

COB-2023-0716

EFFECTS OF THE SOLID CARBURIZING WITH BABASSU COCONUT (*ORBIGNYA PHALERATA*) ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES ON LOW CARBON STEELS

Denilson Santos Monteiro

Universidade Estadual do Maranhão, Departamento de Engenharia Mecânica, Cidade Universitária Paulo VI – Avenida Lourenço Vieira da Silva 1.000 – São Luís/MA.

denilson.smonteiro@outlook.com

Sóstenes Pinheiro Campos

Instituto Federal de Educação, Ciência e Tecnologia do Maranhão – IFMA, Av. Getúlio Vargas, Monte Castelo - São Luís, MA – Brasil.

sostenes.spc@gmail.com

Rodrigo da Silva Miranda

Universidade Estadual de Campinas - UNICAMP, Faculdade de Engenharia Mecânica, Cidade Universitária "Zeferino Vaz" - Barão Geraldo, Campinas – SP.

rodrigo.sv.miranda@gmail.com

Paulo Roberto Campos Flexa Ribeiro Filho

Universidade Estadual do Maranhão, Departamento de Engenharia Mecânica, Cidade Universitária Paulo VI – Avenida Lourenço Vieira da Silva 1.000 – São Luís/MA.

paulofilho@professor.uema.br

Adilto Pereira Andrade Cunha

Universidade Estadual do Maranhão, Departamento de Engenharia Mecânica, Cidade Universitária Paulo VI – Avenida Lourenço Vieira da Silva 1.000 – São Luís/MA.

adiltocunha@professor.uema.br

Abstract. *The present study aims to evaluate the effects of solid carburizing using babassu coconut (*Orbignya phalerata*) charcoal on the microstructure and mechanical properties of ASTM A36 steel. Circular samples with a diameter of 12 mm were employed and subjected to carburizing treatments with babassu coconut charcoal at 900°C for 1, 3, 5, and 7 hours, followed by water quenching. Additionally, a commercial carburizing granulate, Duro Carbon 310, was used for comparison. Hardness tests revealed an increase in surface hardness after the treatment, with samples carburized for seven hours displaying a 46.6% increase for coconut charcoal and a 52.3% increase for the granulate in comparison to the as-quenched material. Material analysis showed the evolution of the carburized layer with treatment time, reaching a maximum thickness of 1.36 ± 0.04 mm for the coconut charcoal treatment and 1.56 ± 0.04 mm for the granulate treatment after seven hours. Microstructural analysis indicated the presence of microconstituents such as ferrite, pearlite, bainite, and martensite in the core, while in the carburized layer, a predominantly martensitic structure with bainite nucleation was observed in some regions.*

Keywords: *Carburizing, Babassu Coconut, Martensite, Thermochemical Treatment, ASTM A36 Steel.*

1. INTRODUCTION

The production of ferrous alloys, especially steel, is essential in engineering and various sectors, thanks to its ease of processing and the various manufacturing and treatment techniques available, which allow for a wide range of physical and mechanical properties (CALLISTER; RETHWISCH, 2016; NEVES et al., 2018). The possibility of applying solid-state transformations and treatments that modify their properties makes these materials highly versatile to meet various needs in sectors such as construction, automotive, military, and household utensil manufacturing (ASKELAND; WRIGHT, 2014; BHADSHIA; HONEYCOMBE, 2017).

The properties of steels depend on their structure, and thus, in many applications, it's necessary to modify these properties to adapt them to the stresses resulting from their use. One category of treatments that can alter the properties of steels is thermochemical treatments, which aim to change the chemical composition of the material in specific sections or surfaces, involving the addition of elements such as carbon, boron, and nitrogen to the steel surface. These

treatments can be carried out in solid, liquid, gaseous media, or by plasma techniques, enhancing wear and corrosion resistance (CHIAVERINI, 2012; GALIOTTO et al., 2019; TAO et al., 2017).

The wear of mechanical components is a common problem in the industry, resulting in efficiency loss, power reduction, and high consumption of energy and lubricants. Therefore, techniques that improve the surface resistance of steels are essential to ensure the proper functioning of components and reduce downtime for maintenance and replacement of worn parts (PERINI; VILLANOVA, 2019).

Organic methods for carburization have been under examination for use in the process, aiming to not only reduce costs but also address environmental concerns, minimizing carbon emissions and promoting the clean and responsible utilization of resources (ADEDIPE et al., 2023). Babassu coconut (*Orbignya phalerata*), a product of a native palm tree, is an abundant material in the state of Maranhão, serving as a primary source of income for many families in the region through the production and sale of its derivative products, including oils, olive oils, cleaning products, charcoal, among others. It is estimated that over 300,000 women, known as "coco breakers," make a living from this activity (CONAB, 2021). The production of babassu charcoal in the interior of the state of Maranhão is carried out through the carbonization of the entire fruit in a typically rudimentary process, resulting in a product with a fixed carbon content of approximately 85% (REIS et al., 2015; SOUSA, 2020).

In this context, this study aims to evaluate the effects of solid carburization using babassu coconut charcoal on the microstructure and mechanical properties of ASTM A36 steel, a low-carbon steel widely used in structural components.

2. MATERIAL AND METHODS

2.1 Materials

In this study, ASTM A36 steel was used in circular bars with a diameter of 12 mm, in the as-rolled condition, which has the chemical composition shown in Table 1.

Table 1. Chemical composition of ASTM A36 steel (FERRARI; NEVES; PANÃO, 2019).

Material	C máx	Mn	P máx	S máx	Si
ASTM A36	0,26	0,80 – 1,20	0,03	0,03	0,15 – 0,04

Samples with a 12 mm diameter and 55 mm length were used for the analysis of the material in its initial condition and for the carburizing treatments performed. As a carburizing medium, crushed babassu coconut charcoal (*Orbignya phalerata*), obtained in the municipality of São João Batista in the State of Maranhão (Brazil), was used, along with a commercial carburizing granulate, Duro Carbon 310, for the purpose of comparing the efficiency of carburization with babassu coconut. Figure 1 illustrates the carburizing media used in each treatment.



Figure 1. Carburizing means (a) Babassu coconut (*Orbignya phalerata*); (b) Babassu coconut charcoal; (b) Duro Carbon 310 carburizing granulate.

2.2 Methods

Figure 2 shows the flowchart of the experimental procedure conducted on each sample. The material in the initial condition, as-rolled only, was analyzed through hardness testing and metallographic characterization to evaluate the microstructural formation and hardness of the material without any heat treatment. Subsequently, the material was austenitized at a temperature of 900 °C for 25 minutes, followed by quenching in water at room temperature while still without carburization treatment, to evaluate the ability to form martensite and the hardness obtained by the material without the carburizing treatment.

The solid carburizing treatment was conducted using a steel box to accommodate the samples, immersed in their respective carburizing means for each treatment. Clay was used to seal the box to prevent the escape of gases produced during the carburizing process. The treatments were performed by maintaining the furnace temperature at 900 °C for 1, 3, 5, and 7 hours for each carburizing medium, followed by quenching treatment, in order to evaluate the formation of the carburized layer, the corresponding increases in layer depth with increasing carburizing time, and the evolution of surface hardness of the material through the formation of martensite.

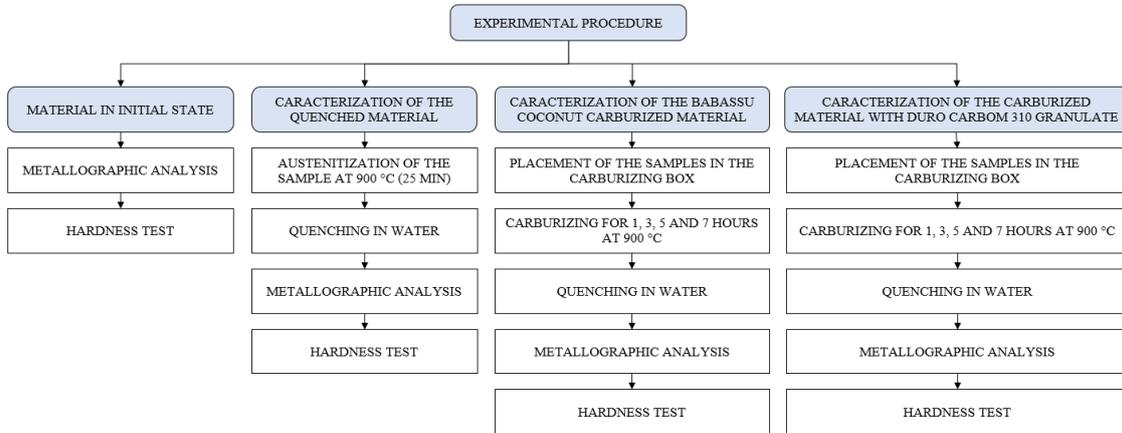


Figure 2. Experimental procedure flowchart.

For hardness analysis, as recommended by Garcia, Spim, and Santos (2012), the Rockwell B method was adopted to evaluate the hardness of the untreated material, and the Rockwell C method was used to evaluate the carburized and quenched samples. Measurements were taken at four points along each specimen, with the first point at the specimen's edge, the second and third points at distances of 2 and 4 mm, respectively, and the fourth point at 6 mm from the edge, representing the center of the material. For the metallographic analysis, the specimens were prepared by starting with grinding using SiC abrasive papers with grit sizes of #100, #220, #320, #400, #600, and #1200. Next, the samples were polished using a 1 µm diamond paste as the abrasive material. After polishing, chemical etching was performed using a 2% nital reagent (98% ethanol and 2% nitric acid) for a duration of 10 seconds for the untreated material and 15 to 20 seconds for the carburized and quenched material.

3. RESULTS AND DISCUSSION

Figure 3 shows the micrographs obtained for ASTM A36 steel in its initial condition. It can be observed that the structure is composed of a ferritic matrix with the presence of dispersed pearlite throughout the material's microstructure. Figure 3b presents the characteristic lamellar formation of pearlite. This microstructural aspect is similar to what was shown by Arif et al (2022) and by Silva and Mei (2021), as this material is a low-carbon steel that is only rolled, and it does not exhibit good hardenability.

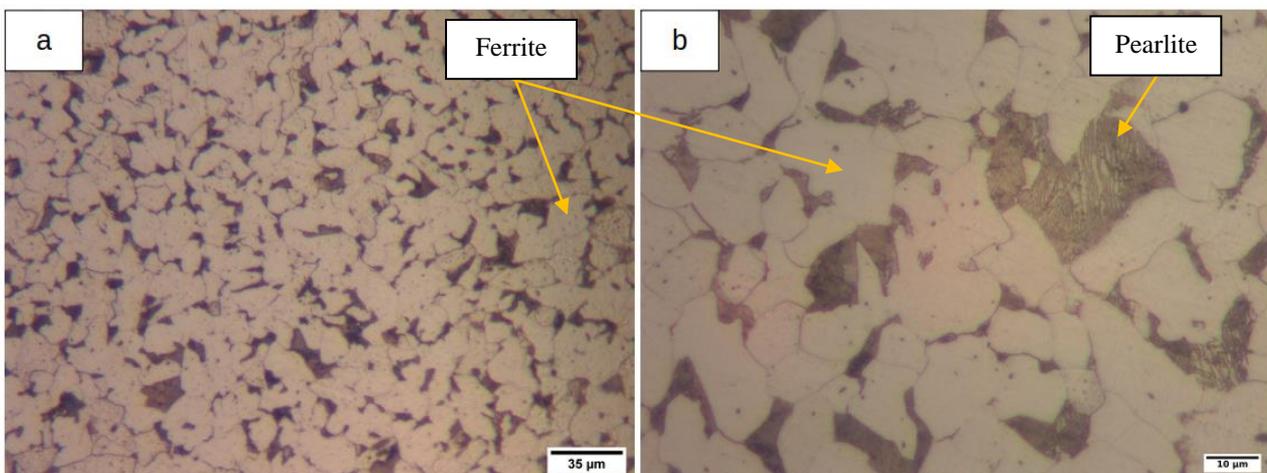


Figure 3. Micrograph of ASTM A36 steel in the initial condition. (a) 200x magnification; (b) 500x magnification.

Figure 4 shows the micrographs obtained for the sample tempered without prior carburization. The formation of martensite can be observed, as well as the formation of bainite (Figure 4b) (YIN; HILBERT; BORGENTAM, 2017; BHADSHIA; HONEYCOMBE, 2017). Due to the low carbon content and the absence of appreciable amounts of alloying elements, the steel has low hardenability. Therefore, even with the rapid cooling induced by quenching, the formation of other microconstituents occurs, in addition to martensite and bainite. This aspect is similar to that shown by Silva and Mei (2021) for a low-carbon tempered material.

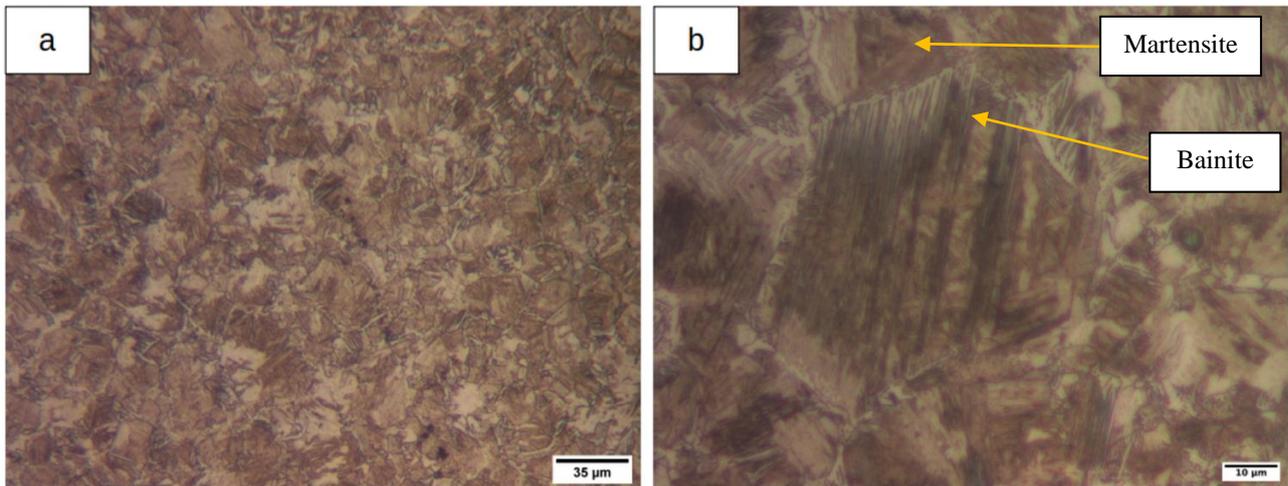


Figure 4. Micrograph of quenched ASTM A36 steel. (a) Microstructure - 200x; (b) Bainite formation - 500x.

Figure 5 shows the carburized specimens for the treatment times of 1, 3, 5, and 7 hours. Through macroscopic analysis of the samples, it is possible to identify the formation of the carburized layer for each specimen and the corresponding increase in layer thickness with longer treatment times, indicating that the process exhibited the expected formation.

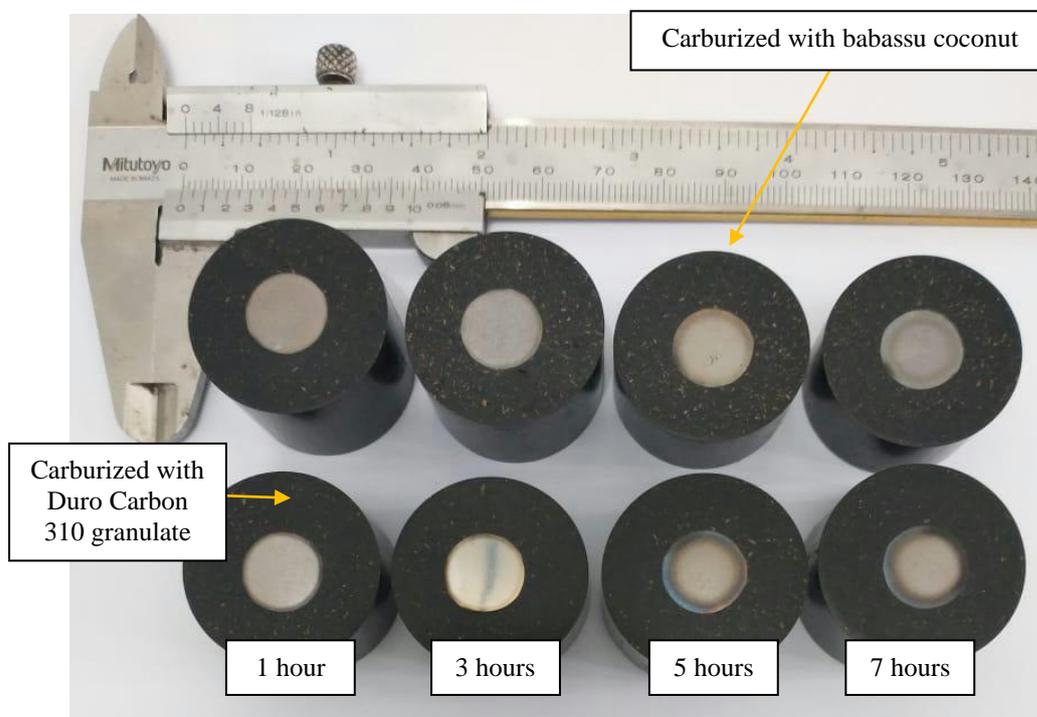


Figure 5. Carburized specimens for metallographic and hardness analysis.

The micrographs for the material carburized for 1 hour are shown in Figure 6. Analyzing figures 6a-c, the carburized layer can be identified, having a similar appearance to that found by Fetig and Pimenta (2021), consisting of

a martensitic structure with the formation of bainite in some regions. In the central region of the samples, as seen in figures 6b-d, the microstructure consists of martensite and bainite, as well as areas where the presence of other constituents is suggested (SILVA; MEI, 2021). This aspect is similar to the tempered material in Figure 4, as the increase in carbon content occurs only at the surface of the sample, and, consequently, the center should retain its original characteristic.

The formation of the carburized layer, as shown in Figure 6, reveals that there was indeed an increase in carbon content on the surface due to the treatment. The sample treated with the granulate exhibits a greater depth of the layer in the material. This can be justified by the fact that the commercial carburizing granulate Duro Carbon 310, in addition to having a higher carbon content than the babassu coconut charcoal, contains a carburizing activator in its chemical composition responsible for accelerating the diffusion process, as it is specifically developed for this application.

Considering that the babassu coconut charcoal was obtained without control over its chemical characteristics due to the rudimentary manufacturing process, its carburizing power is lower than that of the commercial product, which has controlled properties. Another important factor is the non-use of a carburizing activator for the babassu charcoal, to evaluate its performance in its natural state.

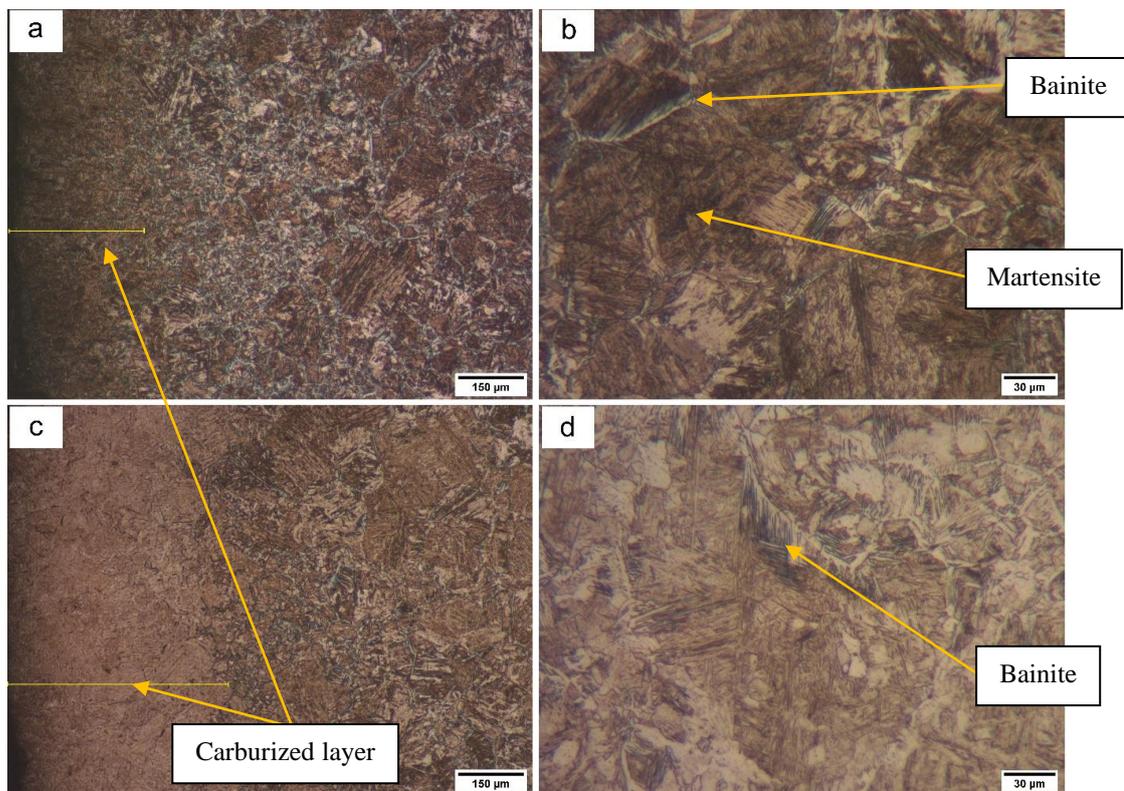


Figure 6. Micrograph of ASTM A36 steel carburized for 1 hour: (a) With coconut - End of the specimen; (b) With coconut - Central region of the specimen; (c) With granulate - End of the specimen; (d) With granulate - Central region.

The same behavior observed for the 1-hour treatment can be seen in the samples treated for 3 and 5 hours. The micrographs for the material carburized for 5 hours are displayed in Figure 7. It is possible to identify the formation and increase in the thickness of the carburized layer for both media. In the central region, Figures 7b-d, the microstructure remains similar to that of the tempered material shown in Figure 4, with the formation of martensite and bainite at the grain boundaries.

Finally, Figure 8 displays the structure formed after the 7-hour treatment. Figures 8a and 8c depict the ends of each specimen with their respective carburized layers, comprising a martensitic structure with the presence of bainite in some regions. It is noticeable that the carburized layer for each sample covers the entire area of the image in both treatments. Therefore, it is not feasible to visualize the complete layer in a single image due to the limited resolution of the 50x optical microscope used.

According to Figures 8b and 8d, differences in the microstructural formations of the carburized materials compared to the as-tempered material in Figure 4 are observed. In this case, an increase in grain size can be identified, which is related to the considerable time that the samples spent at high temperatures, a crucial factor for grain growth, as pointed out by Silva and Mei (2021). This fact can be subsequently confirmed through hardness measurements, where an increase in grain size results in decreased hardness in the material's core. Chiaverini (2012) mentions that for coarser-grained steels, further grain refinement steps may be necessary.

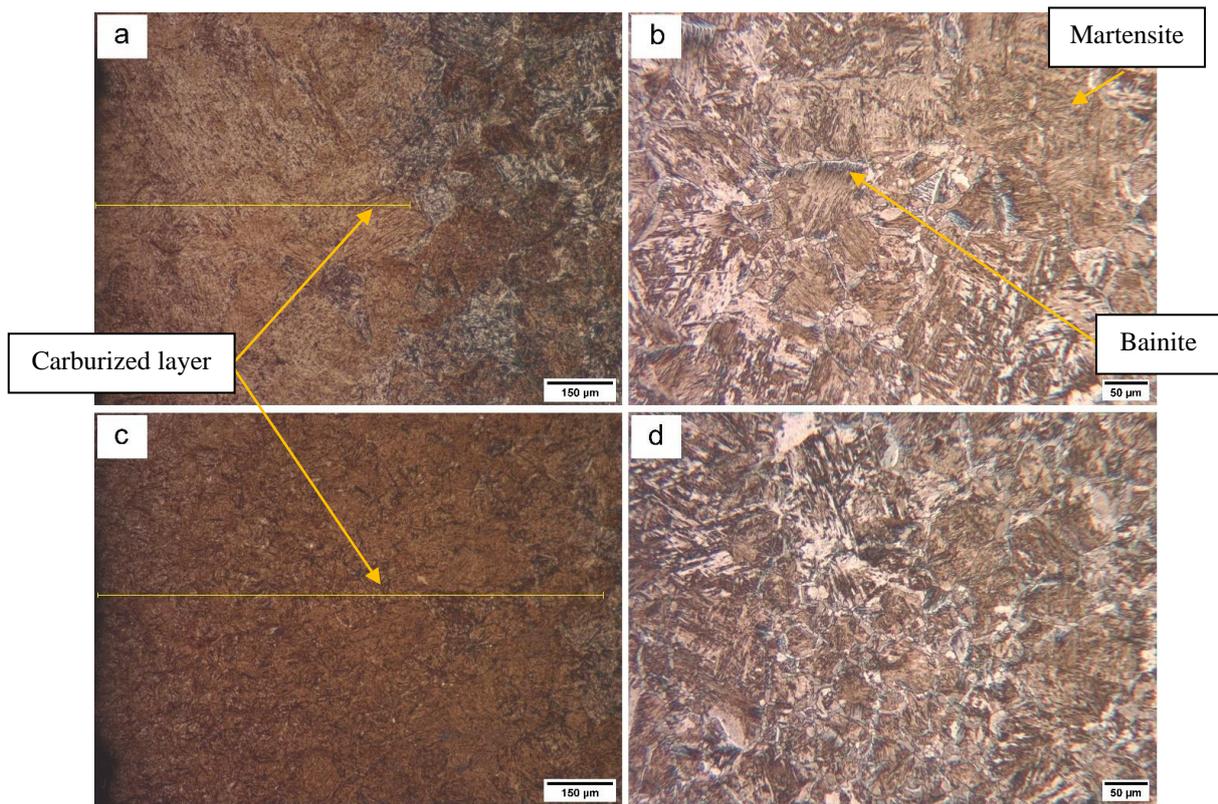


Figure 7. Micrograph of ASTM A36 steel carburized for 5 hours. (a) Coconut - end of the specimen; (b) Coconut - central region of the specimen; (c) Granulate, end of the specimen; (d) With granulate, central region of the specimen.

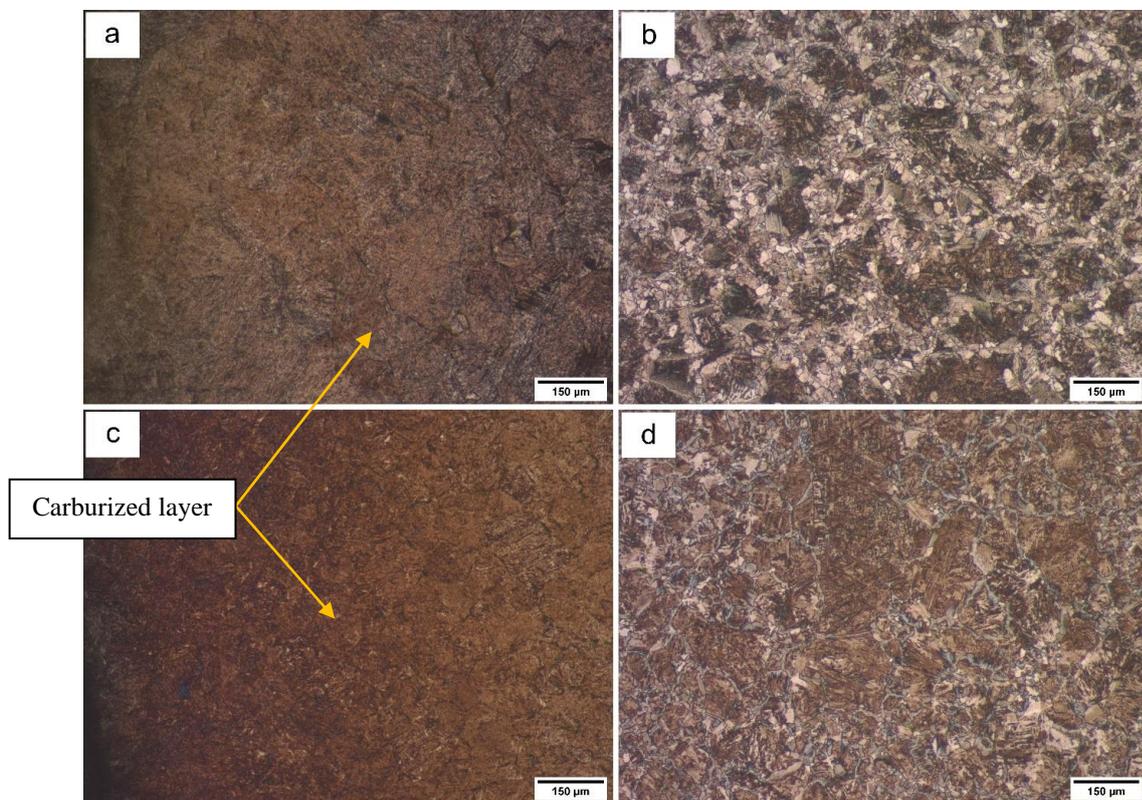


Figure 8. Micrograph of ASTM A36 steel carburized for 7 hours. (a) Coconut - End of the specimen. (b) Coconut - Central region of the specimen. (c) Granulate - End of the specimen. (d) Granulate - Central region.

The measured values for the thickness of the carburized layer for the treatments with babassu coconut charcoal and the commercial granulated hard carbon 310 are presented in Figure 9, which illustrates the evolution of the carburized layer with increasing carburizing time. As observed in the micrographs of the samples, the cementation granulate exhibited better carburizing potential, resulting in greater layer thicknesses for each treatment.

For the 1-hour treatment, the obtained thicknesses for the carburized layer were 0.28 ± 0.023 mm and 0.42 ± 0.018 mm for the samples treated with babassu coconut charcoal and granulate, respectively. In this case, the material carburized with the granulate exhibited a carburized layer approximately 50% thicker than the sample treated with babassu charcoal.

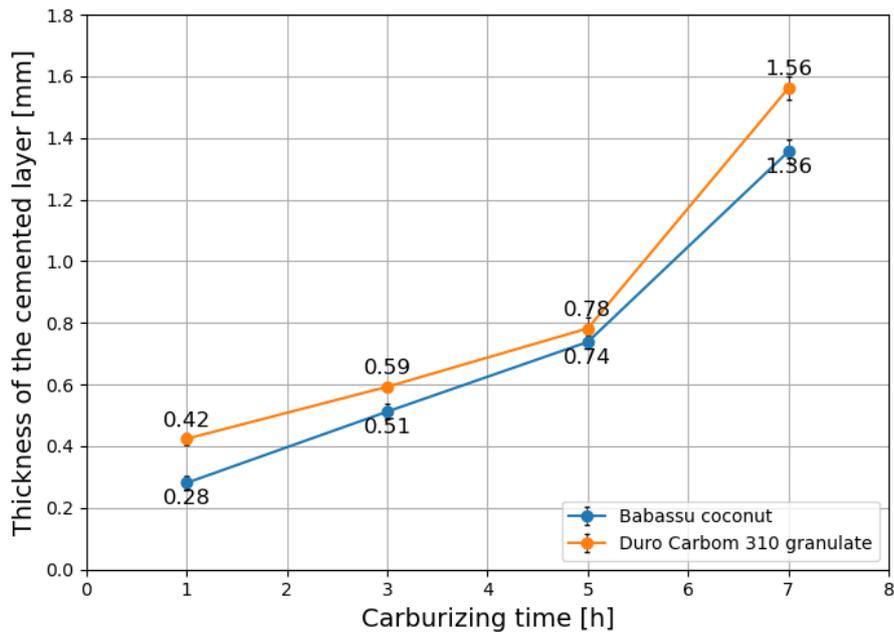


Figure 9. Thickness of the carburized layer for each treatment.

In the 3-hour treatment, the difference was significantly smaller compared to the 1-hour treatment. The carburized material with babassu coconut charcoal exhibited a carburized layer thickness of 0.51 ± 0.024 mm, while for the granulate, the thickness was 0.59 ± 0.011 mm, approximately 15.7% higher than that obtained with coconut charcoal.

The trend of decreasing difference continued for the 5-hour treatment. The carburized layers obtained for coconut charcoal and the granulate had thicknesses of 0.74 ± 0.022 mm and 0.78 ± 0.034 mm, respectively. Therefore, the layer for the granulate was approximately 5.4% thicker than the carburized layer obtained with coconut charcoal.

Finally, with 7 hours of treatment, the sample carburized with babassu coconut charcoal presented a layer thickness of 1.36 ± 0.037 mm. On the other hand, the sample treated with the granulate had a layer thickness of 1.56 ± 0.038 mm. In this case, the percentage difference between the thicknesses increased to 14.7%.

It is evident that, in terms of carburizing efficiency, Duro Carbon 310 granulate outperforms babassu coconut charcoal due to the presence of an activator in its composition, which reduces the energy required for diffusion. However, as a proposed method for achieving a high-quality carburized layer, babassu coconut charcoal has shown great promise, achieving layer thicknesses for longer treatments that are very close to the values obtained with the commercial product. An excellent way to enhance the carburizing efficiency of coconut charcoal would be to explore methods to improve its production process and combine it with an activator, ensuring greater carburizing power and product reliability.

Figure 10 shows the evolution of the average surface hardness of the material for each condition and treatment medium. The surface hardness of the material increases with the increase in treatment time, which is directly related to the greater carbon penetration on the surface and consequently, greater formation of the carburized layer.

It can be observed that for the 1-hour treatment, the surface hardness measurement obtained for the material cemented with coconut was 38 ± 1.41 HRC, while the material cemented with the granulate had an average of 40.5 ± 0.71 HRC, a difference of approximately 6.6%. For the 3-hour treatment, the hardness measurement obtained for the material cemented with coconut was 42.8 ± 1.06 HRC, while for the material cemented with the granulate, the value obtained was 43.5 ± 0.701 HRC, an increase of approximately 1.6% compared to the material cemented with babassu. This treatment showed the smallest percentage difference between the two samples.

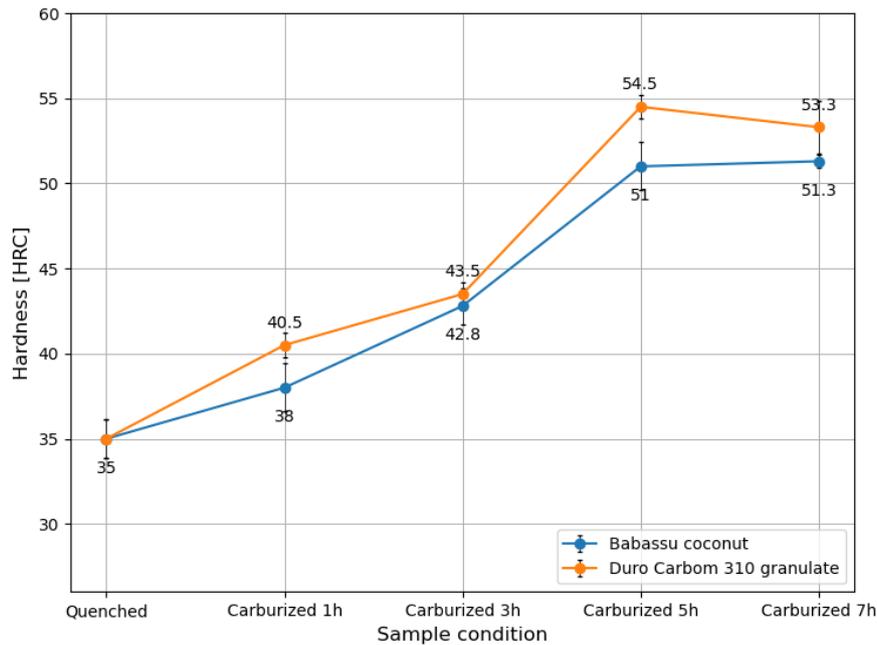


Figure 10. Evolution of surface hardness as a function of treatment time.

As the carburizing time increases, the thickness of the carburized layer also increases, and consequently, the hardness measurement also shows a higher value. This fact can be observed in the 5-hour treatment, where the hardness measurements are 51 ± 1.41 HRC for the treatment with babassu coconut charcoal and 54.5 ± 0.71 HRC for the treatment with the granulated material, representing a difference of approximately 6.9%.

For the material carburized for 7 hours, the hardness measurements obtained for the treatments with babassu coconut charcoal and the granulate were 51.3 ± 0.35 HRC and 53.3 ± 1.57 HRC, respectively, representing a difference of approximately 4%. For this treatment, the hardness values obtained are very close to those of the material treated for 5 hours, indicating that the material may have reached the hardness limit for the treatment used.

The hardness measurements on the surface for the material carburized with babassu coconut charcoal showed a difference of less than 10% compared to the material carburized with the commercial granulate. In the 3-hour treatment, as mentioned earlier, the hardness for the material carburized with Duro Carbon 310 was approximately 1.6% higher than the material carburized with babassu coconut charcoal. Figures 11 and 12 show the comparisons of the hardness values obtained for the treatments with each carburizing medium.

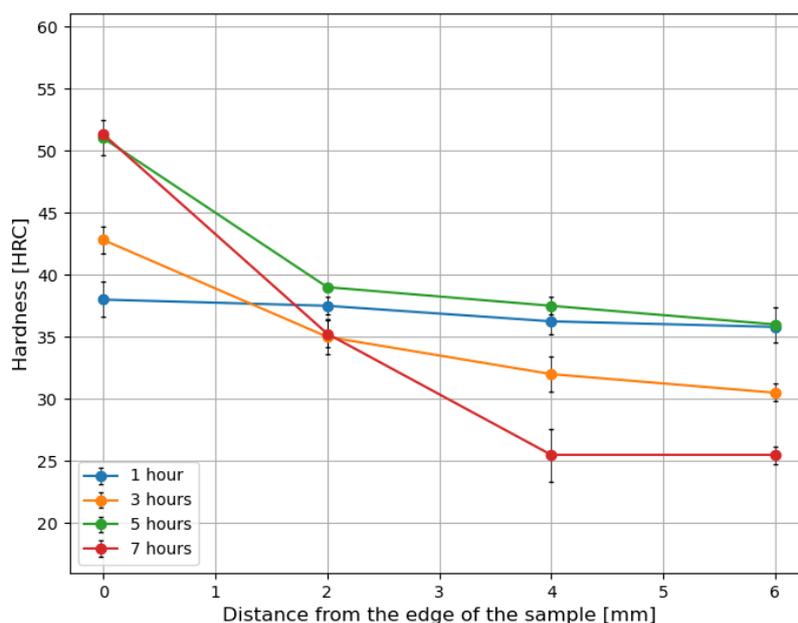


Figure 11. Comparison of the hardness measurements obtained for each treatment with babassu coconut charcoal.

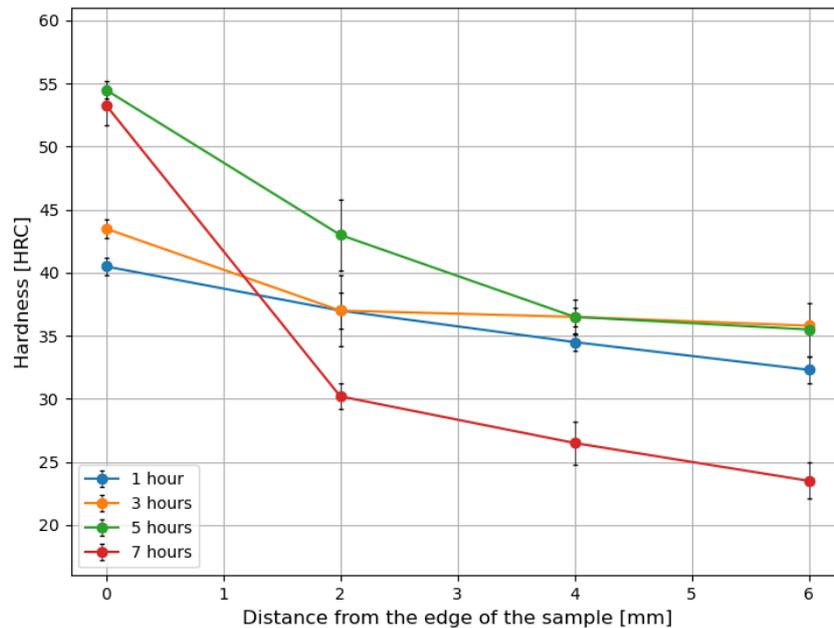


Figure 12. Comparison of the hardness measurements obtained for each treatment with Duro Carbon 310 granulate.

According to figures 11 and 12, it can be observed that the hardness values obtained for each sample during the 1, 3, and 5-hour treatments are similar for both cementation media, and the hardness values obtained in the core resemble those of the material treated with tempering only. This behavior was expected since there is no increase in carbon content in the material's core.

However, for the 7-hour treatment, the hardness measurements in the center of the samples show considerably lower values compared to the material that was only tempered. This fact can be attributed, as mentioned before, to the increase in grain size of the material, which consequently reduces the hardness of the material in the core, as shown in Figure 8. Therefore, to address this issue, a normalization treatment could be employed to refine the austenitic grain and increase the material's core hardness (CHIAVERINI, 2012; SILVA; MEI, 2021). Considering that the hardness values on the material's surface resemble those treated for 5 hours, conducting the carburizing process with this treatment time appears more appropriate.

It is important to highlight that the samples treated with the carburizing granulate show higher hardness values and a greater thickness of the carburized layer compared to the samples treated with babassu charcoal. These characteristics result from the fact that the granulate not only possesses uniformity in its properties due to its high degree of manufacturing control but also contains a carburizing activator that ensures better performance for the product.

On the other hand, the babassu coconut charcoal lacks control over the manufacturing process and its properties, which makes process control more complex, and both the formation of the cemented layer and hardness values tend to be more dispersed. However, when it comes to the formation of the cemented layer and surface strength enhancement, the babassu coconut charcoal has shown great promise, obtaining values very close to those obtained for the industrial material, considering that no cementation activator was used for the treatments.

4. CONCLUSION

Based on the results obtained, an increase in surface hardness was observed, as well as the formation of the carburized layer through the treatment. Microstructural analysis reveals the formation of martensite in the carburized layer region and the evolution of its thickness with increasing treatment time, reaching maximum values of 1.36 ± 0.037 mm for the babassu coconut charcoal treatment and 1.56 ± 0.038 mm for the commercial granulate treatment after seven hours. Hardness measurements indicated an increase in surface hardness with the application of the process, with maximum increases of approximately 46.6% and 55.7% in the treatments using babassu coconut charcoal and the commercial granulate, respectively, compared to the quenched material.

Therefore, babassu coconut charcoal, abundant in certain regions of northern and northeastern Brazil, demonstrates its potential for use as a carburizing medium, delivering results comparable to those achieved with commercial materials. Moreover, babassu coconut plays a crucial role for many families who rely on it as a source of income. Therefore, this alternative has the potential to provide a more sustainable process and, in the future, become an additional source of income for the communities that depend on this resource.

5. REFERENCES

- Adedipe, O. et al. Sustainable carburization of low carbon steel using organic additives: A review. *Sustainable Materials and Technologies*, 2023.
- Arif, S. M. et al. Effect of quenching and partitioning time on microstructure and mechanical properties of low carbon micro-alloyed steel. *Materials Today: Proceedings*, 2022, 3865–3869.
- Askeland, D. R.; Wright, W. J., *Ciência e engenharia dos materiais*. 3ª ed. São Paulo: Cengage Learning, 2014.
- Bhadeshia, H. K. D. H; Honeycombe, R. W. K. *Steels - Microstructure and Properties*. 4ª ed. Butterworth Heinemann, 2017.
- Callister, JR., W. D.; Rethwisch, D. G. *Ciência e Engenharia dos Materiais: Uma Introdução*. LTC, v. 9ª Edição, 2016.
- Chiaverini, V. *Aços e Ferros fundidos*. 7º Ed. São Paulo: Associação Brasileira de Metalurgia, Materiais e Mineração – ABM, 2012.
- CONAB - COMPANHIA NACIONAL DE ABASTECIMENTO. *Boletim da Sociobiodiversidade*, Brasília, DF, v. 5, n. 5, Outubro 2021.
- Ferrari, M.; Neves, M.D.M.; Panão, J. N. Análise das propriedades mecânicas de juntas de aço carbono estrutural soldadas pelo processo arco submerso com os arcos simples e duplo (Tandem-Arc). *Soldagem & Inspeção*. 2019; 24:e2402.
- Fetig, B. L.; Pimenta, J. S. Análise da cementação sólida com têmpera direta aplicada em engrenagens cilíndricas de dentes retos (módulo 2) de aço SAE 8620. *Tecnol Metal Mater Min*. 2021. Vol.18, p.e2333
- Galiotto, A. et al. Characterization of Different Surface Layers Produced by Solid Boron Nitro-Carburizing Thermochemical Treatment on AISI 1020. *Materials Research*. 2019; 22 (5).
- Garcia, A. Spim, J. A. Santos, C. A. *Ensaio dos materiais*. 2. ed. Rio de Janeiro: LTC, 2012.
- Neves, A. S. et al. Caracterização mecânica e microestrutural de um aço com baixo teor de carbono – SAE 1010. *Revista TECCEN* (2018), 10 – 17.
- Perini, M., Villanova, D. L. Análise dos efeitos do tratamento termoquímico de boretação, aplicado a aços comerciais de larga utilização industrial: revisão. *TECNO-LÓGICA*, (2019), 160-166.
- Reis, A. R. S. et al. Comparação entre carvão de coco babaçu e carvão de resíduos madeireiros comercializados em Altamira – PA. *Ciência da madeira*, Vol.6 (2), p.100-106, 2015.
- Silva, A.L.C; Mei, P. R. *Aços e Ligas Especiais*. 4ª ed. São Paulo: Edgard Blücher, 2021.
- Sousa, V. M. C. Potencial do carvão de resíduos do coco babaçu e o desenvolvimento de protótipos de quebra do coco e carbonização. *Monografia (Graduação) - Universidade de Brasília – UnB*, Brasília, 2020.
- Tao, Q. et al. Ultrahigh hardness of carbon steel surface realized by novel solid carburizing with rapid diffusion of carbon nanostructures. *Journal of Materials Science & Technology* (2017), 1210-1218.
- Yin, J., Hilbert, M., Borgenstam, A. Morphology of Upper and Lower Bainite with 0.7 Mass Pct C. *Metall Mater Trans A* 48, 4006–4024 (2017).

6. RESPONSIBILITY NOTICE

The authors are the only responsible for the printed material included in this paper.