the other end of the specimen was fixed in jaws in the top crosshead. The tensile load was hydraulically applied to the specimen by pressing the start button provided in the control unit. The input and output display of applied load unit in the control unit indicated magnitude of applied load. The load was gradually increased until specimen broke off and corresponding extension was noted and recorded. The automatically generated test data were shown on the display unit. The impact test was carried out on an Izod impact testing machine to measure the toughness of the quenched specimens according to ASTM D256. Optical micrographs of quenched specimens were carried out on sectioned surface grinded with silicon carbide papers of different sizes, using Daheng microscope. They were subsequently polished using a cloth impregnated with alumina until a mirror surface was obtained. Grinded surface were cleaned with water and ethanol. Etching with 2% Nital was done and microstructures observed using a high powered optical microscope.

4. Results and Discussion

Figures 1 - 4 shows the effect of the bath temperatures on the rates of cooling with thermocouple cooling temperature. Cooling rate curves (Figure 1 – 4) were obtained by taking the slope at each temperature of time versus temperature curves.

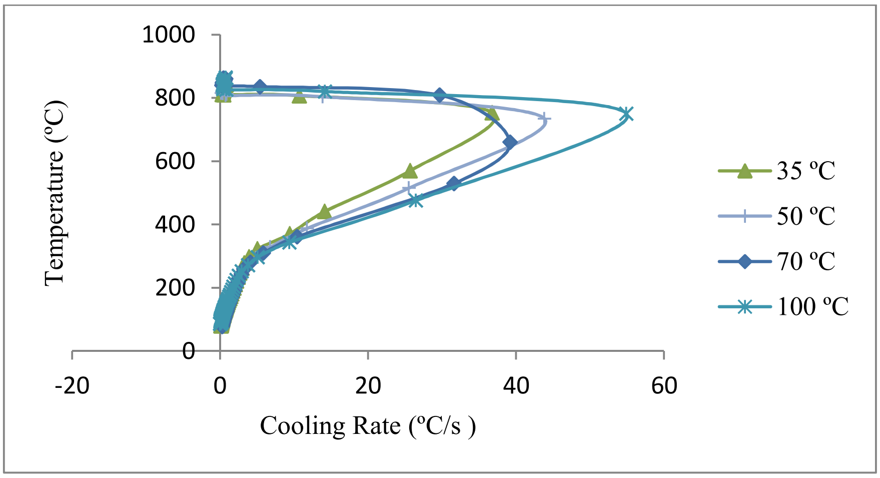

Figure 1. Cooling Rate/Temperature Curves of Cotton seed oil at varying bath temperatures

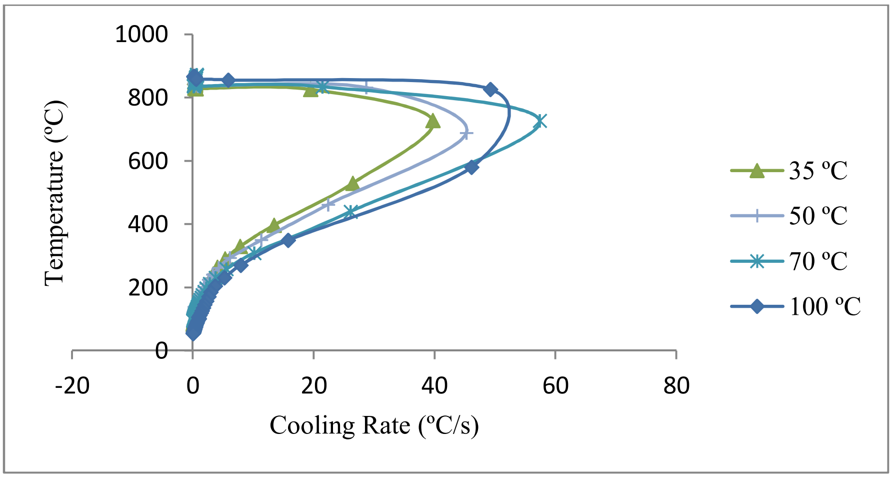

Figure 2. Cooling Rate/Temperature curves of neem seed oil at varying bath temperatures

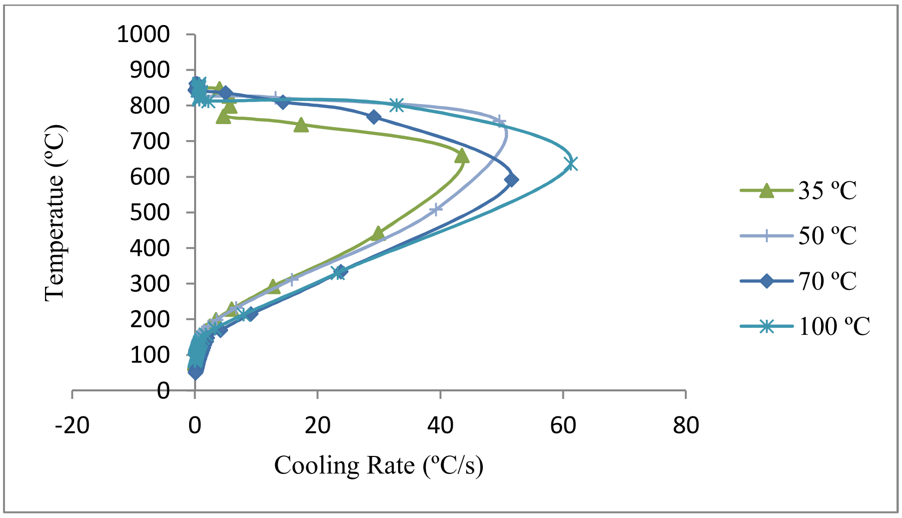

Figure 3. Cooling Rate/Temperature curves of palm kernel oil at varying bath temperatures

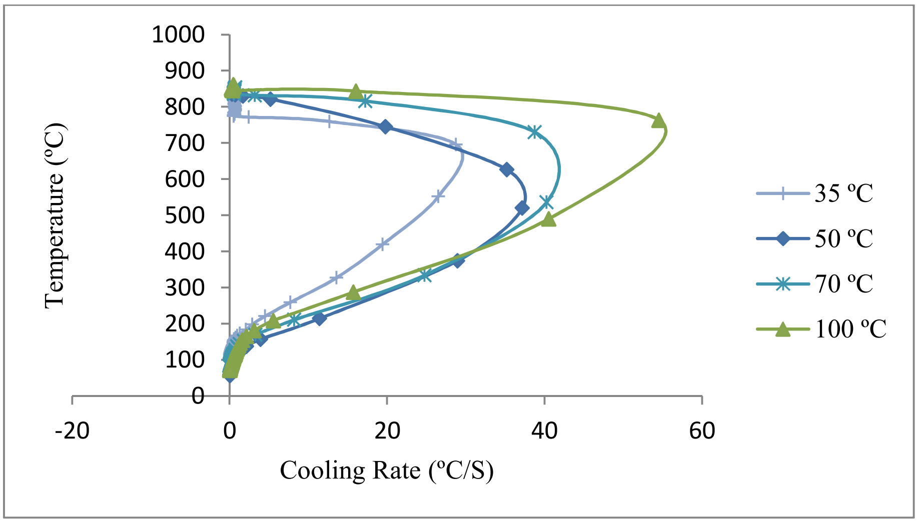

Figure 4. Cooling Rate/Temperature curves of palm oil at varying bath temperatures

Increase in bath temperature resulted in the cooling rate curve shifting to the right (see Figures 1- 4). This gives an increase in cooling rate and maximum cooling rate CR_{max} . Tvp and Tcp moved to the left, i.e convection phase start earlier. The result is that the boiling phase is occurring earlier at higher temperature. Higher bath temperature leads to increase in cooling rate

As shown in Figures 1 – 4, increasing the bath temperature produces slightly faster cooling in stage A because of drop in viscosity of the oil. With increase in bath temperature, cooling is slightly increased in stage B, and in stage C cooling decreases near the end of the quench because the temperature difference between the quenchant and the steel surface is decreased.

Figures 5 to 8 show the effects of varying bath temperatures of cotton seed oil, neem seed oil, palm kernel oil and palm oil on hardness, tensile strength and percentage elongation and impact values of steel samples quenched in the oils.

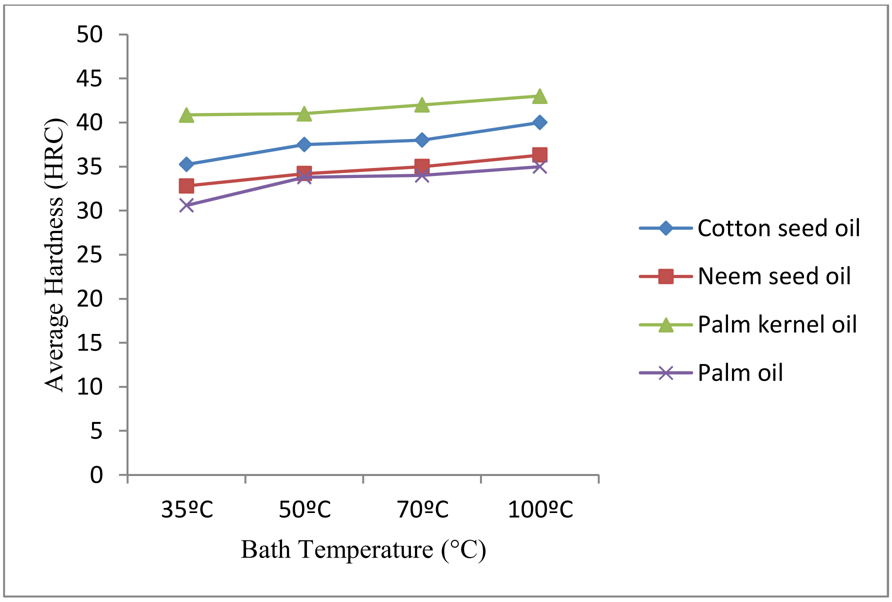

Figure 5. Effect of bath temperature of Different vegetable oils on Average Hardness Values of medium carbon Steel

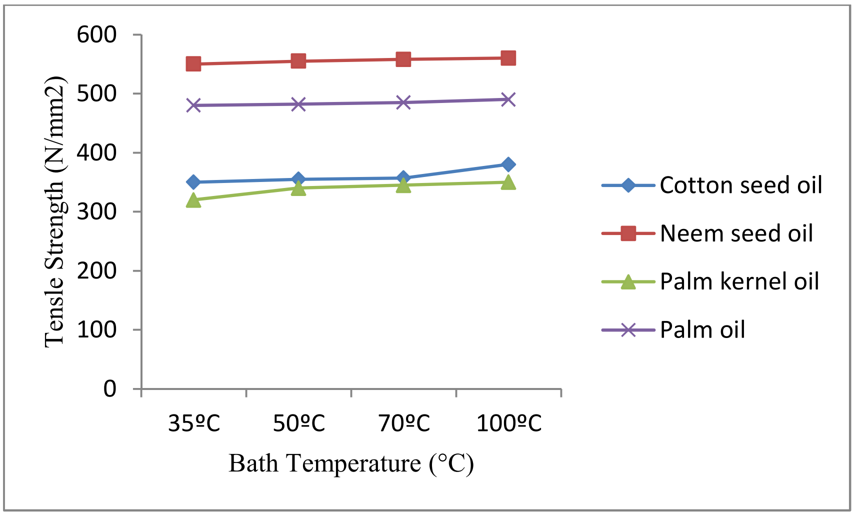

Figure 6. Effect of bath temperature of Different vegetable oils on Tensile Strength of medium carbon Steel

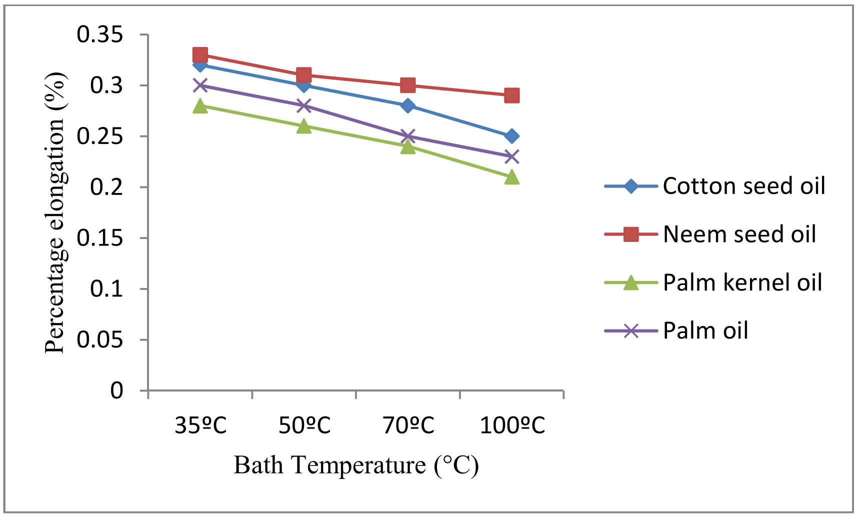

Figure 7. Effect of bath temperature of Different vegetable oils on percentage elongation of medium carbon Steel

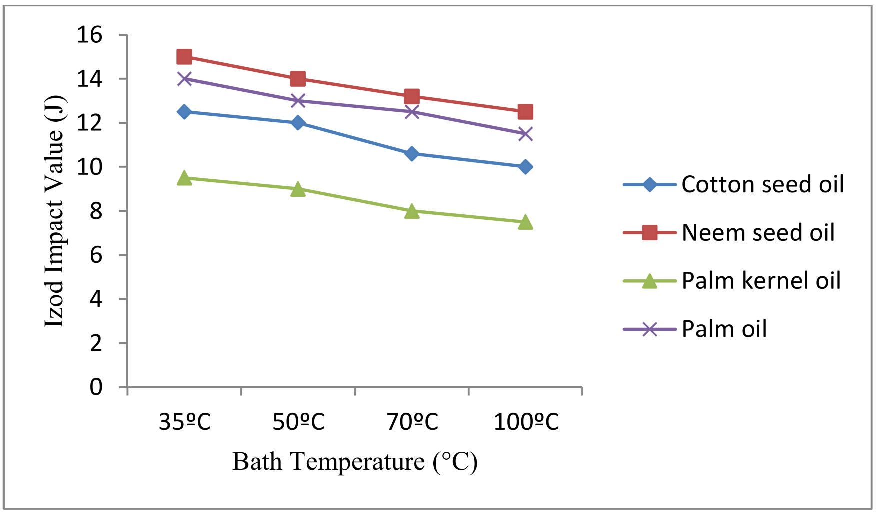

Figure 8. Effect of bath temperature of Different vegetable oils on Impact Toughness of medium carbon Steel

As shown in Figure 5, hardness of the steel samples increased with increase in quenching temperature from

35ºC to 100 °C. Palm oil and cotton seed oil quenched sample had higher change in hardness values between 35 °C and 50 °C compared to other samples .This may be due to the faster cooling rate of the oils as a result of drop in viscosity as the temperature increased. The high level of unsaturation of the fatty acid molecules in cotton seed oil must also have contributed to the high cooling rate.

The tensile strength of the steel samples increases from 320 Mpa to 350 Mpa for palm kernel oil as the bath temperature increases from 35 °C to 70 °C but have no significant change while exceeding 70 °C. The increase in tensile strength as bath temperature increased showed that the bath temperature influenced the strength of the steel sample. The effect of varying bath temperature is more pronounced in cotton seed oil which may be due to the level of unsaturation.

Figure 3 shows that percentage elongation of the steel sample decreased from 28 % to 21 % for palm kernel quenched sample ; 32 % to 25 % for cotton seed oil quenched sample,30 % to 23% for palm oil quenched sample and 33 % to 29 % for neem seed oil quenched sample. Percentage elongation decreased with increasing bath temperature for all the oils. However the elongation was least in neem seed oil quenched samples.

Effect of bath temperature on impact energy is shown in Figure 4. With increase in bath temperature from 35ºC to 100 °C, impact energy of the steel samples also decreased. The different properties observed are as a result of different cooling rates produced by different bath temperatures in different quenching media. Distortion can be remedied by quenching at high bath temperatures. However, one must know the optimum temperature required for different oils and steel grades. To achieve this, optimization of bath temperature using appropriate method is useful.

In general, the effect of bath temperature was higher in neem seed oil quenched samples than in other steel samples which may be due to the high level of unsaturation of fatty acid molecules in the oil which aids fast cooling rates.

The microstructures of the carbon steel samples quenched in palm kernel oil at 35 °C, 50 °C, 70 °C and 100 °C are shown in Figures 9, 10, 11 and 12 respectively.

Figure 9. Microstructure of Steel Sample Quenched at 35°C (400X).

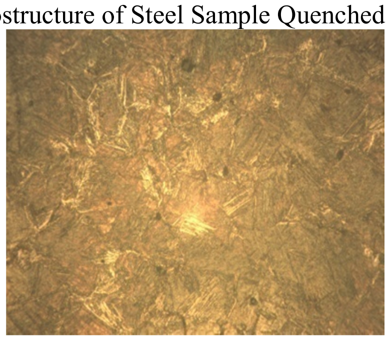

Figure 10. Microstructure of Steel Sample Quenched at 50ºC. (400X)

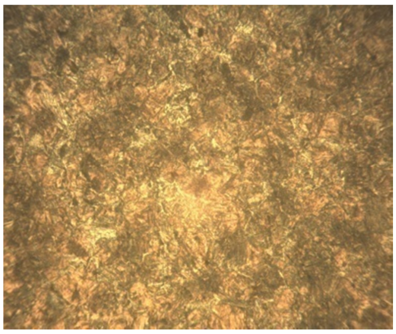

Figure 11. Microstructure of Steel Sample Quenched at 70°C. (400X)

Figure 12. Microstructure of Steel Sample Quenched at 100ºC. (400X)

The photomicrographs of the carbon steel sample quenched at 35 °C revealed bainite structures with a mixture of pearlite while the specimens quenched at 100 ºC showed traces of martensitic structure (see figure 12) thereby making the hardness of the material to increase as the bath temperatures increases. The different microstructures observed are as a result of the different rates of cooling at different bath temperatures.

5. Conclusion

It has been established that bath temperature of the quenchant has significant influence on the cooling rate, hardness value, tensile strength, percentage elongation and impact toughness of carbon steel during quenching.

Cooling rates increased with increased bath temperature due to rapid transformation of the film boiling to convective cooling.

Hardness and Tensile Strength of quenched steel samples increased with increase in bath temperature by as much as 15% with50% increase in bath temperature. Impact strength and percentage elongation change slightly by increasing quenching temperature.

As bath bath temperature increases, cooling rate and microhardness also increases.

The effect of varying bath temperature is highly reduced between 50 ºC and 70 ºC. Optimum cooling rates were obtained within this temperature range.

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