

**THERMOCHEMICAL SOLID CEMENTATION TREATMENT USING RICE HUSK
ASH AND CHARCOAL ON SAE 1020 STEEL**

**TRATAMENTO TERMOQUÍMICO DE CEMENTAÇÃO SÓLIDA USANDO CINZA
DE CASCA DE ARROZ E CARVÃO VEGETAL NO AÇO SAE 1020**

**TRATAMIENTO TERMOQUÍMICO DE CEMENTACIÓN SÓLIDA MEDIANTE
CENIZA DE CASCARILLA DE ARROZ Y CARBÓN VEGETAL SOBRE ACERO
SAE 1020.**

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Abstract: Rice husk ash (RHA) originates from rice husks burning for energy generation, where it is often disposed of inappropriately, causing negative environmental impacts. This is abundant and low-cost, so it could use to new uses. With it in mind, this research aimed the comparative study between effects of carbon source from charcoal and RHA. After carburization at 900°C for 8h and 15h and 950°C for 15h. Next, the samples were undergoing heat treatment by quenching in water and tempering at 180°C. The samples were analysed by optical microscope, microhardness test, X-ray diffractogram and wear test, showing that the heat treatment procedures were performed correctly, obtaining higher surface hardness values. Likewise, optical microscope and hardness analysis, show a surface layer enriched with carbon resulting from cementation. The images show the poor formation of martensite in the microstructure of the samples with heat treatment and tempering; there was a significant increase in surface wear resistance after the procedures performed with RHA powder and charcoal. In general, the results of microhardness and wear resistance in the samples cemented with charcoal were higher.

Key words: Rice husk ash, Cementation, environmental impacts reducing, 1020 steel, thermochemical treatment.

Resumo: As cinzas de casca de arroz (CCA) são originárias da queima de cascas de arroz para geração de energia, onde muitas vezes são descartadas de forma inadequada, causando impactos ambientais negativos. Por ser abundante e de baixo custo, poderia ser utilizado em novos usos. Com isso em mente, esta pesquisa teve como objetivo o estudo comparativo entre os efeitos da fonte de carbono do carvão vegetal e da RHA. Em seguida, as amostras foram submetidas a tratamento térmico por resfriamento em água e revenimento a 180°C. As amostras foram analisadas por

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microscópio óptico, teste de micro dureza, difratograma de raios X e teste de desgaste, mostrando que os procedimentos de tratamento térmico foram realizados corretamente, obtendo valores mais altos de dureza superficial. Da mesma forma, a microscopia ótica e a análise de dureza mostram camadas superficiais enriquecidas com carbono resultante da cementação. Da mesma forma, as imagens mostram a má formação de martensita na microestrutura das amostras com tratamento térmico e revenimento; porém, houve um aumento significativo da resistência ao desgaste superficial após os procedimentos realizados com pó de CCA e carvão vegetal. Em geral, os resultados de micro dureza e resistência ao desgaste nas amostras cimentadas com carvão vegetal foram maiores.

Palavras-chave: Cinza de casca de arroz, cementação, redução de impactos ambientais, aço 1020, tratamento termoquímico.

Resumen: Las cenizas de cáscara de arroz (CCA) se originan a partir de la combustión de cascarillas de arroz para la generación de energía, y con frecuencia son descartadas de manera inadecuada, causando impactos ambientales negativos. Debido a su abundancia y bajo costo, podrían ser utilizadas en nuevas aplicaciones. Con ello en mente, esta investigación tuvo como objetivo realizar un estudio comparativo entre los efectos de la fuente de carbono proveniente del carbón vegetal y de la CCA. Posteriormente, las muestras fueron sometidas a tratamiento térmico mediante enfriamiento en agua y revenido a 180 °C. Las muestras fueron analizadas mediante microscopía óptica, ensayo de microdureza, difracción de rayos X y ensayo de desgaste, evidenciando que los procedimientos de tratamiento térmico se realizaron correctamente, obteniéndose valores más altos de dureza superficial. Asimismo, la microscopía óptica y el análisis de dureza muestran capas superficiales enriquecidas con carbono como resultado del proceso de cementación. De igual manera, las imágenes revelan una formación deficiente de martensita en la microestructura de las muestras sometidas a tratamiento térmico y revenido; sin embargo, se observó un aumento significativo en la resistencia al desgaste superficial después de los procedimientos realizados con polvo de CCA y carbón vegetal. En general, los resultados de microdureza y resistencia al desgaste en las muestras cementadas con carbón vegetal fueron superiores.

Palabras clave: ceniza de cáscara de arroz; cementación; reducción de impactos ambientales; acero 1020; tratamiento termoquímico.

1 INTRODUCTION

Nowadays, there is an increasing search for sustainable alternatives in industries. As such, this area has received much of its research focused on systems and methods that aim at sustainability and the reuse of waste that was previously not properly disposed of (Gonçalves & Bergmann, 2007; Prasad & Pandey, 2012; Islabão et al., 2014). In this context, rice husks produce a significant amount of waste and, consequently, ash when burned in boilers for power generation in rice processing plants. The ash produced in the boilers is normally dark in colour and has a high carbon content, which contains some crystalline silica (Cordeiro et al., 2009).

In solid carburizing or box carburizing, the parts are placed inside a box made of steel together with a mixture of carbon-rich carburizing powder. The carburizing boxes are placed inside the furnace at temperatures between 850 and 1050°C. At

this temperature, the oxygen present in the air combines with the carbon in the carburizing box, producing carbon dioxide ($C + O_2 \rightleftharpoons CO_2$). The carbon dioxide combines with the carbon in the carburizing medium, generating carbon monoxide ($CO_2 + C \rightleftharpoons 2CO$), however, it will adapt to the iron in the steel, generating more CO_2 ($3Fe + 2CO \rightleftharpoons Fe_3C + CO_2$). At this temperature, the carbon in the carburizing medium diffuses to the surface of the part. This technique basically increases the carbon content in a thin layer on the surface of the part, that is, if the steel is low carbon, only the surface layer is changed with the addition of this element, maintaining the initial characteristics in the core (Foreman, 1991).

In steels that undergo thermochemical carburizing treatment, heat treatments of quenching and tempering are used. Thus, resulting in high surface hardness and, consequently, greater resistance to wear. A hard surface with a more tenacious core is widely used, especially because, by using steels with alloy elements, it is possible to obtain a core of high resistance and tenacity. With an extremely hard surface, results in a material capable of withstanding certain types of stress. Gears, for example, have a tenacious core and a wear-resistant surface (Chiaverini, 2012).

According Astunkar and Bonde (2013), to increase the hardness of steels, the most common and usual process is to use the thermochemical process of cementation. This process modifies the surface of the material, making it more resistant to wear. As it is an economical method, it is widely used in industries as a surface hardening method. It is used for steel with low carbon content with a percentage of this chemical element of up to 0.20 or less. In this way, the study of methods and techniques to increase wear resistance in mechanical components is increasingly addressed in scientific research. Linked to this increase in resistance, a good cost-benefit ratio is also sought for heat treatment processes, studying and improving technologies, procedures, and materials for their implementation.

In this context, the proposed work addresses the resistance to abrasive wear and techniques to avoid it, or reduce its incidence. For this, the thermochemical treatment of carburization was carried out using two different carbon sources, charcoal and rice husk ash (RHA), with variations in temperatures and treatment times in SAE 1020 steel; followed by quenching and tempering heat treatment.

To this end, the solid-state diffusion mechanism occurs to mass transport within a material through atomic movement by stages. Where diffusion under non-steady-state conditions means that the diffusion flux and concentration gradient at a specific point in the solid vary with time, resulting in the accumulation or depletion of the component that is diffusing (Callister Jr., & Rethwisch, 2012). In addition, carbon diffusion, from surface to interior causes a carbon concentration gradient varying by distance and time; according to Equation (1) Fick's second Law.

$$\frac{\partial C}{\partial t} = \frac{\partial}{\partial x} \left(D \left(\frac{\partial C}{\partial x} \right) \right) \quad (1)$$

Where x (mm) - distance from the part surface/centre, t (hours) - treatment time, D - diffusion coefficient (T^{-1}) of a chemical element in the austenite and C - carbon concentration at a distance x (% by weight). Since the diffusion coefficient is independent of the composition, Equation 1 can be simplified by Equation (2):

$$\frac{\partial C}{\partial t} = D \left(\frac{\partial^2 C}{\partial x^2} \right) \quad (2)$$

Likewise, the diffusion coefficient of a chemical element in austenite can be calculated by the following Equation (3) (Callister Jr.; & Rethwisch, 2012).

$$D = D_0 \exp - \frac{Q}{RT} \quad (3)$$

Where D_0 - constant (T^{-1}); Q - activation energy for diffusion (J); R - universal gas constant (J/kg.K) and T - absolute temperature (K). With this, the solution of Equation (2) for carburizing in steels is equal to Equation (4):

$$\frac{C_c - C_s}{C_0 - C_s} = \text{erf} \left(\frac{x}{2Dt^{1/2}} \right) \quad (4)$$

Where C_c - carbon concentration at a distance x from the surface after a time t of carburizing treatment (% by weight); C_s - carbon concentration on the surface maintained by the carbon potential of the atmosphere (% by weight); C_0 - initial carbon concentration in the Fe-C alloy (% by weight); D - carbon diffusion coefficient in austenite (T^{-1}) and erf - Gauss error function (T^{-1}).

Lampman (1991) highlights that for thermochemical carburizing treatment is mainly used in low-carbon steels, with the aim of adding this element to the surface of the steels. The absorption of carbon into the material occurs through the phenomenon of diffusion at temperatures generally between 850 and 950°C, creating a carbon concentration gradient between the surface and the interior of the component. This process aims to improve the hardenability on the surface of the material, with the addition of carbon, while maintaining the original characteristics in the core of the part. Além disso, Baumgarten (2003), completam que para qualquer operação de saturação de uma superfície por difusão deve ser executada em um meio que possa liberar o elemento a ser difundido em um estado atômico. Para a cementação, o elemento em questão é o carbono. Deve haver contato entre os átomos que serão difundidos e a superfície que será tratada, o que formará ligações químicas com os átomos da matriz metálica. Então, haverá a difusão propriamente dita, que é a penetração do soluto na matriz metálica. O objetivo principal da cementação é proporcionar um aumento de dureza e resistência ao desgaste superficial, aliada à boa tenacidade no núcleo da peça (Silva, 2010).

Notwithstanding, Chiaverini (2008) points out that the cementation process applied in alone-way doesn't bring high surface hardness to steel. Where this increase is obtained through a heat treatment known as quenching, which is carried out after cementation; in this way, the surface layer will present a greater hardness than the core. With these procedures, residual compressive stresses are also produced on the surface, which normally increases the fatigue life of the component. It is necessary that the steel, in contact with the environment, be heated to a temperature that allows the carbon solution in the iron to be easily dissolved. For this, the temperature must be above the critical zone (850° to 950°C), that is, where the iron will be in the allotropic gamma form. In addition to the time and atmosphere in which the cementation is carried out, there is another variable that directly contributes to obtaining good results in the process, which is the carbon potential of the cementing medium. This potential is the maximum amount of carbon content that can be added to a given steel. Carbon potential in heat treatment refers to the capacity of a furnace atmosphere to either add or remove carbon from the surface of steel during heat treatment. It essentially defines the carbon activity, or the "tendency to donate or accept carbon".

Based on this horizon explained here, the use of RHA represents a viable solution to promote the circular economy and reduce the environmental impacts of agriculture and industry that produce this “waste” material without knowing where to dispose of it. With investment in research and development, RHA can become a fundamental ally on the path to a more sustainable future and eliminate this waste from companies.

2 EXPERIMENTAL PROCEDURES

2.1 MATERIALS

The material used in this study is SAE 1020 steel. The material was purchased in the form of cylindrical bars with 1/2” (12.7 mm) in diameter. Table 1 shows the chemical composition of the steel. The Figure 1, in other hand, show an analysis of the microstructure of SAE 1020 carbon steel. A small amount of carbon can be observed. The steel under study has a low carbon value and is used for cementation treatment, thus achieving good ductility and preventing brittle rupture.

Table 1 - Chemical composition steel SAE 1020.

| Elements | C | Mn | P | S |
|----------|-------------|------------|-------------|-------------|
| % | 0.23 | 0.6 | 0.04 | 0.05 |

Source: Authors.

Figure 1 - Optical microscope 200x magnification – SAE 1020 carbon steel without heat treatment.



Source: Authors.

The RHA was collected directly from the storage silos of the Alegrete Agroindustrial Cooperative (CAAL). It has a dark colour due to the amount of carbon present in its chemical composition, as Figure 2(a) shown. Thermochemical treatments were performed with two different carbon sources, charcoal and RHA. The ash was obtained from Cooperativa Agroindustrial Alegrete Ltda. (CAAL), after burning the rice husk used as fuel in the thermoelectric plant. This ash is fine-grained and dark in colour. This material is garbage for the company, material for solid cementation that has no cost. To prepare charcoal, the charcoal was ground - Figure

2(b), followed by sieving through a 1.2 mm mesh - Figure 2(c), resulting in a fine-grained powder - Figure 2(d). The proportions for the cementation treatment with ash were 60g of RHA and 10g of sodium carbonate. For cementation with charcoal, 140g of charcoal and 25g of sodium carbonate were used.

Figure 2 - Cementation mixtures of charcoal and RHA.



Source: Authors.

2.2 THERMOCHEMICAL CARBURIZING, QUENCHING AND TEMPERING

The thermochemical cementation treatments were performed in an INTI muffle furnace. Table 2 shows the cementation process, which was separated into 3 conditions. The thermochemical treatments were performed with two different carbon sources, charcoal and RHA. The cementation mixtures were prepared with activators to enhance the formation of CO_2 during cementation. The activator used in this work was sodium carbonate (Na_2CO_3), representing 15% of the cementation mixtures, allowing Hafni et al. (2017) parameters.

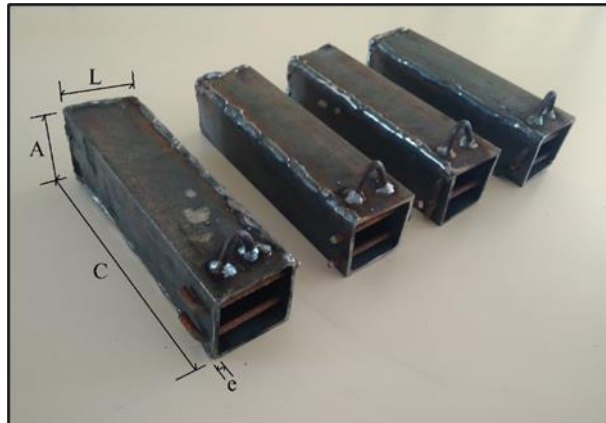
Table 2 - Thermochemical carburizing treatment parameters with 6 conditions.

| Condition | Cementing | Time (hours) | Temperature (°C) |
|-----------|-----------------|--------------|------------------|
| 1 | Charcoal | | |
| 2 | RHA | 8 | |
| 3 | Charcoal | | 900 |
| 4 | RHA | | 900 |
| 5 | Charcoal | 15 | |
| 6 | RHA | | 950 |

Source: Authors.

In complementary way, Figure 3 shows the cementation boxes for the treatment. The experiment was carried out according Aramide et al. (2010) procedures, where the sample were placed into a steel box containing the powder mixture. The steel case has been hermetically sealed with clay to prevent unwanted gas from the oven from accessing it during heating, so the carburizer is no burn. Next, the steel box is then removed from the furnace, the specimens were taken from the steel box and cooled in the water. Sequentially, the quenching process was carried out after austenitization, at temperatures of 900° and 950° C, with a soaking time (temperature homogenization) of 30 minutes. Afterwards, cooling occurred in water with slight agitation, tempering occurred at a temperature of 180° C.

Figure 3 - Cementing boxes.



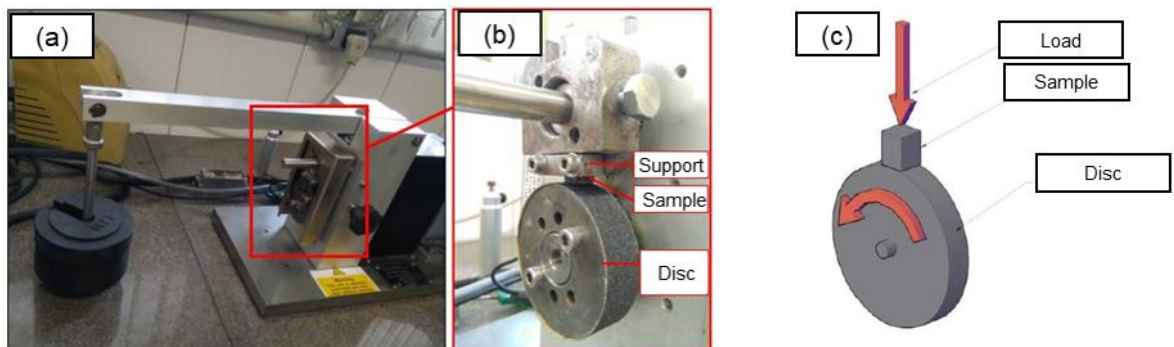
Source: Authors (2024).

2.3 ANALYSIS

The samples were cut, embedded, sanded (gran: 280, 400, 600 and 1000) and polished with diamond paste. The chemical attack for revealing the microstructure was Nital 5%. For the acquisition of micrographs and XRD, the optical microscope and diffractogram was used. The micro hardness test was performed with embedded, polished and sanded samples. The microhardness profile was traced along the entire diameter of the sample with 17 indentations, with the aim of better identifying the layer with the greatest hardness. The load used in the tests was 0.5 kg applied for 10 seconds. The ABNT NBR NM 6507 (Associação Brasileira de Normas Técnicas [ABNT], 2008) standard was used to perform the tests. In addition, another important parameter of carburizing product, was used as effective case-hardened depth (CHD), under EN ISO 2639 standard.

The abrasive wear test, on the other hand, was performed using a tribology equipment coupled to a computer for programming and control. The objective of the test is to identify the improvement in the material's wear resistance with heat treatments. The equipment works with a “block on ring” principle, where the surface of the sample is rubbed against a disc covered with abrasive sandpaper, which is in rotation, while the sample is fixed to the support. Figure 4(a) illustrates the equipment used, where it is possible to see the load discs responsible for applying the force to the sample, and in (b) the system where the sample is fixed to the support, positioned on the abrasive disc (c), according Fantineli's (2015) parameters (Table 3).

Figure 4 – Wear test equipment, UNIPAMPA.



Source: Authors.

Table 3 - Parameters for abrasive wear test.

| Wear Test Parameters | |
|--|------|
| Speed (RPM) | 50 |
| Force (N) | 189 |
| Applied load (kg) | 3 |
| Number of cycles | 300 |
| Disc of diameter (mm) | 60 |
| Sandpaper grain size (Grain/pol ²) | 120 |
| Length of test piece (mm) | 12.7 |

Source: Authors.

To determine the surface wear of each sample after the test, Equation (5) was used. Where wear is quantified by the percentage mass loss in relation to the initial by: Ml - Mass loss (%); I_m - Initial mass (g); F_m - Final mass after the test (g).

$$Ml = \frac{I_m - F_m}{I_m} \times 100 \quad (5)$$

3 RESULTS AND DISCUSSIONS

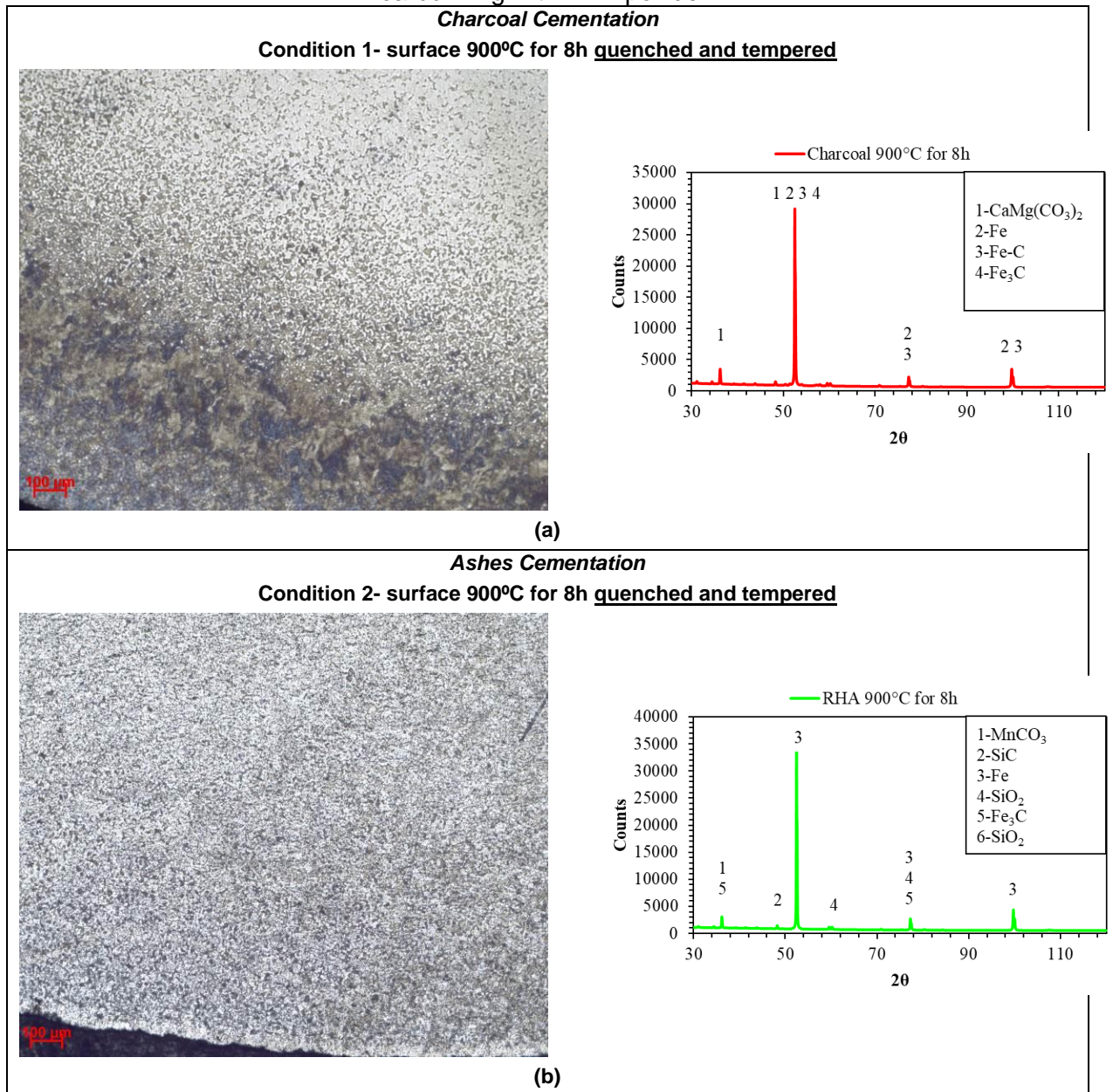
3.1 MICROSTRUCTURE ANALYSIS

After carburizing followed by quenching, tempering the surface layer of the steel can transform into martensite. This occurs because steel with a higher carbon content has a greater hardening capacity and, when rapidly cooled, forms very hard and wear-resistant martensite; since, the carburizing process is superficial, the steel core generally does not receive enough carbon to modify its microstructure. Therefore, the core remains with a more tenacious and less hard microstructure, composed by ferrite/pearlite, which low carbon (Colpaert, 1974; 2018).

In this context, cementation performed with charcoal cementing agent presents a coarser grain size compared to cementing with RHA. Rice husk ash, in other hand, is rich in silica (SiO_2). With this, during cementation, small inclusions of silica occur in the steel, in small quantities. In some cases, silica can contribute to increasing wear resistance, as it acts as a hard phase on the surface. The presence of silica allows the grains refining, making finer microstructure (Cordeiro et al., 2009).

In this way, Figure 5 shows the treatment with the two types of cementing agents at a temperature of 900°C for 8 hours. An increase in carbon content and martensitic grain size and retained austenite on the surface can be seen, Figure 5(a). In Figure 5(b), shown grains of amorphous silicon dioxide that react with metal oxides to form different silicates. As result, during the diffusion of carbon on the surface, there was formation of iron carbide particles (cementite) for both cementing agents along the cemented layer, which contributes to the increase in hardness. In the graph in Figure 5(a), the X-ray diffraction shows an increase in iron carbides (cementite) on the surface of the sample, causing its surface microhardness to increase in relation to its own core. The graph in Figure 5(b) shows the formation of silicon carbide, also increasing hardness. As the cemented alloy contains manganese and the medium contains sources of carbon and oxygen (CO or CO_2), under certain temperature conditions a reaction occurred forming MnCO_3 on the surface.

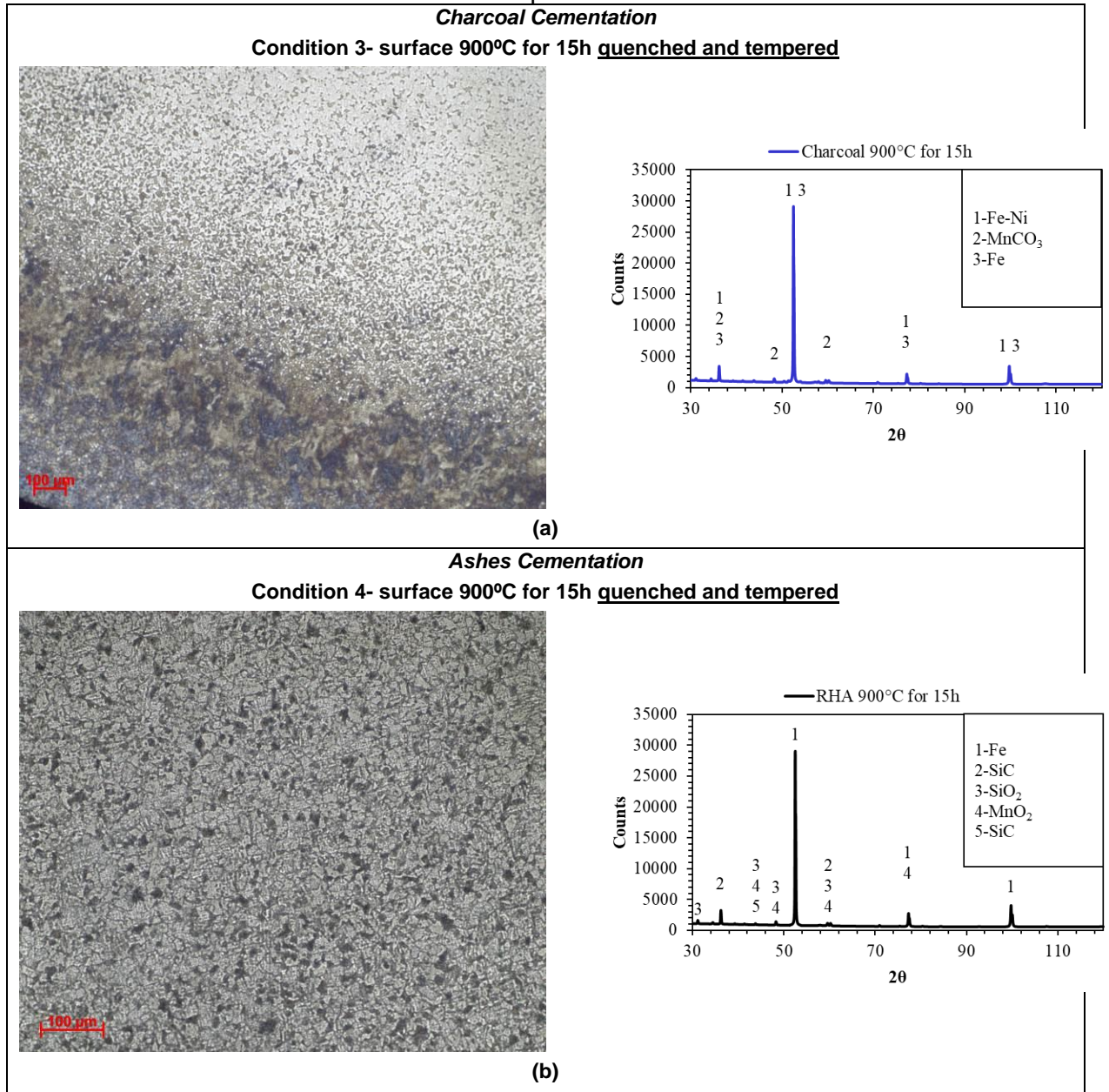
Figure 5 – Optical microscope of microstructure near the surface of sample edge. Carburizing at a temperature of 900°C for 8h, (a) carburizing with charcoal powder (b) carburizing with RHA powder.



Source: Authors.

The carburizing temperature at 900°C, by yourself, with a longer time in the furnace of 15h, shows a structure on the martensite surface; according to Figure 6(a). In the x-ray diffraction graph the analysis showed the formation of iron carbide, consequently increasing the mechanical resistance. Figure 6(b) presents the microstructure with more homogeneous grains and the x-ray analysis graph shows the formation of silicon carbide, contributing to an increase in surface hardness.

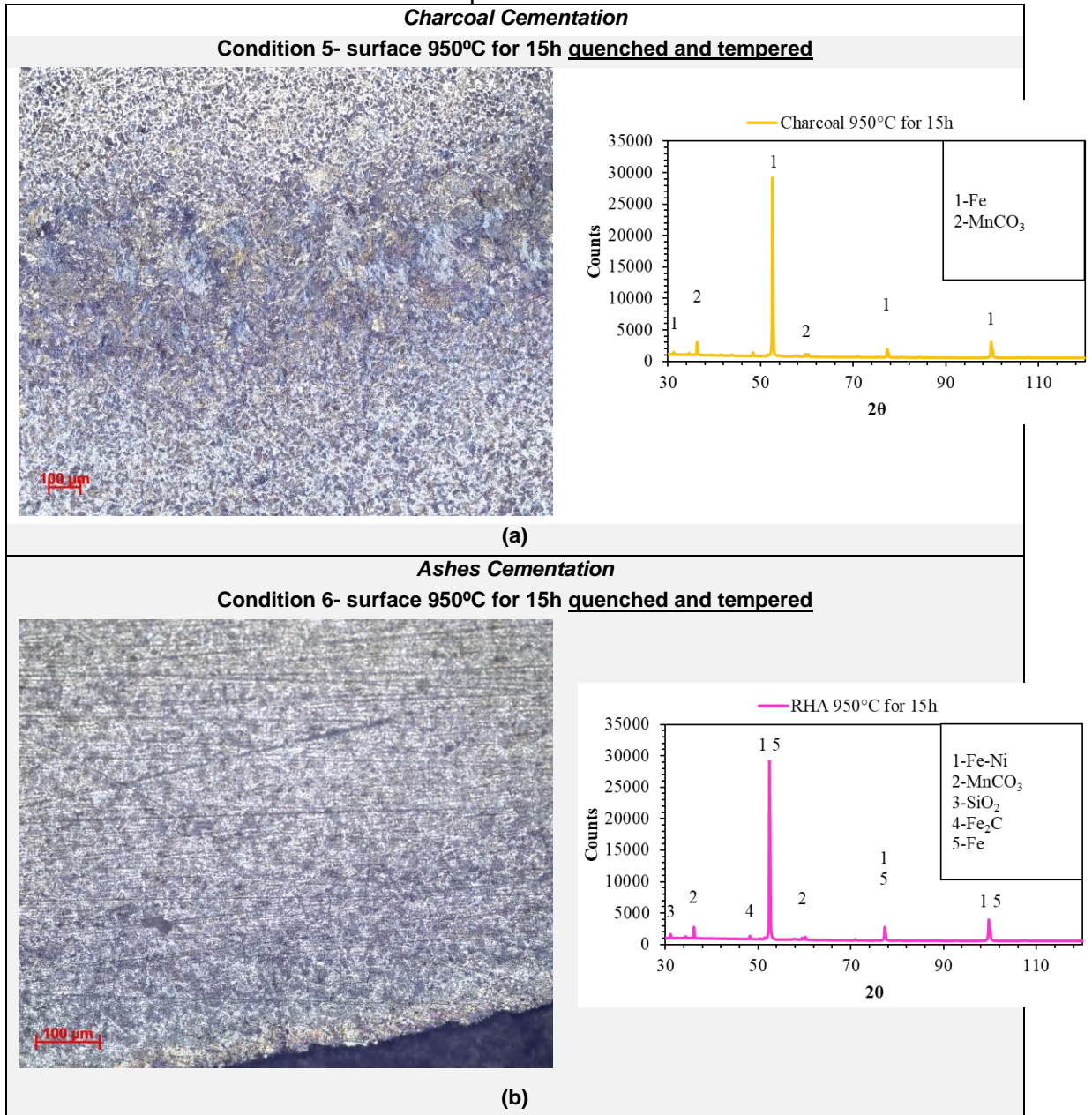
Figure 6 - Optical microscope of microstructure near the surface sample edge. Cementation at a temperature of 900°C for 15h. (a) cementing with charcoal powder (b) cementing with RHA powder.



Source: Authors.

At a carburizing temperature of 950°C and remaining in the furnace for 15 hours, the microstructure can be observed in figure 7. In Figure 7(a) shows the formation of martensite on the surface. The X-ray diffraction graph does not show the formation of iron carbide. Figure 7(b) shows no changes in grains, showing silica formation.

Figure 7 - Optical microscope of microstructure near the surface. Carburizing at a temperature of 950°C for 15h. (a) carburizing with charcoal powder (b) carburizing with RHA powder.



Source: Authors.

3.2 MICROHARDNESS TEST RESULTS

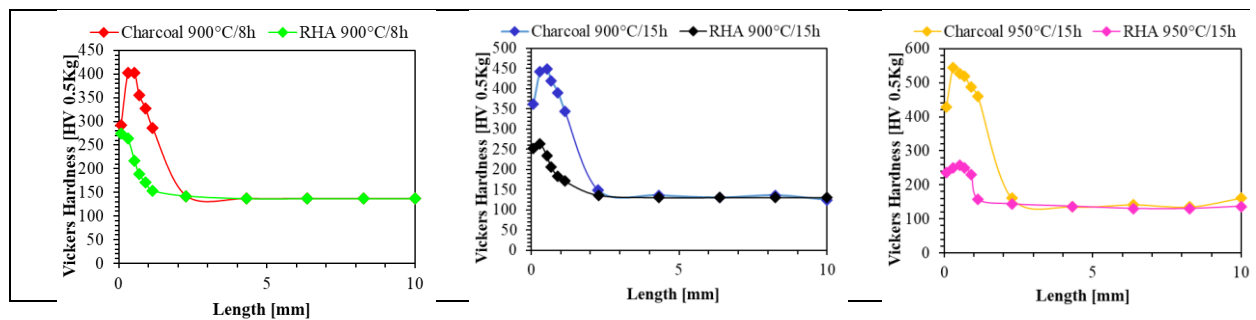
The SAE 1020 steel without heat treatment had an average microhardness of 135.42 HV uniform, with an average standard deviation of 2.17. The figure 8 shows profile graph of micro hardness. Where the increase in hardness is greater for condition 1; according to Figure 8(a); with the charcoal carburizing agent, with a hardness of 409 HV, with martensite formation on the sample surface. In condition 2 with ash carburizing agent, the measurement was 280 HV, which is due to the small formation of silicon carbide and silica. With this, the core maintained the SAE 1020 hardness.

With the longer time in the furnace of 15h, by yourself, condition 3 graph; at

Figure 8 (b); shows a hardness of 454.3 HV with the formation of a structure richer in carbon and greater formation of martensite. In condition 4, the surface hardness was 454 HV due to the small formation of silicon carbide. Comparing with the previous hardness, contributing to more silicon carbide and silica diffusion.

Finally, Figure 8 (c) shows the graph of the treatment with the same time of 15h and an increase in temperature of 950°C. Condition 5 had a higher hardness than the previous ones of 538.8 HV. In condition 6 with the ash carburizing agent, the hardness dropped to 255.9 HV, because the amount absorbed on the silica surface was not enough. According to the X-ray diffractogram, silica formation occurred, contributing to an increase in hardness.

Figure 8 – Profile graph of microhardness as a function of the distance from the surface. These are hardness profiles after quenching and tempering.

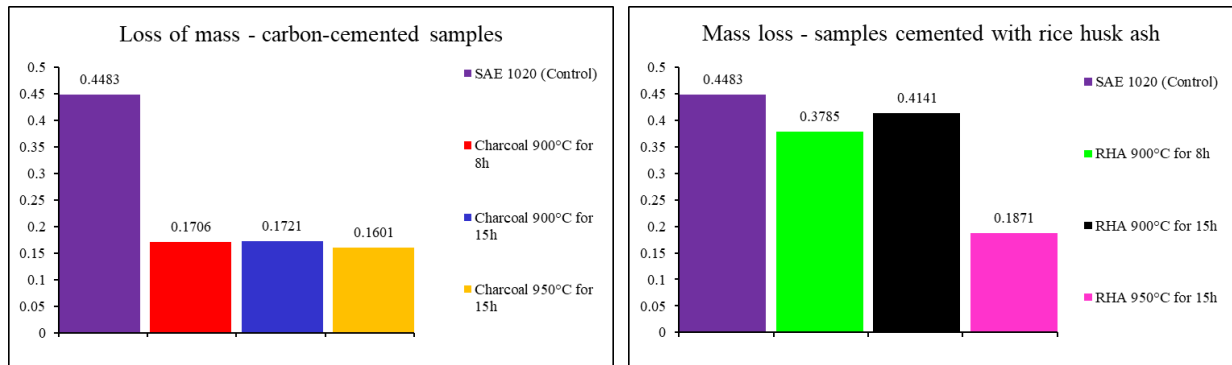


Source: Authors.

3.3 WEAR TEST RESULTS

Figure 9 shows the graph with the average results of percentage mass loss in relation to the initial mass for the samples that received cementation with charcoal and RHA. Analyzing these results presented in the graph, it can be seen that SAE 1020 steel has very low wear resistance, with a mass loss of 0.4483% during the tests. In the samples that received cementation with charcoal and ash; in (a); a significant improvement in the results can be seen. Where the best result was the condition where the cementation was carried out at 950°C, for 15 hours, with carbon cementing agent, where it lost 0.1601% of mass, respectively. Therefore, it can be concluded that the SAE 1020 steel cemented with charcoal at 950°C for 15 hours, after quenching and tempering, presents an improvement in wear resistance compared to the SAE 1020 steel without any heat treatment. Figure 9(b) shows the wear of the treatment with ash cementing agent and the average results of percentage mass loss in relation to its initial mass for the samples that received cementation with RHA. This wear resistance result found in the process with the RHA cementing agent was due to the formation of SiO_2 , reducing the mass loss.

Figure 9 - Wear analysis (mass loss): a) charcoal cementing, after quenching and tempering b) RHA cementing, after quenching and tempering.



Source: Authors.

4 CONCLUSIONS

- Rice husk ash contains residual carbon and silica; the carbon diffused into the steel surface during the carburizing process. The microstructure exhibited a martensitic structure with silica. The silica present forms thin protective layers of oxides/silicates, which contribute to oxidation resistance;
- After analyzing the experimentally obtained data and the graphs generated for the microhardness profiles of the 6 treatment conditions compared, it is possible to observe a large difference in the surface hardness of the samples. The results were superior for the samples that received carburization with charcoal. For the samples that received carburization with RHA, condition 6 (950°C 15h) obtained the best result, with an average surface hardness of 361.0 HV, while the average hardness of 1020 steel without heat treatment is 135 HV. In the samples that received carburization with charcoal, condition 5 (950°C 15h) presented the best result, increasing the average surface hardness of 1020 steel to 538.8 HV;
- The results obtained with the wear test were satisfactory and showed a significant improvement in wear resistance when compared to SAE 1020 steel without heat treatment for the 2 types of carburizing agent. The best result was observed in the cementation carried out at 950°C for 15 hours, where the material with cementation using charcoal obtained an improvement of 62.23%;
- In general, RHA can be used as a source of carbon and silica for thermochemical solid cementation treatment, only for mechanical components where a small thickness of the cemented layer is desired, and an average surface hardness of around 360 HV.

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