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Mechanical properties and microstructural evaluation of heat-treated aluminum alloy using formulated bio-quenchants

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ABSTRACT

Heat treatment industries require various quenching media to improve the properties of the materials to be quenched. Petroleum based mineral (PBM) oil, a non-biodegradable oil, is popular amongst others quenchants in heat treatment processes. Recently, biodegradable oils mostly in their raw, unblended and unbleached forms have been employed for quenching of various engineering materials. Therefore, the present study examined the effects of some selected bio-quenchants in blended raw (BR) and blended bleached (BB) forms on the mechanical properties and microstructure of solution heat treated aluminum (Al)-alloy. Edible vegetable oil (70% by volume) was blended with 30% by volume of jatropha oil to form the bio-quenchant oils. Another set of bio-quenchants were formed by bleaching the raw oils before mixing so as to reduce the oxidation level and contaminations in the oil. The Al-alloy is solution heat treated at 500 °C and soaked for 15 min in an electric muffle furnace before quenching in the various established bio-quenchants. Results showed that samples treated in blended raw melon (BRM) oil have higher tensile strength of 151.76 N/mm^2 while samples quenched in blended bleached melon (BBM) oil have higher hardness value of 61.00 HRC. In accordance to the results obtained the bio-quenchants were found to be effective replacement to the PBM oil.

KEYWORDS

aluminum alloy, bio-quenchant, mechanical properties, microstructure, solution heat treatment

1. INTRODUCTION

Aluminum alloys are known to be widely used in aerospace industry, automobile industry, machine parts construction, home appliance, electrical equipment and many other areas [1] as a result of their excellent corrosion resistance, good formability, and high electrical and thermal conductivities [2–4]. The strength of heat treatable Al-alloys can be improved by precipitation hardening which consists of three basic steps namely: solution heat treating, quenching, and aging. In solution heat treatment, the aluminum alloy is heated to a temperature range between 400 and 540 °C in order to re-dissolve the soluble alloying elements in solution to produce a single phase solution, that is, a solute-rich solid solution after holding at the liquidus temperature between 10 min for thin parts and 12 h for heavy parts while the general rule of thumb for soaking an inch of cross-sectional thickness part is 1 h. This is to allow for the diffusion of solute elements into the solvent matrix, and then quenched to a lower temperature (for example, room temperature) to keep the alloying elements trapped in solution.

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After quenching, the quenched aluminum alloys are aged. During aging, the solute elements are trapped in the solvent matrix to form evenly distributed particles. Al-alloys that undergo hardening process at room temperature after a few days are called natural aging and those that undergo harden process by reheating to an intermediate temperature are called artificial aging [5–7]. Rapid quenching is required to circumvent unwanted concentration of alloying elements along the boundary structure grain. More so, failure not to quench faster than necessary in order to lessen residual stress can result into excessive cracking or distortion [8].

Over the years, water and water solution have been the most commonly used quenchants for ferrous, non-ferrous metal, and their alloys because they are economical, non-toxic, readily available with no smoke generation, easily handled, and disposed off with no health or environmental concerns [9–11]. In addition, water and water-based liquids are the fastest quenching media due to their fastest cooling rate, which in turn results into high hardenability values and outstanding mechanical properties of the materials. Although, they possess high consistent cooling rate during quenching process, however; they can also cause problems like distortion and cracking due to high thermal gradients induced upon cooling [12]. The first quenchant developed as alternative to water and water based quenchants was petroleum-based quenching oil around 1880 by Houghton in Philadelphia. Since then, highly specialized and localized quench products have been in search [13]. Mineral oils have been established to have the best cooling capacity for engineering materials such as steel and aluminum alloy [14–18] but they are relatively expensive, toxic, non-renewable, and non-biodegradable [1]. Thus, they are subjected to ever-increasing controls due to stringent environmental regulations regarding their use [18]. Routine disposal into the environment causes negative effects to soil, underground water, and ecosystem and these consequently encourage the use of vegetable oil base stocks such as canola oil, groundnut oil, palm oil, and soybean oil [19–21] derivatives and so on for the formulation of environmentally friendly quenchants.

There have been various investigations on the use of vegetable oils as quenchants as reported in references [21, 22]. One of the studies involved the use of shea nut oil, palm kernel oil, and control sample (engine oil, SAE 20W-50) on 6061 aluminum material [22]. Tensile strength results showed that when the materials are quenched in shea nut oil and palm kernel oil at solutionizing temperature of 530 °C, the highest values obtainable are 88.0 and 90.1 N/mm^2, respectively, with the yield strength values of 66.72 and 64.94 N/mm^2, respectively obtained. Highest impact energy value of 65 J was obtained in sample quenched in palm kernel oil while the ultimate tensile strength for the control sample is 83.8 N/mm^2 and yield strength is 64.44 N/mm^2. Odusote et al. [3] evaluated the effects of vegetable oils such as groundnut, melon, palm kernel, shea butter, and palm oil as quenchants on the properties of pure commercial aluminum heat treated at different temperatures of 200, 250, 300, and 350 °C. The results indicated that the bio-quenching oil that provided the highest ductility is shea butter oil at 200 °C while groundnut oil gives the best result at 350 °C. Meanwhile, the highest hardness values were obtained from sample quenched in melon oil. Durowoju et al. [21] investigated the impact of severity and hardness using eco-friendly quenchants such as groundnut, shea butter, jatropha, melon, mineral, and palm oil as quenchants on heat treatment of AISI 4137 medium carbon steel. It was reported that melon oil and palm oil gave the highest hardness values of 657 and 649 HVN, respectively. To achieve an effective solution heat treatment of aluminum alloy, vegetable oil has been considered as alternative bio-quenchant to petroleum based mineral (PBM) oil, water, and water-based liquid. This is due to the increasing interest in tackling environmental challenges because vegetable oils are biodegradable, safe, offer great performance, cost effectiveness, and high productive advantages.

Therefore, this study aims at investigating the performance of aluminum alloy quenched in formulated bio-quenchant media in the blended raw (BR) and blended bleached (BB) forms. The effects of the formulated quenchants on the mechanical properties of solution treated cast aluminum alloy as alternative quenchant to PBM oil as well as the microstructural behavior of the material were examined.

2. EXPERIMENTAL STUDY

2.1. Materials

The materials used in this study were cast Al-alloy, edible raw vegetable oils (melon oil, groundnut oil, and palm oil) and non-edible oil (jatropha oil), bleaching agent (activated kaolin clay and commercial activated charcoal).

2.2. Preparation of bio-quenchant oils

Vegetable oils were bleached into refined form using adsorption bleaching technique. The percentage color reduction between the raw and bleached oils was measured using Ultra Violet/Visible Spectrophotometer captured at wavelength range of 350–550 nm. 70% by volume of each of the edible vegetable oils (melon oil, groundnut oil and palm oil) were blended with 30% by volume of non-edible oil (jatropha oil) in the raw and bleached form to formulate three (3) unrefined bio-quenchant oils and three (3) refined bio-quenchant oils making a total of six bio-quenchant media namely; blended raw melon (BRM) oil, blended raw groundnut (BRG) oil, blended raw palm (BRP) oil, blended bleached melon (BBM) oil, blended bleached groundnut (BBG) oil and blended bleached palm (BBP) oil while PBM oil namely Total Quenchant IID served as control for comparison. The fatty acid composition of the bio-quenchant oils were determined using Gas chromatographic (GC-MS) system model (7890AGC, 5675C inert MSD) with procedure using methyl ester derivatives of the different bio-quenchant oils. Also the physiochemical properties of the bio-quenchant oils such as kinematic viscosity, acid value, iodine value, saponification value, flash point, pour point, and free fatty acid were analyzed in accordance to International Standard.

2.3. Casting of Al-alloy rods

Al-alloy rods were produced using sand casting method to obtain 15 mm diameter cast cylindrical rods and then machined using ASTM E8/E8M and modified to test probe size of 10 mm diameter × 200 mm length as shown in Fig. 1. Chemical composition of the cast material (see Table 1) was analyzed using Olympus Delta Professional XRF at Midwal Engineering, Lagos, Nigeria.

2.4. Solution treatment of Al-alloy probes

The cast Al-alloy cylindrical rod of 27.27% Si was machined into a 10 mm diameter × 200 mm length probe with a type K-thermocouple screwed into a tight fitted hole of 5 mm diameter ×13 mm depth threaded to a depth of 10 mm centrally to one side of the probe for soaking temperature tracking from Digital SD card data logger. Prepared probe was then placed in an electric muffle furnace with solution treatment temperature of 500 °C and soaked for 15 min in order to homogenize the composition.

2.5. Quenching procedure

The heated probe was quickly transferred laterally under 2 s into a quench bath containing 1,000 ml of quenching oil at room temperature followed by natural aging for 5 days at room temperature of 35 °C before it was subjected to mechanical properties tests and microstructural examination.

2.6. Mechanical properties

2.6.1. Tensile test. Tensile tests were carried out on the specimens using Universal Tester model, 38140. Each of the specimens was mounted to the jaw of the machine and a gradually increasing load applied till fracture occurred and the data generated for tensile strength and percentage elongation on each specimen was recorded.

2.7. Hardness test

Rockwell hardness tester with steel ball indenter model, HR-150A was used for the determination of the hardness value of the as-received, as-cast and quenched specimens. Small piece from test specimen was placed on the anvil of the machine and then loaded to create a round indentation. The impression created was automatically recorded as hardness value through the dial gauge.

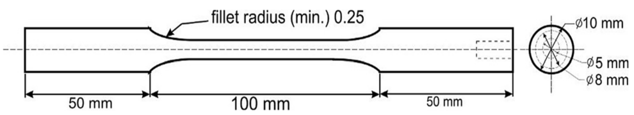

Fig. 1. Schematic diagram of the test sample for tensile test

2.8. Impact test

Impact test was performed on the samples using Charpy V-Notch pendulum impact testing machine. The specimens were prepared into dimension of 55 × 10 × 10 mm with 45° V-notch at the center. Single blow of the impact testing machine hammer of about 300 J was released to bend or break the specimen at the center. Readings were obtained for each specimen.

2.9. Metallography samples preparation

Samples for microstructure examination were prepared and analyzed at Obafemi Awolowo University (OAU) Ife, Nigeria using Accuscope Metallurgical Microscope, model 0524011. Cut samples of 10 × 3 mm square rods from the quenched samples were grinded using different grades of abrasive paper sizes in the order of 220, 400, and 600-grits to obtain a smooth surface finish under running water and then polished with cloth swamped with solution of 0.5 μm silicon carbide until a mirror-like surface was attained. The surface was then viewed under the metallurgical microscope.

3. RESULTS AND DISCUSSION

3.1. Bio-quenchant oils analysis

The gas chromatographic analysis of the formulated bio-quenchant oils using methyl ester derivatives revealed that the blended raw and BB bio-quenchant oils possess a di-glyceride structure namely: saturated fatty acid (palmitic and stearic) and di-unsaturated fatty acid (linoleic) as presented in Tables 2 and 3. The BB palm oil with lower double bonds showed the most stable form of fatty acid component which was as a result of high total saturated fatty acid (stearic and palmitic). BRM oil and BBM oil exhibited lower stability to oxidation due to its high total unsaturated fatty acid (oleic and linoleic) as reported by Kodali [23]. Moreover, the ultra/violet visible spectrophotometer analysis showed that BRP oil has the highest peak absorbance value of 3.701 Abs and BRM oil has the lowest peak absorbance value of 3.602 Abs with respective peak wavelength (λ) value of 534 and 388 nm as shown in Table 4.

Jatropha oil which was used to blend other vegetable oils was observed to be darker in color after bleaching due to its low thermal and oxidation stability and this is in agreement with the work of Kapilan and Birdar [24]. Thus, the BR and BB for both melon and groundnut oil exhibited negative value for change (Δ) peak absorbance and Δ peak wavelength value. However, the positive value obtained for Δ peak absorbance and Δ peak wavelength value in palm oil

Table 1. Chemical composition (wt%) of the as-cast Al-alloy

Elements	Al	Si	Cu	Mg	Pb	Fe	Ti
% Composition	70.20	27.27	1.25	0.61	0.003	0.14	0.12

Table 2. Percentage saturated fatty acid composition of formulated blended raw (BR) and blended bleached (BB) bio-quenchant oils

Bio-oils	Saturated fatty acids					
	Behenic $C_{22}H_{44}O_2$	Myristic $C_{14}H_{28}O_2$	Palmitic $C_{16}H_{32}O_2$	Stearic $C_{18}H_{36}O_2$	Arachidic $C_{20}H_{40}O_2$	Lignoceric $C_{24}H_{48}O_2$
Blended raw (BR)						
Melon oil	–	–	11.88	10.51	–	–
Groundnut oil	8.43	–	18.85	9.65	4.49	3.80
Palm oil	–	0.78	39.69	7.11	–	–
Blended bleached (BB)						
Melon oil	–	–	17.86	16.19	–	–
Groundnut oil	7.95	–	19.80	9.87	4.23	3.54
Palm oil	–	1.341	43.16	11.12	–	–

Table 3. Percentage unsaturated fatty acids composition of formulated blended raw (BR) and blended bleached (BB) bio-quenchant oils

Bio-oils	Unsaturated fatty acids		
	Oleic $C_{18}H_{34}O_2$	Linoleic $C_{18}H_{32}O_2$	Gondoic $C_{20}H_{38}O_2$
Blended raw (BR)			
Melon oil	14.86	51.69	–
Groundnut oil	–	52.35	2.44
Palm oil	41.92	10.49	–
Blended bleached (BB)			
Melon oil	–	65.94	–
Groundnut oil	–	52.32	2.29
Palm oil	14.95	25.14	–

was due to its rich carotenoids from which deep red color pigment is derived as explained by Poku [25]. Percentage (%) color reduction was highest in palm oil and lowest in melon oil. The specific gravity for the bio-quenchant oils is relatively close to each other as shown in Table 5. BBM oil was observed to have exhibited the highest flash point of 240 °C, lowest pour point of 9 °C, lower percentage free fatty acid of 1.169% and lowest kinematic viscosity of 31.87 mm^2/s at 40 °C while BRP oil exhibited the lowest flash point of 160 °C, highest pour point temperature of 19.5 °C, highest percentage free fatty acid of 2.76% and highest kinematic viscosity of 43.95 mm^2/s at 40 °C. This shows that the higher the free fatty acid, the lower the flash point. The kinematic viscosity change was observed to have direct relation to the degree of saturation/unsaturation of fatty acid in the oils, that is, kinematic viscosity decreases with an increase in unsaturation but increases with an increase in saturation and this is in agreement with the work of Kim et al. [26].

Chemical composition of the investigated cast Al-alloy sample is shown in Table 1. The mechanical properties conducted on the cast Al-alloy solution heat treated at 500 °C when quenched in each formulated bio-quenchant oils favors some specific mechanical properties as against the petroleum based mineral oil.

3.1.1. Tensile strength and percentage elongation. The tensile strength of as-cast Al-alloy was 131.37 N/mm^2 while quenched samples in BRM oil and BRP oil have higher tensile strength of 151.76 and 146.22 N/mm^2, respectively, as against 146.07 N/mm^2 for PBM oil (see Fig. 2). This may be

Table 4. Ultra violet/visible spectrophotometer of blended raw (BR) and blended bleached (BB) Bio-quenchant oil

Oil samples	Peak absorbance (Abs)	Peak wavelength λ (nm)	Integral value	*Δ Peak absorbance (Abs)	*Δ Peak wavelength λ (nm)	% Color reduction
Raw jatropha oil	3.614	398.00	241.47			
Bleach jatropha oil	3.647	486.00	270.41	−0.033	−88	−0.9131
BR melon oil	3.602	388.00	235.67			
BB melon oil	3.608	392.00	230.63	−0.006	−4	−0.1666
BR groundnut oil	3.611	394.00	223.68			
BB groundnut oil	3.615	400.00	224.00	−0.004	−6	−0.1108
BR palm oil	3.701	534.00	536.34			
BB palm oil	3.700	528.00	486.84	0.001	6	0.0270

*Δ – change in blended bleached bio-quenchant oils against the blended raw bio-quenchant oils.

Table 5. Physicochemical characterization of blended raw (BR) and blended bleached (BB) bio-quenchant oils at room temperature

	Blended raw (BR) oils			Blended bleached (BB) oils		
Quality parameter	Melon oil	Groundnut oil	Palm oil	Melon oil	Groundnut oil	Palm oil
Specific gravity at 35 °C	0.952	0.951	0.948	0.951	0.950	0.949
Kin. viscosity (mm^2/s) at 40 °C	32.34	33.24	43.95	31.87	32.99	38.33
Flash point (°C)	220	220	160	240	230	190
Pour point (°C)	13	15	19.5	9	12	18.7
Acid value (mgKOH/g)	2.328	2.418	5.509	2.098	2.224	4.89
Peroxide value	1.94	1.86	1.74	1.20	1.31	1.55
Iodine value (gI_2/100g)	42.44	46.00	41.30	34.11	32.30	40.04
Saponification value (mgKOH/g)	196.20	193.34	200.14	202.08	197.39	210.18
Moisture content (%)	0.70	1.50	1.50	0.20	0.80	0.80
Free fatty acid (%)	1.164	1.209	2.755	1.169	1.262	2.446
pH value	7.46	7.61	6.80	7.26	7.07	6.24

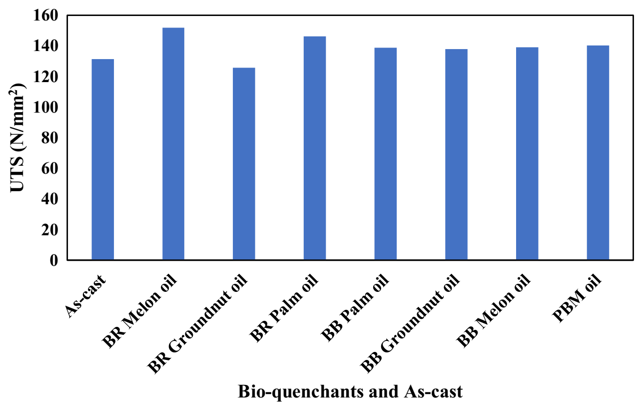

Fig. 2. Tensile strength of Al-Alloy in formulated bio-quenchant oils

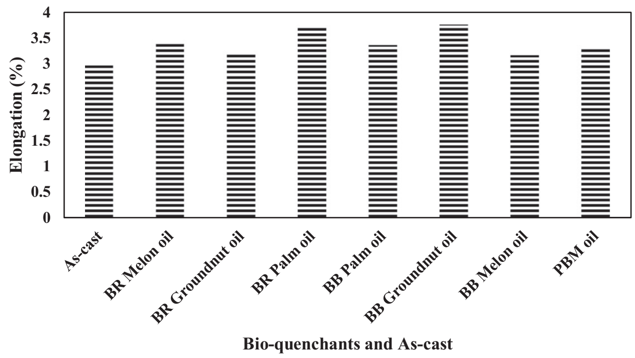

Fig. 3. Percentage elongation of Al-Alloy in formulated bio-quenchant oils

due to the presence of oleic acid value (above 10%) in their total unsaturated fatty acids and likewise the presence of high peroxide and iodine in the oils (see Table 4). The tensile strength obtained in other formulated bio-quenchant oils i.e., BBM oil, palm oil, groundnut oil, and BRG oil are 139.0, 125.86, 138.70, and 137.83 N/mm^2, respectively, which are slightly less than the value for PBM oil. This may be attributed to the lower peroxide and iodine value in the bio-quenchant.

However, the increase in tensile strength of all the quenched samples was largely influenced by the heat treatment temperature and quench behavior [21, 22, 27]. Percentage elongation of the cast Al-alloy was 2.96%, which was less than the elongation for petroleum based mineral oil of 3.30%. The percentage elongation obtained using BRM oil, BRP, oil and BBG oil as bio-quenchant were much higher than other bio-quenchants with their values been 3.40, 3.70, and 3.76%, respectively, as shown in Fig. 3. The increase in percentage elongation offered by all the bio-quenchants might be as a result of total saturated acid (palmitic and stearic) present in the oil. BRG oil and BBM oil offered the quenched samples the lowest elongation value of 3.19 and 3.16%, respectively, which was due to the high percentage of linoleic acid value; however, bio-quenchant oils with higher percentage of linoleic acid gave lower elongation value. Also the increase in percentage elongation was influenced by the presence of palmitic, stearic, and linoleic acid. The tensile strength of the samples using the formulated bio-quenchants decreases in the following order: BR Melon oil > PBM oil > BR Palm oil > BB Melon oil > BB Groundnut oil > BR Groundnut oil > As-cast > BB Palm oil, while the percentage elongation decreases in the order: BB Groundnut oil > BR Palm oil > BR Melon oil > BB Palm oil > PBM oil > BB Melon oil > BR Groundnut oil > As-cast. In general, tensile strength and percentage elongation of samples quenched in formulated bio-quenchant oils were influenced by their respective cooling rate, thus, leading to overall increase in percentage elongation which makes the material to be more ductile and less brittle as against the as-cast material and this is in agreement with the work of Rajan et al. [28].

3.1.2. Hardness and impact values. The Rockwell hardness of the as-cast material was 50.25 HRC but higher hardness values were obtained after solution treatment of the samples in various bio-quenchant oils with highest hardness value of 61.00 HRC obtained in samples quenched with BBM oil while lowest hardness value of 52.00 HRC was obtained in BBP oil. However, BRP oil, melon oil, groundnut oil, and

BBG oil have hardness values of 56.75, 55.50, 54.25, and 54.75 HRC, respectively, while petroleum based mineral oil has hardness value of 53.00 HRC, as shown in Fig. 4. The hardness value increases as the total unsaturated fatty acid (oleic and linoleic) increases and is in line with the work of Durowoju et al. [21], except for BRP oil, which was due to the presence of high oleic value above 40%. Also, BRM oil and groundnut oil with higher saponification and lower peroxide value as against its corresponding blended bleach bio-quenchants gave higher hardness value.

The impact strength value of the as-cast material was 69.83 J with high impact strength of 73.17, 75.33, 74.83, and 74.83 J achieved in samples quenched in BRG oil, BRM oil, BB melon oil and BBP oil, respectively; while the impact strength of samples quenched in BBG oil and BRP oil have 72.50 and 72.67 J, which is lower than that of petroleum based mineral oil of 72.83 J as shown in Fig. 4. BRM oil and BB melon oil, which have higher total unsaturated fatty acid (oleic and linoleic), gave the highest impact strength values while BBG oil and BRP oil with lower total unsaturated fatty acid gave the least impact strength values. The general increase in the impact strength values of the materials quenched in formulated bio-quenchants were in line with that reported by Abubakre et al. [22] and is due to the presence of palmitic, stearic, and linoleic acid in the oils. The impact strength values increased as the hardness values increased at solutionizing temperature of 500 °C for the bio-quenchant oils expect for BBP oil, whose impact strength value increases as the hardness value decreases and the impact strength value of BBG oil decreases as the hardness value increases as shown in Fig. 4; while the impact strength and hardness value of petroleum based mineral oil decrease.

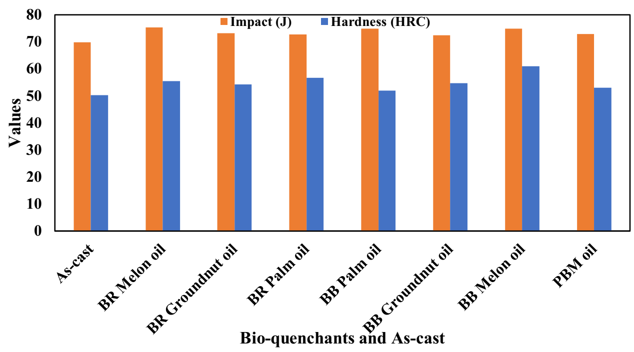

Fig. 4. Rockwell hardness and impact strength of Al–Si–Cu–Mg alloy in formulated bio-quenchant oils

3.2. Microstructure examination

Two major phases; primary Si-particles in light α-Al phase (light patches) and eutectic (α+Si) phase (dark patches) with Si-particles as displayed in microstructure of the as-cast and solution heat treated Al-alloy material showed large volume fraction of the Al-matrix as shown in Fig. 5 [29, 30]. In the as-cast Al-alloy, the material showed large concentration of Si-particles in the Al-matrix due to high grain growth in sand casting leading to lower mechanical properties like percentage elongation and hardness value as shown in Fig. 5a and is in agreement with the work of Raji [30]. The microstructure of cast Al-alloy materials quenched in the formulated bio-quenchant oils, as shown in Fig. 5b–h, showed that the mechanical properties such as tensile strength, impact strength, percentage elongation and hardness values of all the Al-alloy samples increased significantly as against the as-cast alloy materials. As

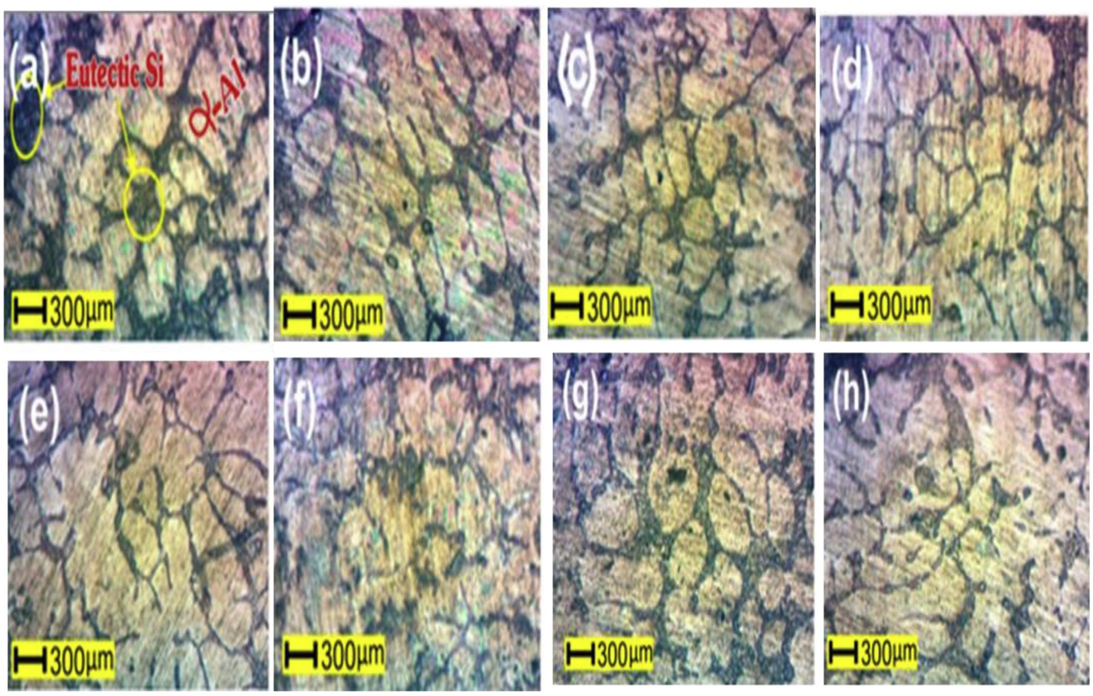

Fig. 5. Microstructure of: (a) As-cast Al-alloy (b) Al-alloy quenched in BRM oil (c) Al-alloy quenched in BBM oil (d) Al-alloy quenched in BRG oil (e) Al-alloy quenched in BBG oil (f) Al-alloy quenched in BRP oil (g) Al-alloy quenched in BBP oil (h) Al-alloy quenched in PBM oil

a result of grain structure refinement as the alloying elements dissolve thereby re-distributing the elements to form different precipitates within limited solution heat treatment time, mechanical properties of the quenched materials increase. BR and BB melon oil showed higher tensile strength, impact strength and hardness values. This may be attributed to better grain structure re-distribution of the grain structure under very short solution heat treatment time in cooling. The rapid cooling of the dissolved alloying elements in the Al-matrix gave rise to the rapid dissolution of the alloying elements at a short solution heat treatment time forcing the alloying elements to induce higher mechanical strengths.

Although, BBP oil exhibited lower tensile strength and hardness values which could be said to be influenced by its slow cooling rate, thereby resulting into lower fragmentation of the eutectic particles and other alloying elements after quenching as shown in Fig. 5g. The samples quenched in PBM oil have microstructure showing that the fragmentation of eutectic Si-particles into smaller sizes with even distribution in the Al-matrix results from the influence of rapid cooling effect of the oil.

4. CONCLUSION

The present study has evaluated the effect of 70% by volume of edible vegetable oils (melon oil, groundnut oil, and palm oil) blended with 30% by volume of jatropha oil on cast Al-alloy when BR and BB. The mechanical properties and microstructure examination of cast Al-alloys quenched in these formulated bio-quenchants have been examined. The tensile strength, percentage elongation, hardness value, impact strength, and microstructure of sample solution heat treated at 500 °C were improved with the formulated bio-quenchant oils compared to the industrial petroleum based mineral oil due to the influence of saturated fatty acid (palmitic and stearic) and unsaturated fatty acid (oleic and linoleic) present in the bio-oils. The general improvement in mechanical properties of quenched samples using formulated bio-quenchant oils as against the petroleum based mineral oil was associated with the cooling behavior of the quenching oils which led to the rapid dissolution of the fragmented eutectic particles and other alloying elements into a more refined and better re-distribution in the Al-matrix.

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