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The Effect of Palm Kernel Shell Ash on the Mechanical and Wear Properties of White Cast Iron

Kayode I. Fesomade*, Damilola D. Alewi, Saliu O. Seidu, Sheriff O. Saka, Bonaventure I. Osuide, Godwin C. Ebidame, Marybeth C. Ugoh, Damilola O. Animasaun

Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Nigeria

ABSTRACT

This study investigates the influence of palm kernel shell ash (PKSA) on mechanical and wear properties of white cast iron (WCI) particularly its influence on its microstructure, elemental composition, hardness and wear resistance. The PKSA was characterized to determine its elemental composition, and it was found to contain high amount of silicon (Si) and iron (Fe) followed by calcium (Ca) and other trace elements. The cast iron was cast into rods of specific dimension with sand casting method using rotary furnace to re-melt cast iron scrap. The WCI rods were then cut into bits for the various test. Heat treatment operation was carried out to determine its properties. Upon completion of the examinations, it was found that the PKSA increased the cementite phase within the matrix of the cast iron, and reduced the pearlitic phase and graphite formation, which gave it increased hardness, and perfect wear resistance due to the increment in carbon content and reduction in silicon content. Also, upon heat treatment, it was found that the PKSA reduced the pearlitic phase within the matrix of the cast iron, increases the formation of transformed ledeburites, austenitic dendrites and tempered graphite, which lead to increased machinability and ductility as well as to reduced hardness, and wear resistance when compared to non-heat treated samples.

Key words: *Microstructure; Heat treatment; Wear Resistance; Hardness; palm kernel shell ash (PKSA)*

1. INTRODUCTION

Researchers are focusing on utilizing white cast iron by modifying its microstructure for enhanced performance in engineering applications. In this light, several works have been done on this white cast iron to improve its quality. Cast iron in words of Razikowza [1] is the cast alloy with carbon content (>2.03%) and some other alloying elements in minute quantities that certifies the solidification of the finishing phase with a eutectic transformation. Depending on chemical stipulations, cast irons can be non-alloyed or alloyed. The range of alloyed irons is much broader, and they comprise either higher volume of common components, such as silicon and manganese, or special additions, such as nickel, chromium, aluminum, tungsten, molybdenum, copper, titanium, vanadium, and others. Free graphite is a usual constituent of non-alloyed and low alloyed cast irons [1]. Applications of white cast iron include gear case, universal joint yoke, rear axle banjo housing, truck tandem axle assembly part, wearing plates, pump liners, mill liners, grinding balls, automotive crankshaft and crankshaft sprocket etc [2-4]. They are most prevalent and common engineering materials characterized with a wide range of mechanical properties of hardness, strength and ductility, wear, machinability, abrasive and corrosion resistance as well as foundry properties. They are tonnage product of the foundry industry. It is said that out of 5 tons of casting product, every 3 tons are that of cast iron [5]. Cast iron is an alloy, which have been used extensively for so many years because of its price and excellent castability. The castability of the alloy is a result of carbon precipitates as graphite during solidification. Due to the precipitate of carbon as graphite, there will be an expansion which counteracts the general shrinkage of the metal during solidification [6]. Since the compositions of most cast irons are about the eutectic point (lowest liquid

* *Corresponding author's.e-mail: kayodefesomade@gmail.com*
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point) of the iron–carbon system, the melting temperatures frequently ranges from 1150 to 1200 °C (2100 to 2190 °F), which is about 300 °C (572 °F) lesser than the melting point of pure iron [7]. The properties of cast iron depend to a great extent on the morphology, amount and anisotropy of the graphite phase. Graphite flakes with random orientation existing in grey cast iron limit its application due to the low tensile strength and low ductility [8].

White cast iron is categorized by its low silicon and increased carbon content with alloy addition, its structure gives a hard brittle iron carbide (Fe_3C) which is called cementite containing no free graphite, which causes that fracture of the material have a white appearance. The white cast iron has been used in a limited way due to its poor properties except in the aspect of wear resistance and hardness, making it comparatively utilized for few engineering applications. The structure of white cast iron has been found to contain pearlite and ledeburite, a eutectic mixture of cementite and pearlite (converted from austenite). The cementite dominates the microstructure of white cast iron, giving it a hard and brittle nature giving it an almost impossibility of machining. White cast iron has fewer tensile strength and it is difficult to machine compared to steel, but its compressive strength is similar to low - and medium - carbon steel. These mechanical properties are controlled by the size and morphology of the cementite present in the microstructure and can be characterized according to the rules given by the America Society for Testing and Materials (ASTM) standard [9] and other standardization organizations.

The large mass of carbide present in white cast iron, especially when alloyed, gives it an excellent resistance against abrasion and wear, making it commonly used for wear resistant surfaces. It also finds application in the following; rolling mills rolls, crushers, slurry pump housings and so on.

The Palm kernel shells (PKS) are obtained after extraction of the palm oil, the nuts are broken and the kernels are removed with the shells mostly left as waste. The PKS have hard stony endocarps which surround the kernel and the shells come in different shapes and sizes. There are two main types of shells; the “Dura” and “Tenera”. The Tenera is a hybrid which has specially been developed to yield high oil content and it has a thin shell thickness compared to Dura type. The shell is made up of 33% charcoal, 45% pyroligneous liquor and 21% combustible gas as reported [10-13].

However, most of the detailed biomaterials properties are scarcely found in literatures. There are several efforts being made towards the utilization of the PKS. Some of the areas where palm kernel shell is used or are being considered for use include: automobile disk brake pad, carbon activation for water purification, concrete ingredient in building industry, thermal insulator and fuel for heat generation [14-17]. The choice of the local product as alloyant is based on the large quantity of oil palm grown in Nigeria. These agricultural wastes (waste biomass) impact negatively on the environment because of indiscriminate disposal of such wastes. Hence utilizing this PKSA to influence the mechanical and wear properties of white cast iron is an alternative method of waste reduction and reuse.

This paper therefore studies the effect of palm kernel shell ash as supplements in influencing the mechanical properties and wear behavior of white cast iron, with respect to the requirements for white cast iron usage.

2. MATERIALS AND METHOD

2.1 Materials

The materials used for this research include the following: (automobile) cast iron scrap, ferrosilicon, palm kernel shell ash (PKSA), black oil, diesel, green sand and bentonite. The charge metal are cast iron scrap (1.8% C), PKSA as an additive for introducing Fe and Si into the melt and ferrosilicon, FeSi (78% Si, 0.21% Al, Fe-balance) as inoculant. The equipment used for this work are the following: furnaces (muffle, crucible, rotary), ball mill and set of sieves, cutting machine, grinding machine, rockwell hardness tester, x-ray florescence, optical microscope, wear testing machine.

2.2 Method

The additive PKSA, was prepared by calcining palm kernel in gas-fired crucible furnace, in order to remove moisture constituents and other volatile materials, and to reduce smoking when heated in the muffle furnace. The PKSA was then taken after calcinations to the muffle furnace for ashing at 900°C and 2 hours soaking period, the samples were allowed to cool and milled for 30 minutes in the ball mill. It was screened with sieve shaker with sieve size 300 microns. The prepared PKSA was stored in air tight container. The pattern was made and mold was prepared using green sand molding technique.

White cast iron was obtained by melting weighted automobile parts scrap in a rotary furnace. The furnace was preheated for 70 minutes for effective melting. The temperature of the melt was measured and monitored at a 25 minutes interval, the rotary furnace was heated to temperature of about 1485°C, and at this temperature the charges had become melted completely and ready for tapping.

The ladle was preheated at same temperature to receive the molten metal, the casting was poured after ladle inoculation. The FeSi alloy inoculant (78% Si, 0.21% Al, Fe-balance) of particle sizes in the range 0.2 to 0.7 mm was added constantly to the metal stream when tapping from rotary furnace to ladle at 1470 °C temperature. Inoculated white cast iron without PKSA was tapped which served as the initial control. The second, third, fourth, fifth and sixth melt tapped were impregnated with 0.25%wt, 0.35%wt, 0.45%wt, 0.55%wt. and 0.65%wt. PKSA, respectively.

The casts were removed after been cooled, fettling was done, and the samples were prepared for chemical analysis and microstructural examination.

2.2.1 *Chemical Analysis, Treatments, Heat Treatment, Microstructural Examination and Mechanical Tests*

Energy Dispersed X-Ray Fluorescence (XRF) was performed for the analysis at EMDI, Akure, Nigeria using Epsilon 1, Spectris, from United Kingdom. The samples of the were cut to a uniform length of 15 mm using the cutting machine and subjected to examination under the XRF to determine the elemental composition of the PKSA sample. The sample was crushed using an agate mortar and pestle, finely ground and sieved with a mesh size of 75 µm. Afterwards, 15–20 g of the samples was placed into a plastic sampleá holder with replaceable plastic support film for each sample analyzed. This plastic support film is attached to the bottom of the sample holder, keeping the sample in the holder and serving as a screen over the X-ray window for direct analysis of the sample. The film also helps to provide a flat surface for the sample as rough surfaces can cause scattering of X-rays. The XRF presented the result of the compositional analysis discussed in next section

Sequel to the casting operation of the five samples with different composition by weight of PKSA and the control, heat treatment was carried out on the samples. This gave rise to heat treated set of samples and non-heat treated set of samples, and both were subjected to the same test, and the variation in result were studied and discussed in the next section.

The samples (12 mm diameter, 200 mm length) were subjected to increased heating in a muffle furnace to 900 °C (AC_3 temperature) and held for 1 hour. This makes it sufficient for some of the cementite to go into solution in the austenite, and to secure partial or complete diffusion and equalization in the austenite, forming a homogeneous matrix of substantially saturated austenite. After the holding time has elapsed, the sample is allowed to cool in furnace to 450°C and held for another 1 hour to equalize the temperature of the white cast iron body, before quenching in oil.

Machined samples of heat-treated and non-heat treated white cast iron with PKSA compositions 0.25%wt.PKSA, 0.35%wt.PKSA, 0.45%wt.PKSA, 0.55%wt.PKSA, 0.65%wt.PKSA and control (0%wt.PKSA) were used for the Optical Microscopic examination (EXI-310 series, Accu-Scope Inc. & Unitron, USA - New York). The rough surfaces of the cut samples were filed to give them good surface finish. The filed surfaces were then grounded using laboratory grinding machine with different sets of emery papers starting from the coarsest to the finest and changing the orientation of the samples during each round of ground. Emery papers of 220, 320, 400, 600, 800 and 1200 grits were used. Polishing was done using laboratory polishing machine to give a mirror-like surface using polishing cloth. Diamond paste of 3 and 1 micron was applied on the polishing cloth and the samples were polished by holding it with less pressure against the rotating disc until a mirror-like surface was obtained. The polished samples were then packed carefully with tissue paper to protect the polished surfaces (especially from rough contact). The samples were examined under scanning electron microscope (JSM 7001F, JEOL, Tokyo,Japan) x600 and x100 micrographs. Hardness and wear tests were carried out on each of the machined heated and un-heated samples of varying PKSA composition using the Rockwell Hardness Tester (FENIX 300RS, Innovatest, Germany) with 60 kgf (0.59 kN) test force and 120° diamond spheroconical indenter, method HRA. Wear test was carried out using the disc on pin type machine, ROTOPOL V product (struers, United states).

3. RESULTS AND DISCUSSION

3.1 *Compositional Analysis*

The result of the compositional analysis for PKSA is shown in Fig. 1, it was observed that this additive was rich in Fe, Si and Ca, with trace amount of other elements. This goes to prove that PKSA will supply more Fe compared to Si to the melt leading to an increase in the formation of iron carbide (Fe_3C) in place of graphite. Also, other elements present in PKSA in trace amount such as calcium (10%wt, chromium (0.06%wt), titanium (0.4%wt), manganese (0.2%wt) and vanadium (0.03%wt) counteracts silicon (28%wt), enhancing the retention of carbon and the formation of carbides.

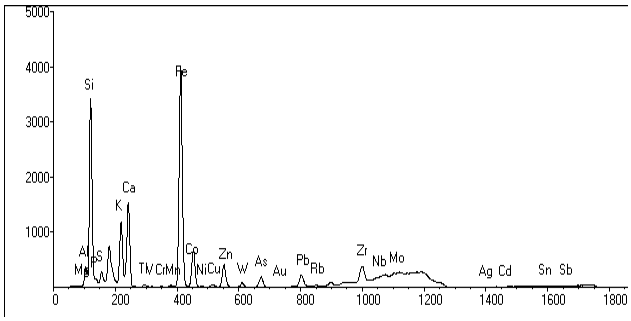

Fig. 1 *Graph of compositional analysis of PKSA*

Table 1 presents the compositional analysis for WCI, The major constituents of WCI are increased Fe and C, accompanied with reduced Si. The reduced silicon prevents the formation of graphite, while the increased C and Fe increases the formation of hard and brittle cementite, which is the primary structure of white cast iron, and to give it its perfect wear resistance and hardness.

Table 1 - *Elemental compositions (%) of white cast iron*

Samples	***C***	***Fe***	***Mn***	***Si***	***P***	***S***	***CE= C+((Si+P)/3)***
Control	3.79	93.3	0.37	1.67	0.21	0.20	4.42
C14	4.01	93.3	0.33	1.51	0.24	0.14	4.59
C15	3.88	93.4	0.30	1.42	0.25	0.18	4.46
C16	4.23	93.0	0.32	1.48	0.24	0.22	4.80
C17	3.56	93.2	0.36	1.64	0.14	0.33	4.15
C18	4.18	93.2	0.30	1.37	0.21	0.17	4.71

The white cast iron produced; C14, C15, C15, C16 and C18 were all hypereutectic with CE values of 4.42%, 4.59%, 4.46%, 4.48% and 4.71% respectively, whereas C17 was hypoeutectic having CE of 4.15%.

3.2 Microstructure of the Developed White Cast Iron Alloys

3.2.1 Non-Heat Treated White Cast Iron

The micrograph of each sample as obtained from the Optical Microscope analysis, depicts the way the cementite phase, graphite flakes and transformed ledeburite are distributed within the pearlite matrix, and it is shown in Fig. 3.

The microstructure of white cast iron can normally be observed as interdendritic cementite (white) and pearlite (dark) or cementite-pearlitic matrix without free graphite [1]. It comprises pearlite and ledeburite, which is a eutectic mixture of pearlite (converted from austenite) and cementite. Cementite being hard and brittle, dominates the microstructure of the white cast iron.

According to Seidu et al. [3], pearlite contributes strength without brittleness to white cast iron which improves machinability. Also, cementite enhances wear resistance but reduces machinability, while ledeburite produces increased hardness and wear resistance, with reduction in machinability. These phases are present in the microstructure of white cast iron as shown in Fig. 3.

The microstructure of the control sample showed pearlitic grains, with traces of cementite towards the right of the micrograph. This reveals reduction in hardness, as the carbon content is lower and silicon content is higher within this sample, which did not favor the formation of cementite and led to more pronounced pearlite matrix.

For samples C14, the formation of graphite flakes, and more cementite in a randomly dispersed pearlitic matrix was evident. This shows that the increment in carbon content and reduction in silicon content caused by the addition of PKSA improved the formation of cementite, and graphite flakes, which will give the sample improved hardness compared to the control. For samples C15-C18, the microstructures revealed a persistent increment in the cementite composition as well as reduction in dispersed orientation of pearlitic matrix, and the formation of graphite flakes from C15 to C18 consecutively. This is the basis for the increment in hardness property of the cast iron, as carbon content increased, forming more cementite, and silicon content reduced, reducing the formation of graphite flakes.

3.2.2 Heat Treated White Cast Iron (WCI)

For the control sample, the microstructure consists completely of pearlite matrix with no trace of cementite, which implies good machinability of the material and reduction in hardness. As percentage composition of PKSA increased, the formation of transformed ledeburite as graphite inclusions and tempered graphite as dark spots within the pearlitic matrix as is observed for sample C14 to C18, as well as the presence of austenitic dendrite. This goes to explain a persistent reduction in hardness compared to the non-heat treated samples. It also explained that the samples will have good machinability due to the formation of austenitic dendrite, in an almost complete pearlitic matrix.

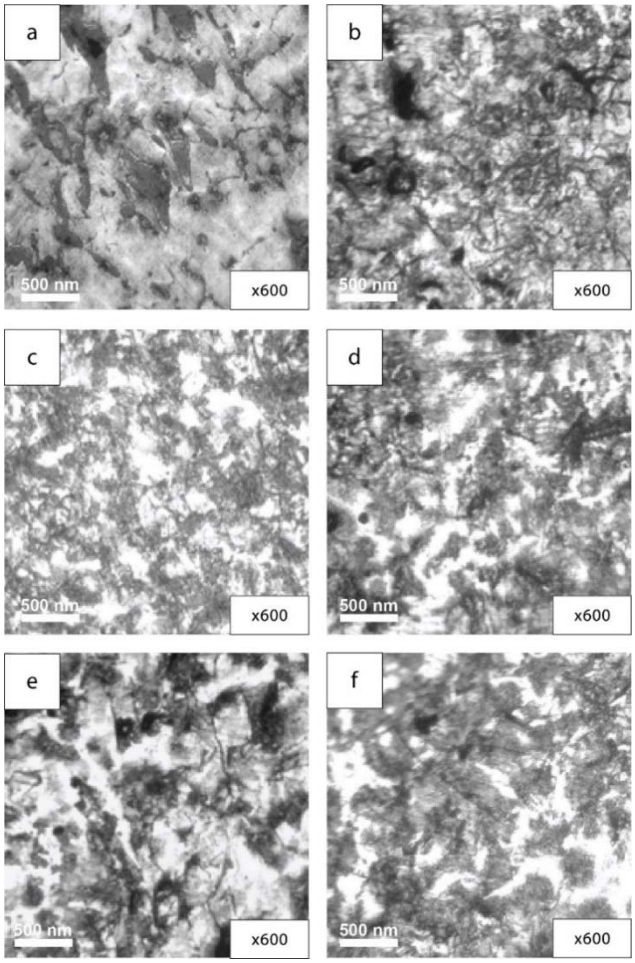

Fig. 3 (a -f) *Microstructures of the Control, C14, C15, C16, C17 and C18 of non-heat treated samples respectively.*

Comparing the microstructures of the heat treated and non-heat treated WCI samples, we can infer that the heat treatment on the samples increased the formation of pearlite and reduced cementite, which gave the material better machinability and helped widen its applications, while the additive in increasing order, increased the formation of ledeburite, tempered graphite and austenitic dendrites within the microstructure of the heat treated samples, allowing for easy machinability and reduced hardness.

3.3 Hardness Analysis

For WCI, the hardness is a function of the carbidic phase such as cementite, because the cementite gives the cast iron its hardness poses resistance to its machinability.

Fig. 5 shows the hardness of non heat treated (N HT) WCI. With respect to the control, the hardness of the material

increased as the percentage composition of PKSA increased all the samples has hardness value higher than that of the control sample except the C17 sample, this anomaly could be as a result of inclusion during casting operation. This goes to show that the PKSA enhanced the formation of cementite within the pearlitic matrix, due to an increment in carbon content, and a reduction in silicon content. Whereas, systematic reduction in the hardness of the samples was achieved with increased PKSA addition in the heat treated (HT) WCI, Fig 6. This is due to the formation of tempered graphite and austenitic dendrites seen in the micrographs in Fig. 3. Also the absence of cementite within the microstructure played a big role in the reduced hardness of the samples, as cementite has been found to increase hardness of WCI.

Comparing both heat treated and non-heat treated samples, it can be deduced that the heat treated samples possessed lower resistance to surface indentation compared to non-heat treated samples with an exception to the control sample due to the absence of PKSA. The reduction in hardness is due to the heat treatment which reduced the formation of cementite and increased pearlite formation and grain growth leading to presence of ductile grains within the matrix.

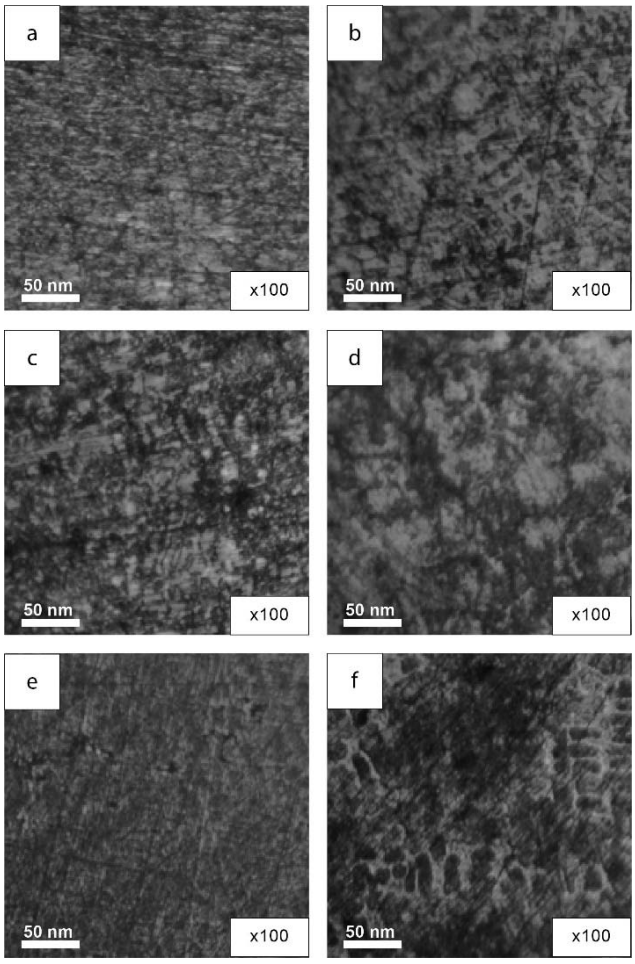

Fig. 4 (a -f) *Microstructures of the Control, C14, C15, C16, C17 and C18 of heat treated samples respectively.*

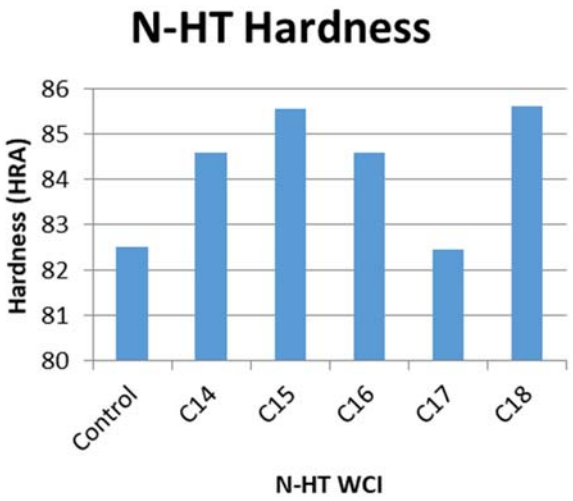

Fig. 5 *Chart showing variation in non-heat treated (N-HT) hardness values.*

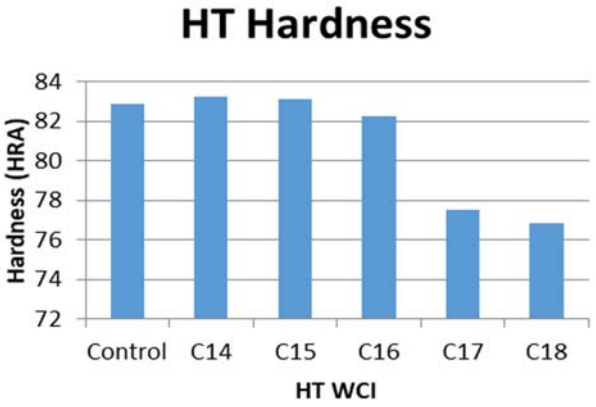

Fig. 6 *Variations in heat treated (HT) hardness values*

3.4 Wear Analysis

From Fig. 7, it can be seen an increased wear rate value for the heat treated control sample compared to the Non-heat treated control, and a progressive reduction in wear rate as increased % composition of PKSA was being added. The increased wear rate value for the HT control sample is as a result of complete absence of cementite phase and transformed ledeburite within the pearlitic matrix, because presence of cementite or transformed ledeburite has been found to decrease wear rate of WCI and increased the hardness.

Also, comparing both samples, we can infer that the N-HT samples possessed better wear rate compared to the HT samples.

From the wear resistance chart in Fig 8, it can be observed a better wear resistance for the N-HT samples compared to the HT samples. This is due to the formation of cementite within the pearlitic matrix of the N-HT samples, and the absence of cementite within the pearlitic matrix of the HT samples. The minimal increase in wear resistance observed for the HT samples is as a result of the formation of

transformed ledeburites as increased % composition of PKSA was being added. Also, the formation of austenitic dendrite in compositions C18 together with the presence of tempered graphite and transformed ledeburite gave it an increased wear resistance compared to other compositions for both HT and N-HT samples.

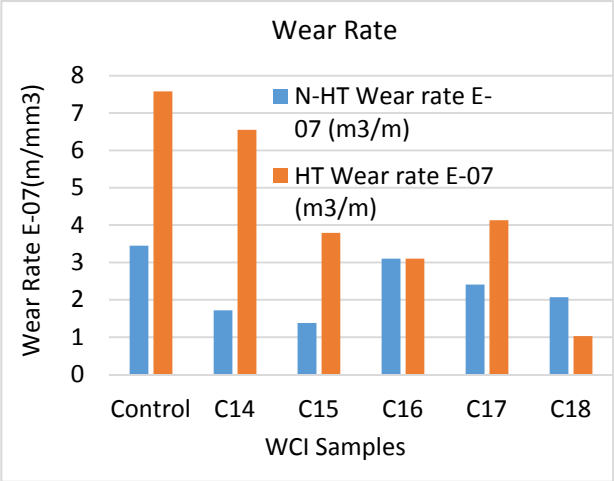

Fig.7 *Chart showing Wear rate comparison for N-HT and HT samples*

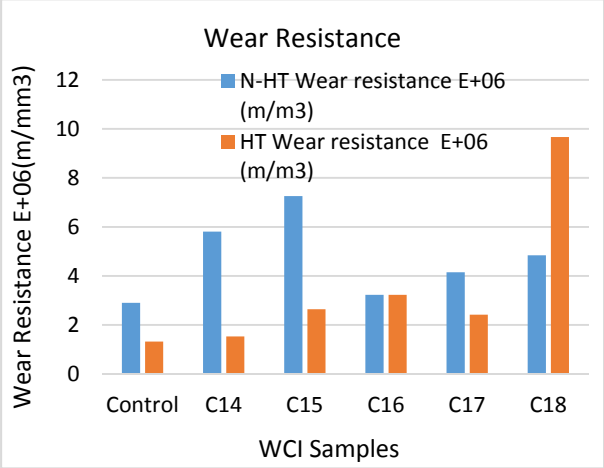

Fig. 8 *Chart showing Wear resistance comparison for N-HT and HT samples*

4. CONCLUSION

The following conclusions are drawn empirically from the result of this study.

The PKSA having been characterized to possess more Fe than Si and Ca. The presence of alloying elements such as Mo, V, Ni, S, and P, has been found to enhance the formation of cementite than graphite by the combination of Fe and C within the matrix.

The micro-structure of the non-heat treated (N-HT) samples showed an increase in cementite phase as well as a reduction in graphite flakes formed when the PKSA was being added, due to increase in carbon content and reduction in silicon content. This showed that an increase in the PKSA addition reduced the hardness of the samples and reduced its machinability.

Moreover, the micro-structure of the heat treated (HT) samples showed an increase in the formation of transformed ledeburite, tempered graphite and austenitic dendrites which reduced the hardness of the samples, as cementite formation has been hindered due to the heat treatment carried out. This showed that an increase in the PKSA reduced the hardness of the samples and increased its machinability.

The hardness of the WCI can be improved by the introduction of the PKSA. This is due to the fact that it contributes to the formation of cementite rather than pearlite in the matrix of the cast iron. Although for the heat treated (HT) samples, the hardness decreased as increased % composition of the PKSA was added, due to the formation of tempered graphite, austenitic dendrites and transformed ledeburites.

Finally, the wear resistance and wear rate of the WCI can be influenced by the addition of the PKSA, as a significant increase in wear resistance was observed, as well as a significant decrease in wear rate for both N-HT samples and HT samples.

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