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Influence of Austempering Temperature on Physiochemical and Microstructural Properties of Ductile Cast Iron (ADI) with Selected Local Oil Quenchants

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A B S T R A C T

Austempering ductile irons (ADI) also refer to as nodular cast iron is an enriched carbon-iron alloy with some outstanding characteristics have marked it highly suitable for application in manufacturing industries. In past decades, the use of molten salt or lead are widely used in quenching ductile cast iron due to its highly soluble in water and tends to surmount phase barriers that exist during quenching. One challenging factor with the use of salt bath in a developing country like Nigeria is that its expensive and difficult to maintain. Non- edible vegetable oils (jatropha and neem) that are readily available and exhibits good quenching characteristic was selected as substitute to brine in ADI. Ductile iron was austenized at 900 oC held for 90 min, then quenched at varying temperatures (250 – 450 oC) and held isothermally at constant time (1800 s). The influence of temperature on the physiochemical and microstructural properties was assessed on each quenched specimens. Results show that ADI is dependent on austempering temperatue and its corresponding holding time. Specimens austempered in both oils at 450 oC for 1800 s was found to be adequate to develop the necessary enhanced mechanical properties and enriched microstructures than other temperatures.

Keywords: *Austempering, Ductile Cast Iron, Bainite, Temperature, Quenchant, Microstructure.*

1. Introduction

Cast iron, one of the oldest ferrous metals predominantly used in our present automobile and industrial engines [1]. It consists of iron (Fe), silicon (Si) and carbon (C) with some traces of other elements. It possesses some characteristics such as brittle, it cannot be hammered, stretch or bend into shape. It exhibits high tendency to fracture with little deformation, this make cast iron not suitable for applications where sharp or flexible edges are required but are suitable under a compressive condition [2]. In the past decade, researches have results in the production of desirable engineering materials tailored for industrial applications with adequate toughness, corrosion resistance and high tensile strength [3]. Ductile iron, otherwise known as spheroidal graphite cast iron or nodular iron is known for its excellent castability, outstanding mechanical properties and less expensive [4,5].

The recent progress in cast irons is the formation of bainite in the matrix of spheroidal graphie cast iron instead of the pearlite. Austempered ductile iron (ADI) castings, belonging to the family of cast-irons and is obtained by the heat treatment of ductile iron. It requires an interrupted quenching (Austempering), usually into a salt bath at a predetermined rate to achieve desirable austempered result. This results to a combination of exceptional mechanical and physical characteristics such as simplified casting processes, higher consistency of quality, less processing cost, good castability, 10 % lower density than steel, good ductility and wear resistance, excellent resistance to crack propagation, Tensile and yield strength values are twice to that of standard ductile iron [6]. These outstanding marked properties make it potential for numerous engineering applications in the engineering industries such as machine - building, civil engineering, transport, mining, military industry and in the production of brake shoes, crankshafts, axles, friction blocks, camshaft, bearing sleeves, connecting rods, abrasive protection liners, pulleys, rack and pinion gearing, rocket arm [7,8]. The microstructure structure is

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obtained by a systematic heat treatment process of nodular cast iron otherwise known as ductile cast iron. It involves heating to the austenitization temperature (850 - 950 oC), hold at that temperature. It is followed by fast quenching to avoid the formation of pearlite to a temperature relatively above the martensite start (Ms) generally known as austempering temperature (between the temperature range 250 - 450 oC, depending on heat treatment parameters). It is then hold at that temperature for a determined time until austenite is transformed into bainite in steels and ausferrite in ductile cast iron and is slowly cooled to atmospheric temperature to prevent the development of thermal stresses. Molten salt is most widely recognised quenchant for this process due to its ability to extract heat rapidly with consistent viscosity within a wide range of temperature; soluble in water and overcomes phase barrier during the first stage in quenching [9-11]. Molten still show some disadvantages such as; it can be dangerous or hazardous to experts during heat treatment, cleaning process as produces toxic vapours, surface material tends to corrode faster when left for a period of time in the bath [11] and the cost of running the salt bath in a developing country like Nigeria is relatively high to maintain. Considering the disadvantages with the use of salt bath, the need for an alternative quenchants becomes imperatively important to consider. This study seeks to bridge the gap in the use of non-edible oils as alternative to brine water as quenchants for ductile cast iron (DCI). Two (2) non-edible oils (jatropha and neem vegetable oils) were used as alternative to the use of molten salt as quenchant for the ductile cast iron (DCI). Influence of varying austempering temperatures was evaluated to ascertain their suitability and test for mechanical properties.

2. Experimental Procedures

The main concept of the experiments was to heat treat these steel specimen (nodular cast iron) at different austempering temperatures ranges from 250 ^oC – 450 ^oC within intervals of 100 ^oC in some selected non-edible vegetable oils such as jatropha and neem oils. The quenched specimens were subjected to mechanical properties such as hardness, tensile and microstructural analysis. The ductile base iron material of 50 × Ø12 mm rods was obtained from the National Metallurgical Research Centre (NMDC), Jos, Nigeria. Chemical composition of these rods were analysed at the Nigeria Geological Survey Agency, Kaduna State, Nigeria is summarised in Table 1. The quenching media used in the study are non-edible vegetable oils such as; Jatropha oil and neem oils. Oils (jatropha and neem seed) was extracted locally from the seed plants and transferred to an extraction chamber of soxhlet extractor for final oil extraction which was then stored into a round bottom flask for further investigation.

2.0.1 Austempering

Physiochemical properties that directly or indirectly affect the quenching media use in the course of the study such as saponification value, iodine values, peroxide values, specific gravity, viscosity, free fatty acid and viscosity were measured and recorded. The specimens were heated to an austentised temperature of 900 ^oC and soak for 90 min. The isothermal transformation of austenite was obtained in quench bath at varying temperature of 250, 350, 450 ^oC and held isothermally at this temperatures for 30 min and allowed to cool naturally under still air.

2.0.2 Mechanical Investigation

Mechanical properties of the as-received nodular cast iron material, austempered ductile iron after heat treatment that directly affects the materials was evaluated, properties such as ultimate tensile strength (maximum tensile strength in Mpa), yield strength (Mpa), percentage elongation (%), Hardness (HBW) and impact energy (Joules). The mechanical properties were evaluated on specimens after isothermal heat treatment.

2.0.2.1 Hardness Measurement

Four section of the specimen was selected as appropriate to evaluate the macro-hardness of the ductile iron specimen in A scale. An indenter Rockwell hardness testing machine (Model-6187.5B) at room temperature under an applied load of 187.5kg and remain for about 10 seconds. Hardness values were automatically read from the digital counter and average harness value was recorded respectively. The obtained hardness values were converted to terms of required hardness values such as Brinell's or Vickers hardness values.

2.0.2.2 Tensile Strength Test

The tensile test material was machined to standard specified in BS 2789:2002. Hounsfield Tensiometer testing machine was used to carry out the ultimate tensile strength using the stress-strain curves. Percentage elongation was calculated.

2.0.2.3 Impact Strength

Impact strength was measured on nodular iron steel specimen subjected under isothermal transformational heat treatment condition. A 150 Joules Charpy capacity with a striking velocity of 5.24 m/s on the notched face of impact specimen. Energy absorbed was measured as the impact strength of the material. An average of three reading was measured and recorded.

2.0.3 Metallography Analysis

Metallography examination was observed on the ductile cast iron specimens after been subjected to heat treatment condition. Specimens were etched using 2 % nital for 10 – 20 sec after been polished on a rotary disc. The microstructures were observed in direct illumination on a metallographic microscope (Model: NJF-120A) with camera attached.

3. Results and Discussion

3.0.1 Chemical Composition

The chemical composition of heat treatment material is summarised in Table 1 below.

Table 1: Chemical Composition of Material used.

Elements	C	Si	Mg	Mn	P	S	Ni
% Composition	3.82	2.28	0.048	0.39	0.041	0.011	0.54

Table 1, shows the chemical composition of ductile base material used in the study. It meet the minimum requirement to be use for heat treatment .

3.0.2 Physiochemical Properties of the Quenching Media

Physio-chemical properties that directly affect the quenching performance was evaluated its influence in quenching ductile cast iron are summarised in Table 2.

Table 2: Physiochemical Properties of the Quenchants

Parameters	**Jatropha oil**	**Neem oil**
Saponification Value (mgKOH/g)	179.62	184.96
Iodine value (meq/g)	56.38	60.02
S. G. at 15/4^{0}C	0.9228	0.9141
Viscosity (Mpa)	68.35	61.23
FFA (mgKOH/g)	4.03	8.36
Flash point (^{o}C)	283	271

Vegetable oils predominantly consist of fatty acids in glycerin molecule. They are generally polar substances exhibit long carbon chains and can exist as unsaturated and saturated acids [12, 13]. The unsaturated fatty oils (carbon-carbon double bonds) constitute a stronger molecular adhesion (highly reactive and unstable) to metal surface. The iodine number of the oils varies according to the degree of unsaturation present in the oil [14] and it indicates amount of reactive radicals present in the oil and by extension, the instability of the fatty acids molecules film that forms on the metal surface [15].
According to these stated properties, neem vegetable oil exhibits least effective as austempering oil due to the instability of the film, higher iodine value, higher saponification value and lower flash point. On the other hand, jatropha vegetable oil gave the best austempering characteristics as it had the least instable film due to its low iodine value [11,16]. Results obtained as indicated in Table 2 shows that neem oil like seed oils has very high degree of unsaturation due to a higher percentage of unsaturated fatty acids present in the oil, while jatropha oil has the least value of unsaturated fatty acids and therefore the most stable at high temperature.
The oils viscosity, flash point is presented in Table 2. Jatropha oil was found to exhibit higher viscosity of 68.35 cSt and flash point of 283 ^{o}C. This variation in viscosity justifies the temperature dependence of the oil characteristics as shown in the results. The higher viscosity of a quenchant, the lower is the rate of heat extraction from the steel and consequently the better the probability of producing bainite in the austempered cast iron (expected microstructure for desirable ADI properties). This is due to the considerable thermal resistance that exists in viscous oils [11,17]. This confirmed that vegetable oils in ADI quenching performance is absolutely composition dependent as it relates to the use of vegetable oil as alternative for mineral oil and salt bath for ADI quenching applications.

3.0.3 Mechanical Properties

Mechanical properties such as tensile strength, ductility, hardness and impact energy after heat treatment was evaluated as it affect the quenching characteristics of ADI in both vegetable oils is shown in Table 3 below.

Table 3: Mechanical properties of specimens Heat Treatment (Jatropha oil)

Specimen	**U.T.S (Mpa)**	**Y.S (Mpa)**	**% Elongation**	**Hardness (HBW)**	**Impact Energy (J)**
Basic material	982	780	0.4	97	12
250 ^{o}C/1800s	1600	1290	0.8	535	31
350 ^{o}C/1800s	1320	940	5	450	64
450 ^{o}C/1800s	1400	1100	1.2	462	47

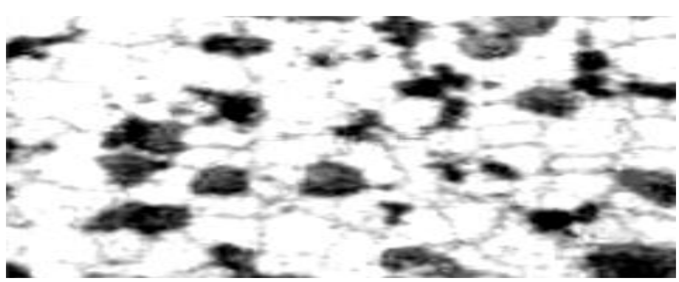
Table 4: Mechanical properties of specimens Heat Treatment (Neem oil)

Specimen	**U.T.S (Mpa)**	**Y.S (Mpa)**	**% Elongation**	**Hardness (HBW)**	**Impact Energy (J)**
Base material	620	465	0.7	98	14
250 ^{o}C/1800 s	1200	848	2	389	55
350 ^{o}C/1800 s	800	500	11	285	92
450 ^{o}C/1800 s	950	606	09	346	79

Table 3, shows a maximum tensile strength of 1600 Mpa was measured with specimen quenched in jatropha oil with a corresponding increase in yield strength (1290 Mpa), lower percentage elongation (0.8%) at austempering temperature of 250 ^{o}C while the least tensile strength (1320 Mpa) was observed at isothermal transformation temperature of 350 ^{o}C. Maximum tensile strength (1200 Mpa) was measured using specimen quenched in neem vegetable oils with corresponding increase in yield strength (848 Mpa) and a decrease in elongation (2%) at austempering temperature of 250 ^{o}C. Least tensile strength (800 Mpa) with corresponding yield strength (500 Mpa) and higher elongation of 11% was measured at austempering temperature of 350 ^{o}C (see Table 4). Generally, percentage elongation is a parameter in measuring the ductility of a material. Ductility exhibits steady increase with increase in impact energy and a corresponding decrease in hardness, tensile and yield strength. This decrease in hardness value may be attributed to drastic decrease of martensitic structures, higher percentage of ferrite than pearlite present in the matrix for both quenchants used at increasing austempering temperature. Increase in austempering temperature shows corresponding increase in ductility. Increase in ductility may be attributed high presence of stable carbon austenite around the microstructure [18]. Steel specimen quenched in selected vegetable oils shows improvement in mechanical and microstructural properties when compared to the as-received specimens.

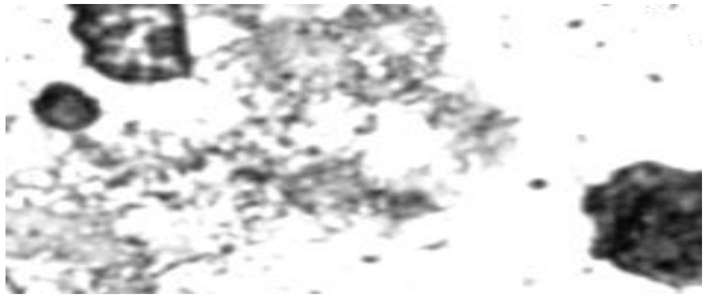
3.0.4 Microstructural Analysis

Micrography 1 – Microstructure of as-received nodular cast iron (200×). Structure consist of graphite nodule surrounded by ferrite ring (white) in a pearlite matrix (black).

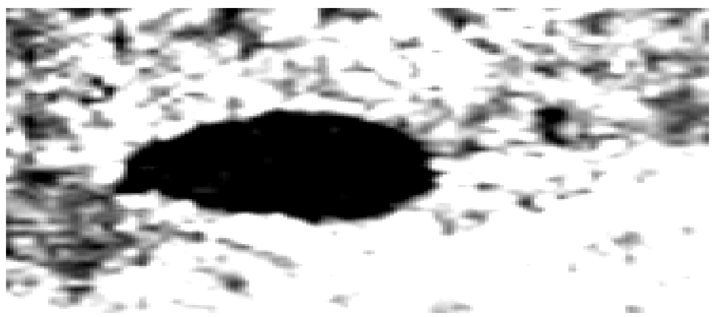


Micrography J_1 – Austempered microstructure of nodular cast iron at 250 ^{o}C quenched in jatropha oil (200×). Structure consisit of mixture of martensite structure with traces of austenite structures (white).

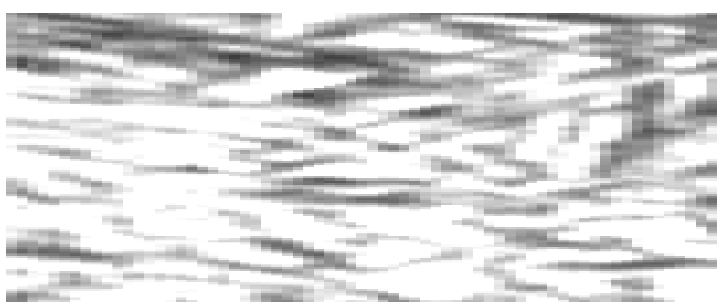
Micrography N_1 – Austempered microstructure of nodular cast iron at 250 ^{o}C quenched in neem oil (200×). Structure consisit of mixture of martensite matrix with traces of austenite structures (white).



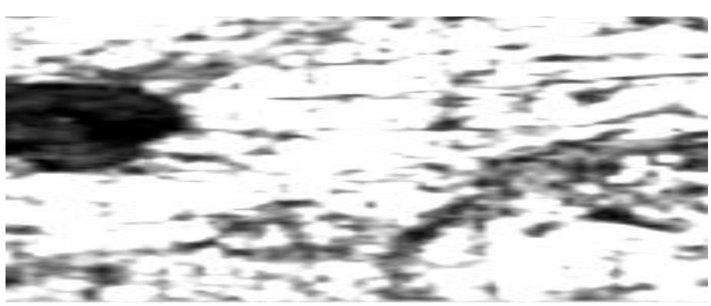
Micrography J_2 – Austempered microstructure of nodular cast iron at 350 ^oC quenched in jatropha oil (200×). Structure consisit of mixture of martensite with traces of austenite structures (white).



Micrography N_2 – Austempered microstructure of nodular cast iron at 350 ^oC quenched in neem oil (200×). Structure consisit of mixture of martensite with traces of austenite structures (white).



Micrography J_3 – Austempered microstructure of nodular cast iron at 450 ^oC quenched in jatropha oil (200×). Structure consisit of Needle-like bainitic ferrite (ausferrite) structures with an enriched residual carbon austenite



Micrography N_3 – Austempered microstructure of nodular cast iron at 450 ^oC quenched in neem oil (200×). Structure consisit of Needle-like bainitic ferrite (ausferrite) structures with an enriched residual carbon austenite

Metallographic structures for as-cast and heat treated specimens are represented in Micrography 1. Micrography J_1 - J_3 represents the specimens isothermally quenched in jatropha oil and Micrography N_1 - N_3 forspecimen quenched in neem oil. The microstructure of as-received nodular cast iron material shows graphite nodules in pearlite matrix (Micrography 1). The effect of austempering temperature on microstructure of austempered ductile iron is shown in micrography J_1 - J_3 and N_2 - N_4.

Micrography J_1, N_1 a slight mixture of martensite along side with some traces of retained austenite (that was not reacted with) was observed during isothermally quenched in jatropha and neem vegetable oils at austempering temperature of 250 ^oC. Retained austenite may be identified with areas in white.

Micrography J_2 and N_2 constitute a mixture of martensite with some traces of austenite structures, but martensitic structures constitute the majority at this temperature. As austempering temperature increases to 450 ^oC, the degree of martensite structures decreases drastically with simultaneous increase in the formation of a needle like microstructure of bainitic ferrite (ausferrite) structures with an enriched residual carbon austenite (remain untransformed at austempering temperature) is the major microstructural constituent present (micrography J_3 and N_3). However, austempered specimens quenched in jatropha and neem oil showed more ausferrite structure than other specimens in both quenchants at 450 ^oC austempered temperature. At high austempering temperature, the formation of martensite structure is prevented which usually is one of phases formed in case of low temperature austempering [18].

4. Conclusions

Final microstructure and mechanical properties of ADI are markedly dependent on the isothermal transformation temperature of austenite and holding time at this temperature. The influence of temperature on the microstructure and mechanical properties of ADI using some selected vegetable oils has been evaluated with the following deductions:

i. Austempering at 450 ^oC in 1800 s produces a typical austempered ductile iron microstructure which consistutent a bainitic ferrite structure, stable and highly carbon enriched retained austenite present in specimens quenched in both non-edible vegetable oils.

ii. Quenched ductile iron in jatropha and neem vegetable oils shows significant improvement in mechanical and microstructure properties compare to as-received specimens.

iii. The tensile strength, hardness of quenched specimens in various vegetable oil decreases with increase in austempering temperature with a corresponding increase in ductility.

iv. ADI resembles British standard EN-1564:1997 and ASTM A897M-06, since the mechanical properties are similar to this grade of ADI

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