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Effects of Time-Aged Heat Treatments on the Hardness and Corrosion Resistances of some Sand-Cast Aluminum-Silicon-Iron Alloys

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***Abstract*- The aim behind this research work was to develop a suitable Al-Si-Fe alloy as an alternative material to scrap aluminum used by foundry artisans for casting aluminum cookware in some West African metropolitan areas. Samples from five cast-fabricated 10mm-diameter and 300mm-length Al-Si-Fe alloy rods named A, B, C, D, and E of compositions within 85.4-96.7%Al, 3-13%Si, and 0.3-1.6%Fe were similarly subjected to natural and artificial time-aged heat-treatments. Tests indicated marked improvement in hardness of the heat-treated samples, and their corrosion resistances in 12.1-pH NaOH solution. The hardness and corrosion resistances were greater and increased with the silicon and iron contents relative to the as-fabricated rod samples and pure aluminum. Average maximum hardness value of 370.8HV was obtained with the artificial time-aged heat-treated samples from rod E which contained 85.4%Al, 13%Si, and 1.6%Fe. The least average corrosion weight loss of 0.068g and penetration rate of 0.0594mm/yr were obtained with artificial time-aged heat-treated samples from rod D which contained 87.7%Al, 11%Si, and 1.3%Fe. In totality, rod D with artificial time-aged heat-treated hardness value of 306HV was found to be the best potential raw stock for aluminum cookware out of the five Al-Si-Fe rods in terms of hardness and corrosion resistance.**

***Index Terms*- Development, Aluminum alloys, Alternative, Cookware materials**

I. Introduction

Engineering application of materials for particular purposes depends greatly on their mechanical and chemical and physical properties. Aluminum alloys are used in applications ranging from aluminum foil for food packaging and cans for beverages to structural members in automobiles, aircraft, etc. [1], [2]. The wide application of aluminum alloys stems from their many practicable combinations of engineering properties and the ease which they are produced in varieties and forms [3]. The outstanding properties of aluminum metal are its lightweight and corrosion resistance. Much research works have been done towards improving the properties of aluminum alloys so that they could advantageously replace steel where strength and hardness, lightweight, and corrosion resistance are of importance. Different levels of alloying and/or heat treatments are commonly exploited technologies of enhancing the strength and hardness properties [4].

Heat treatment is a group of industrial and metal working processes used to alter the physical and/or chemical properties of some engineering metals to better levels by controlled heating and cooling of the metals without changing their product shapes [4], [5]. Heat treatment involves heating suitable material to a pre-determined temperature followed by controlled cooling to achieve desired results such as hardness or softness levels of the material [6]. The common heat treatment processes applied to aluminum alloys are homogenizing, annealing, solution heat treatment, and natural and artificial time-aging [7], [8]. Over the years, engineering technology has produced various types of cast and wrought aluminum alloys with vast range of much better properties for various engineering applications than pure aluminum but the utopia has not been reached whereby developed aluminum alloy has unsurpassable strength and hardness and lower cost to steel. In other words, the overall extent to which strength and hardness of aluminum alloys have been improved by technology is still generally much lower in average values compared to values for steel while the costs of most of the alloys for given applications are greater than the cost of conventional steel [7], [8], [9]. Although aluminum metals have better advantages in many applications compared to steel as a stronger and most popular but corrodible structural material, pure aluminum and even most of its alloys have some problems that are encountered in engineering applications [10], [11]:

i. Pure aluminum is much softer with much lower hardness and strength compared to steel as the most popular structural material. In engineering application of metals, adequate hardness is a basic requirement to avoid abrasion and/or corrosion. The low hardness of pure aluminum and some of its alloys is particularly undesirable in applications such as food containers or equipment where it can leach into foods due corrosion or abrasion ions. According to Foods and Drug Administration, frequent intake of aluminum ions due to corrosion or other means into the body above a minimum recommended level of 10mg/Kg is health-hazardous [11].

ii. Aluminum metal does not resist corrosion in all environments. It can corrode appreciably in high chloride, and acidic or alkaline environments with pH outside the range of 4-9 [11].

Artisanal green sand casting of cookware such as pots, and frying pans is a flourishing business in Kaduna and some other metropolitan areas in West and Central Africa. The casting process involves recycling scrap aluminum products by foundry artisans. However, the artisans do not analyze and

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provide information on chemical compositions and hardness of cookware made from these scraps to ensure they meet requirements for food handling containers [12], [13], [14].

This is not a standard engineering practice and can raise suspicion on the food-safety and quality of the cookware and lower their marketability by making many people not interested in buying them [15]. The maximum tolerances laid down for various elements in aluminum alloys used in the food industry by European Food and Feedstuff law EN 602:2007 and other reputable authorities in connection with the use of aluminum metal for food handling are; 13.5%Si, 2%Fe, 0.6%Cu, 4%Mn, 11%Mg, 0.35%Cr, 3%Ni, 0.25%Zn, 0.3%Zi, 0.3%Ti, and Al the balance [10], [11]. Aluminum metal used to make cookware, utensils, foils, and industrial food equipment is alloyed with these elements for strength, hardness, corrosion resistance, durability, and safety requirements for food contact [10], [11], [12]. The main alloy elements that are used to impart hardness and corrosion resistance of aluminum metal used for cookware are however silicon, and iron [12]. That is why Al-Si-Fe alloys are widely used as foil and sheet products for food packaging; and other applications like capacitors, lithographic printing sheets, magnetic alloy for transformers [9], [12], [16]. Al-Si-Fe alloys also have very good casting properties and play important roles in making food industry equipment; building and architecture, high pressure gas cylinder, ladder and access equipment, sporting equipment, road barriers and signs, domestics and office furniture, and lithographic plates [9], [12].

The aim behind this research work was develop an Al-Si-Fe alloy of high hardness and corrosion resistance by cast-fabrication and time-aged heat-treatment as an alternative material to scrap aluminum used by artisans for sand-casting cookware and other applications where desirable and practicable.

II. METHODOLOGY

A. Materials

The materials used for this research were pure aluminum (Al), pure silicon (Si), pure iron (Fe), and 12.1-pH NaOH for corrosion test. The Al and Si used were obtained in rod forms in Kaduna metropolis, in Nigeria; while the Si was also obtained in rod form but from Germany. The metals were procured in dimensions of about 25mm-diameter and lengths up to 2000mm

B. Purity test of the procured metals

The procured Al, Fe, and Si rods were individually analyzed by nominal chemical compositions to know their purity levels. The Japanese-made PDA Shimadzu 7000 spectrometric metal analyzer was used at the Research and Development unit of Defence Industries Corporation of Nigeria (DICON) Kaduna for the analyses. The analyses confirmed that the procured rods were 99.9%-Al, 99.98%-Fe, and 99.95%-Si materials

C. Production of metal powders

Metal powders were produced from the confirmed Al, Fe, and Si rods and used to facilitate melting of Fe and Si which have much higher melting points than Al around the melting point of Al. The powders were produced by the grinding process using hardened steel files. This was done by rigidly holding a given rod in a machinist vise and grinding out bits of metal from it into an appropriately placed underneath steel container. By this process, about 2.1kg of Al powder, and 0.3kg of both Fe and Si powders were produced for the test.

D. Fabrication of Al-Si-Fe alloy rods

Five compositionally different Al-Si-Fe alloy rods were cast-fabricated using the Al, Fe, and Si powders. The fabricated rods were labeled with identification letters A, B, C, D and E according to their weight compositions of Al, Si, and Fe. The study design compositions of the alloy rods were A-(96.7%Al, 3%Si, 0.3%Fe); B-(93.4%Al, 6%Si, 0.6%Fe); C-(90.6%Al, 9%Si, 0.9%Fe); D-(87.7%Al, 11%Si, 1.3%Fe); and E-(85.4%Al, 13%Si, 1.6%Fe).

To produce an Al-Si-Fe alloy rod, the required quantity of each element in the alloy rod were first weight-determined using a Mettler Toledo digital weighing scale with accuracy of up to 0.01g. Plate I shows an instance of weight-determining the required amounts of metal powder for fabrication of a rod alloy.

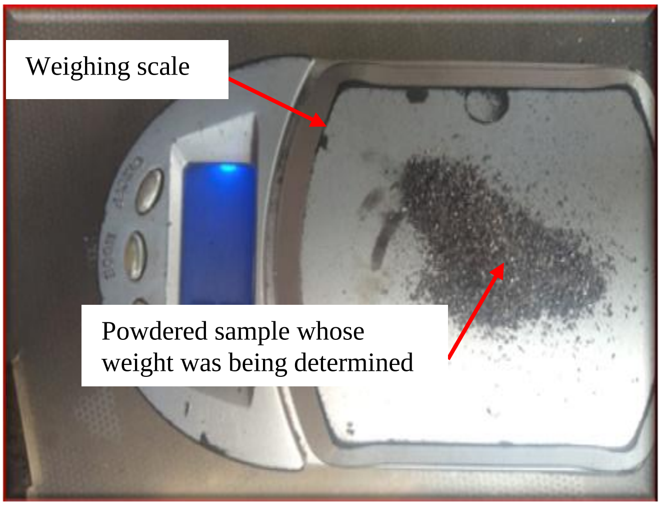

Plate I: Process of weight-determining the required amount of metal powder

After the weight determinations, the Al powder was first heated to molten state in the furnace in a crucible at 700°C just above the melting point of aluminum. This was followed by addition of the Fe powder to the molten Al, and then the Si powder and homogenizing the constituents by thorough mixing with a clean stainless steel rod. After observed satisfactory melting of the Al, Si, and Fe powders and homogenization of the mixture, the molten mixture in the crucible was lifted with a hand-held tong and poured into an already designed and made sand mold with 11mm-diameter and 330mm-length cylindrical cavity. The molten mixture was allowed to solidify in the mold to form the required Al-Si-Fe alloy rod. After proper solidification, the formed alloy rod was extracted from the mold with a hand-held tong. The fabrication process was procedurally repeated to sand-cast all the alloy rods used for the study.

The fabricated alloys rods were trimmed off of surface undesirability on them using polishing facilities to show their true surface appearances. Plate II shows the trimmed and labeled cast-fabricated alloy rods .

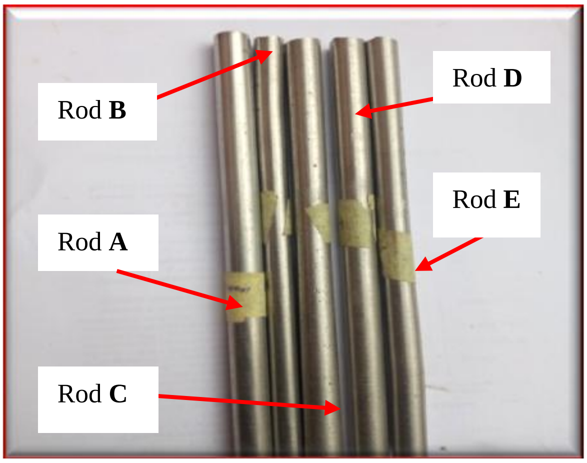

Plate II: The cast-fabricated Al-Si-Fe alloy rods after trimming their surfaces

E. Time-aged heat treatment of samples

Six sample pairs of10mm-diameter and 10mm-length were cut from each of rods A, B, C, D, and E for the hardness test, while six others of dimensions 10mm-diameter and 15mm-length were similarly cut from the same rods for corrosion test. The sample pairs for both hardness and corrosion tests were grouped into three groups with each group consisting of sample pair from each rod. The first group of sample pairs were designated to be un-heat-treated (control samples) while the second group of samples were subjected to natural time-aged heat treatment, and the third group of samples to artificial time-aged heat treatment for both hardness and corrosion testing. Samples were heat-treated by first heating them in the furnace to the temperature of 570^{0}C and soaking them thereat for one hour to obtain solution of their constituents. The samples were thereto removed and quenched in warm water at 75^{0}C to minimize quenching distortion. After the quenching, the group two samples for both the hardness and corrosion tests were allowed to undergo natural time-aging for 5 days; while the group 3 samples were held at room temperature for 18 hours, followed by artificial time-aging at a temperature of160 °C for 12 hours and cooling to room temperature [5]. The heat treatment was carried out using a Vecstan electric furnace with heating capacity of up to 1200^{o}C with accuracy of ± 1^{o}C. Infra-red thermometer was used for determining the process temperatures.

F. Vickers micro-hardness test of samples

Vickers micro-hardness of given coupon was determined in accordance to ASTM E92 and ISO 6507-1:2018 standard test methods for Vickers micro-hardness testing of metallic materials using the LECO M-400 tester with its 40X objective lens of 400X total magnification. The micro-hardness was evaluated using equation 1 [17], [18].

$$HV = 1.854\frac{F}{d^2} \qquad (1)$$

Where F was the applied force in Kgf for which 1 Kgf = 9.81N, and d was the measured average length of the diagonals of the indented base on the coupon in millimetres.

The evaluated Vickers micro-hardness test results were reported as the average pair values for the respective sample pairs of the control, natural time-aged, and artificial time-aged heat-treated samples.

G. Corrosion test of the fabricated alloy rods

All the control and time-aged heat-treated samples for corrosion test were procedurally cleaned to uniform shiny surface finishes in accordance to the ASTM-93 standard procedure for cleaning metallic materials for corrosion tests. The cleaned coupons were separately weight-determined using a Mttler Toledo digital weighing balance and their respective average pair weight values determined in grams and recoded as w_1. The prepared coupons were then soaked in NaOH solution to simulate effects of alkaline environmental corrosivity on the coupons. The NaOH solution was prepared by diluting analytical grade NaOH with distilled water until a 12.1-pH solution was obtained. The coupons were left in the 30.9^{o}C-NaOH solution in glass beakers at ambient laboratory temperature for 154.58 days (3710 hours) to undergo any possible level of corrosion. At the end of the soaking duration, the coupons were removed with hand-held tongs and procedurally cleaned-off of any surface adherents on them in accordance to the ASTM-93 standard procedure. The cleaned coupons were each again weight-determined in grams and recorded as w_2. The pH and temperature of the prepared NaOH solution were determined with an infrared thermometer and pH meter respectively. Plate III shows an instance of monitoring the pH of the prepared NaOH solution using a pH meter, while Plate IV shows coupons from the un-heat-treated (control), natural time-aged heat-treated, and artificial time-aged heat-treated under immersion in NaOH solution in three beakers. In each case identification marks were made on the samples to indicate the cast-fabricated aluminum alloy rods they were produced from.

The test was conducted in a chemistry laboratory at the Department of Chemistry Nigerian Defence Academy using NaOH and facilities available there.

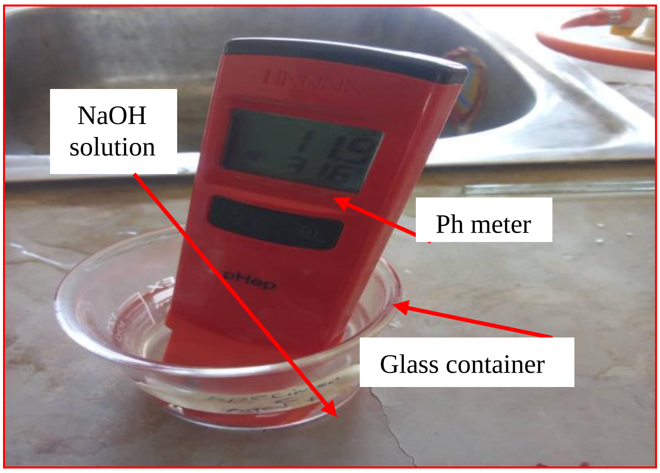

Plate III: Process of determining the Ph of the prepared NaOH solution in the laboratory using a Ph meter

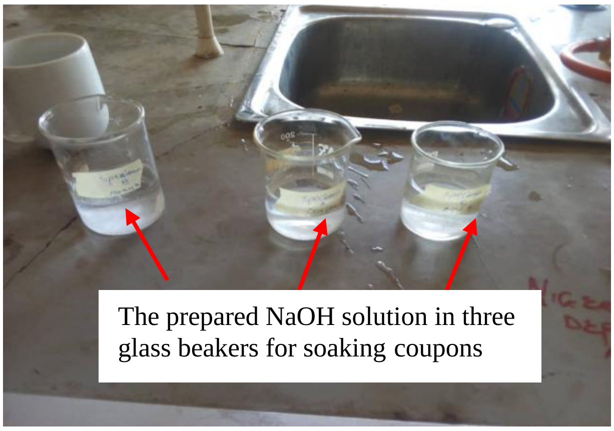

Plate IV: Coupons from groups A, B, and C under immersion in NaOH solution in glass beakers

The corrosion penetration rate of the specimen (CPR) of the coupons was determined in accordance to equation 2, given as [20];

$$CPR = \frac{87.6w}{\rho AT} \qquad (2)$$

Where w was the average pair weight loss of respective coupons in the medium in milligrams, ρ was the determined average density of the coupons, A was the surface area of the cylindrical coupons before immersing them in the test medium, and T was the time in hours (3710 hours) the coupons were immersed in the medium [20].

The weight loss w was determined according to equation 3, given as;

$$w = w_1 - w_2 \qquad (3)$$

The area (A) of the cylindrical coupons was determined according to equation 4, given as;

$$A = \pi\frac{d^2}{2} + 2\pi dh \qquad (4)$$

Where; d was the average diameter of each coupon for 3 measured diameter values at different locations with a venire caliper, and h was the average length of the coupons for 3 measured length values at different locations with a venire caliper.

The determined individual surface areas of the 30 study coupons were averaged and a value of $10.15cm^2$ was obtained.

The average relative density (ρ) of the fabricated rods was determined in accordance with the ASTM D70-17 procedure for determining relative density of solid materials. A 250-milllitre glass container of internal diameter 75mm, an electronic digital weighing scale, a venire depth gauge, and a small piece of the fabricated rods that could fit in the container were used for determining the relative densities of the fabricated rods. By procedure, mass of the requisite rod piece (M) was determined with the digital weighing. The piece was immersed in fresh water poured to a depth level and volume (V_1) in the 250-milliliter glass container and the new volume (V_2) noted. The relative density (ρ) of the piece was determined according to equation 5, given as;

$$\rho = \frac{M}{V_2 - V_1} \qquad (5)$$

The determined densities of the five fabricated aluminum alloy rods were averaged and 2.68g/cm^3 obtained as the working density of the fabricated aluminum alloy rods. Plate V shows the process of determining the mass and volume of the requisite rod piece in the laboratory. Using the respective value of w, A , T, and ρ; the CPRs were evaluated from equation 2.

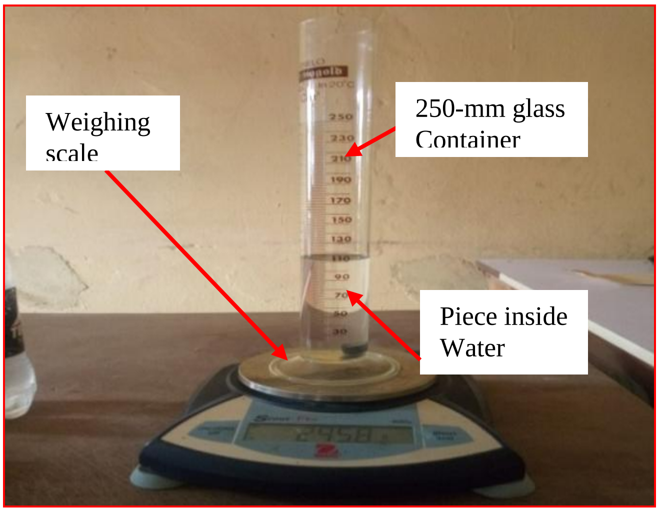

Plate V: Process of determining mass and volume of an alloy rod piece in the laboratory

III. RESULTS AND DISCUSSION

A. Hardness test results

Results of the obtained Vickers micro=hardness of the cast-fabricated Al-Si-Fe alloys is presented in Fig.1.

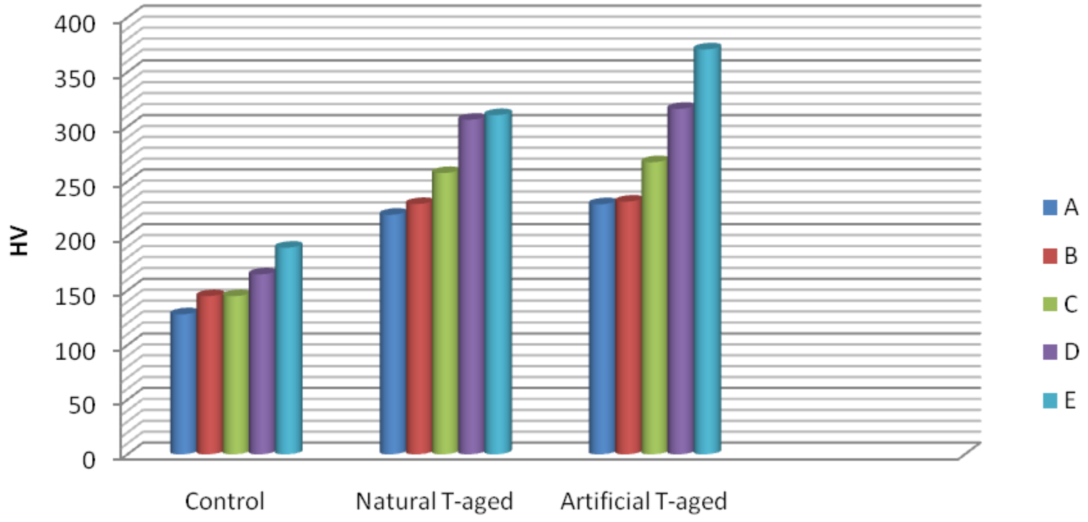

Fig. 1: Vickers hardnes numbers of the control, and natural time-aged and artificial time-aged heat-treated samples produced from rods A, B, C, D, and E

B. Corrosion test results

Results of the test-obtained weight losses (w) and corresponding CPRs of the control, natural time-aged, and artificial time-aged coupons produced from the cast-fabricated Al-Si-Fe rods-A, B, C, D, and E after immersing the coupons in 12.1-pH NaOH solution of 30.9^{o}C for 154.58 days (3710hrs) at average ambient room temperature are presented in Tables 1, 2 and 3 respectively.

Table 1: Weight losses and CPRs of the control coupons produced from the five fabricated rods after 3710-hr soaking duration in 12.1-pH NaOH solution

Coupon pair	w_1 (grams)	w_2 (grams)	$(w = w_1 - w_2)$ grams	CPR (mm/yr)
A	2.700	2.471	0.229	0.2001
B	2.800	2.566	0.234	0.2045
C	2.800	2.571	0.229	0.2001
D	2.000	1.829	0.171	0.1495
E	2.900	2.656	0.244	0.2132

Table 2: Weight losses and CPRs of the natural time-aged heat-treated coupons produced from the five fabricated rods after 3710-hr soaking duration in 12.1-pH NaOH solution

Coupon pair	w_1 (grams)	w_2 (grams)	$(w = w_1 - w_2)$ grams	CPR (mm/yr)
A	2.600	2.382	0.217	0.1897
B	2.900	2.682	0.218	0.1905
C	2.600	2.384	0.216	0.1889
D	2.000	1.832	0.168	0.1468
E	3.200	2.932	0.268	0.2342

Table 3: Weight losses and CPRs of the artificial time-aged heat-treated coupons produced from the five fabricated rods after 3710-hr soaking duration in 12.1-pH NaOH solution

Coupon pair	w_1 (grams)	w_2 (grams)	$(w = w_1 - w_2)$ grams	CPR (mm/yr)
A	2.500	2.417	0.084	0.0734
B	2.600	2.510	0.091	0.0795
C	2.600	2.510	0.091	0.0795
D	2.800	2.733	0.068	0.0594
E	2.900	2.801	0.099	0.0865

C. Discussion of Results

From Fig. 1, it can be observed that the hardness values of the artificial time-aged heat-treated samples are generally greater than the values for the natural-aged heat-treated samples, and the hardness values of the natural-aged heat-treated samples are greater than those for the control (un-heat-treated) samples for the same alloy rod or composition. Generally, the hardness values increase with increase in silicon and iron content of all the samples from rod A which was fabricated with 96.7%Al, 3%Si, and 0.3%Fe contents to samples from rod E with 85.4%Al, 13%Si, and 1.6%Fe contents..The hardness values varied from 128.2HV for the control samples from rod A to 370.8HV for the time-aged heat-treated samples from rod E as can be observed from Fig. 1. Thus, samples from the alloy rod E with 85.4%Al, 13%Si, and 1.6%Fe contents that were heat-treated by artificial time-aging produced the best average hardness value of 370HV out of all the Al alloy rods. This trend of variation in the hardness values could be due to increasing degree of alloying Al with more Si and Fe contents and effectiveness of artificial time-aged heat treatment to stabilize the alloy structures. All the test-obtained hardness values are however greater than the average value for pure aluminum (Al) per se which is round 95HV [10]

Tables 1, 2, and 3 present the corrosion test results in terms of w and the corresponding CPRs. It is observable from the results that the values of w and CPRs generally decrease from the control samples to the natural-aged heat-treated to the artificial-aged heat-treated samples for the same alloy rod. The artificial time-aged heat-treated samples generally exhibited the least weight losses and corrosion penetration rates as can be seen from Tables 1, 2, and 3. The average weight losses and CPRs decrease from respective values 0.229g and 0.2001mm/yr for the control samples produced from rod A to the corresponding least values of 0.068g and 0.0594mm/yr for the artificial time-aged heat-treated samples produced from rod D as can be observed from Tables 1 and 3. This also shows that the average corrosion resistances of the fabricated rods decrease with increase in their silicon and iron contents from rod A with 96.7%Al, 3%Si, and 0.3%Fe contents to rod D with 87.7%Al, 11%Si, and 1.3%Fe contents. This also indicates that rod D which was fabricated with 87.7%Al, 11%Si, and 1.3%Fe alloy composition produced the best average corrosion resistance compared to rod E which was fabricated with alloy content of 85.4%Al, 13%Si, and 1.6%Fe and produced best hardness results. In totality, it is clear that the artificial time-aged heat-treated fabricated Al-Si-Fe alloy rod D can produce optimal practicable hardness and corrosion resistances out of the five cast-fabricated rods.

IV. CONCLUSION

The following conclusions are drawn from the research work:

1. Al, Si, and Fe powders have been successfully used to sand-cast-fabricate five compositionally different Al-Si-Fe alloy rods with 85.4-96.7%Al, 3-13%Si, and 0.3-1.6%Fe contents.
2. Analyzed hardness and corrosion test results of the natural and artificial time-aged heat treated samples from the alloy rods indicate marked improvement in their hardness values, and corrosion resistances in 13.1-pH NaOH solution compared to the un-heat-treated samples from the rods. The hardness and corrosion resistances of the fabricated rod samples also improve appreciably with increase in their silicon and iron contents and heat treatment. Maximum average hardness value of 370.8HV was obtained from the artificial time-aged heat-treated samples from the rod alloy which contained 85.4%Al, 13%Si, and 1.6%Fe. The least corrosion weight loss of 0.068g and penetration rate of 0.0594mm/yr were obtained with samples from the rod alloy which contained 87.7%Al, 11%Si, and 1.3%Fe.
3. In totality, the produced rod alloy with 87.7%Al, 11%Si, and 1.3%Fe contents that was heat-treated by artificial time-aging has been found to be the best potential raw stock for aluminum cookware in terms of hardness and corrosion resistance out of the five cast-fabricated and heat-treated Al-Si-Fe alloy rods.

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