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Analysis of the Effect of Saltwater (NaCl) Solution Concentration During the Quenching Process on the Microstructure and Hardness of Medium Carbon Steel

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Abstract

This study analyzes the effect of sodium chloride (NaCl) solution concentration on the hardness and microstructure of medium-carbon steel. The heat treatment process was carried out using the hardening method, which included pre-heating at 600 °C for 30 minutes and austenitization at 850 °C for 25 minutes. The samples were then quenched in NaCl solutions with concentrations of 0%, 5%, 10%, and 15%. Analysis using the Spark-OES method revealed that the samples contained 0.303% carbon. The results showed that the NaCl solution concentration influenced the hardness by 71.73% and the toughness by 45.03% in medium-carbon steel. Furthermore, the hardening process induced a phase transformation from ferrite (a) and pearlite to martensite.

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Abstrak

Penelitian ini menganalisis pengaruh konsentrasi larutan garam (NaCl) terhadap kekerasan dan struktur mikro baja karbon menengah. Proses perlakuan panas dilakukan melalui metode hardening, yang mencakup pemanasan awal (pre-heating) pada 600 °C selama 30 menit, diikuti oleh austenisasi pada 850 °C selama 25 menit. Sampel kemudian di-quenching dalam larutan NaCl dengan konsentrasi 0%, 5%, 10%, dan 15%. Analisis menggunakan metode Spark-OES menunjukkan bahwa sampel memiliki kandungan karbon sebesar 0,303%. Hasil penelitian menunjukkan bahwa konsentrasi larutan garam berpengaruh sebesar 71,73% terhadap kekerasan dan 45,03% terhadap ketangguhan baja karbon menengah. Selain itu, proses hardening menyebabkan transformasi fasa dari ferrit (a) dan perlit menjadi martensit.

1. Introduction

The advancement of science and technology demands the availability of high-quality materials to meet industrial requirements. However, the limited availability of such materials necessitates the development of methods to optimize their utilization more effectively and efficiently. One widely used method for enhancing the mechanical properties of materials is hardening, a heat treatment technique aimed at increasing steel's hardness and wear resistance.

Medium-carbon steel is commonly used in hardening processes due to its balanced toughness and wear resistance. This material is widely applied in machine components subjected to friction or cyclic loading, such as gears, shafts, and bearings. However, these components undergo mechanical property degradation during operation due to continuous stress and friction (Surdia & Saito, 1985). Therefore, strategies for enhancing the mechanical properties of medium-carbon steel are essential to improve the efficiency and reliability of machine components, particularly in the automotive and railway industries.

Heat treatment is one of the primary methods for improving the mechanical properties of medium-carbon steel. It involves a controlled heating and cooling process to modify the material's microstructure, enhancing its hardness, toughness, and ductility (Karmin, 2009). A critical factor in this process is the quenching medium (quenchant), which determines the steel's final mechanical properties. An inappropriate selection of quenchant can lead to distortion and undesirable changes in mechanical properties (Karmin, 2009).

Various quenchants have been employed in quenching processes, including water, ice water, oil, NaOH, polymer, and saltwater (Sumiyanto & Abdunnaser, 2015). Each quenchant exhibits a different cooling rate, significantly influencing steel's phase transformation and mechanical properties. Among these, saltwater solutions

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offer several advantages over plain water, such as a faster and more uniform cooling process across the specimen surface, thereby reducing the risk of distortion. Additionally, quenching with saltwater promotes a more homogeneous microstructure and enhances material hardness through oxygen binding at the steel surface (Jordi et al., 2017).

Based on these considerations, this study aims to analyze the effect of saltwater concentration in the quenching process on the microstructural transformation and hardness of medium-carbon steel. The findings of this research are expected to provide insights into the optimal heat treatment parameters for improving material performance in industrial applications.

2. Research Methods

2.1 Materials and Equipment

The equipment used in this study includes a furnace, optical emission spectrometer (OES), grinding machine, shaper machine, milling machine, lathe machine, Vickers hardness tester, impact testing machine, optical microscope, crushing pliers, analytical balance, and spatula. The materials used include sodium chloride (NaCl), distilled water, and medium-carbon steel.

2.2 Sample Preparation

The medium-carbon steel was cut into specific dimensions for different tests. The specimens were prepared in the following forms:

- a. Cylindrical specimens with a diameter of 20 mm and a thickness of 20 mm were designated for Vickers hardness testing.
- b. Cubic specimens with dimensions 10 mm × 10 mm × 10 mm are used for microstructural characterization under an optical microscope.
- c. Rectangular specimens with dimensions 50 mm × 10 mm × 10 mm, prepared for impact toughness testing.

2.3 Saltwater Solution Preparation

Saltwater solutions were used as the quenching medium in this study. The solutions were prepared by dissolving NaCl in distilled water, with concentrations varied according to a predetermined procedure. The salt concentrations were expressed in weight percentage (wt%), calculated based on the mass ratio of NaCl to the total solution mass. The concentration variations used were 0%, 5%, 10%, and 15% wt%.

2.4 Chemical Composition Analysis

A chemical composition analysis was conducted to determine the elemental composition of the medium-carbon steel. The analysis was performed using Optical Emission Spectrometry (OES), a technique that enables precise identification of metal elements.

2.5 Heat Treatment Process

The heat treatment process was conducted using a furnace and consisted of three main stages:

- a. Pre-heating – The initial heating stage uniformly distributes heat within the material to ensure consistent treatment results. The pre-heating temperature was set at 600°C for 30 minutes (Ayodeji et al., 2011), below the critical temperature of 723°C (Surdia & Saito, 1985).
- b. Austenitization – The steel was heated to 850°C for 25 minutes to form an austenitic phase (Adawiyah et al., 2014).
- c. Quenching – In the final stage, the samples were rapidly cooled using saltwater solutions as the quenching medium. The quenching process was performed in solutions with NaCl concentrations of 0%, 5%, 10%, and 15% wt% to investigate the effect of salt concentration on the mechanical properties and microstructural transformation of the steel.

A detailed flowchart of the experimental procedure is presented in **Figure 1**.

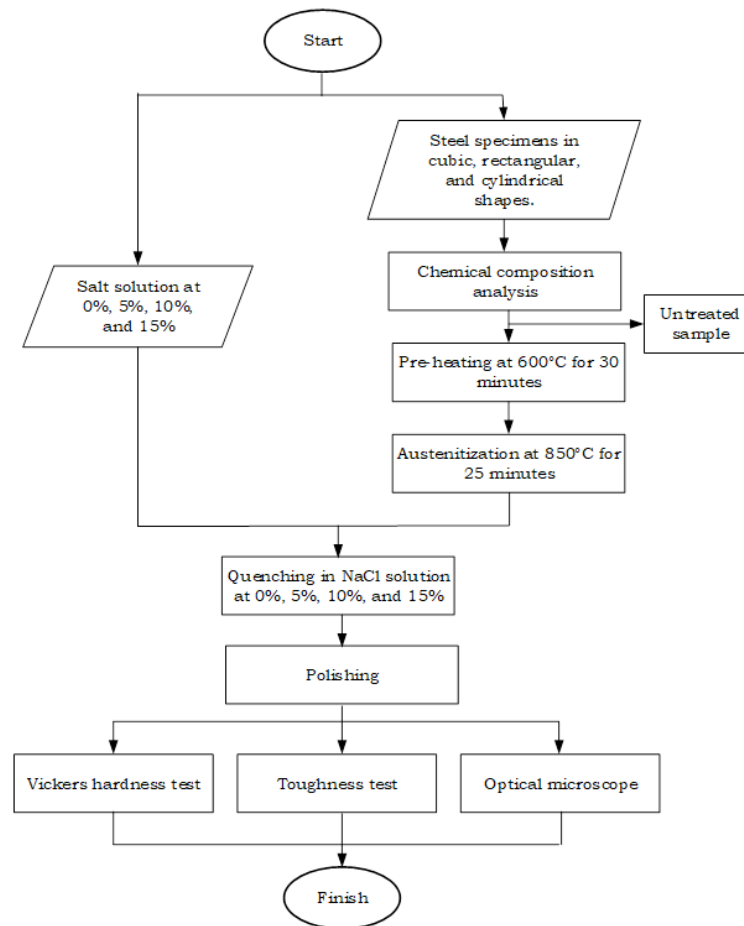


Figure 1. Research flowchart

3. Results and Discussions

In this study, the type of steel used contains elements, as shown in **Table 1**. The chemical composition analysis results indicate that the tested steel specimen contains various elements with different concentrations. Iron (Fe) is the predominant element, with a concentration of 98.5%. In addition to Fe, the most critical element in steel is carbon (C), which determines its classification and mechanical properties. According to the data in **Table 1**, the carbon content in the specimen is 0.303%. Based on the classification of steel by carbon content, this specimen is categorized as medium carbon steel (Suarsana, 2017). Apart from Fe and C, several other elements are present in varying concentrations, which can also influence the material's mechanical properties.

The mechanical properties of steel are crucial parameters in determining its suitability for various industrial applications. Characteristics such as hardness and toughness significantly affect steel's performance under operational conditions. Therefore, mechanical testing is necessary to evaluate material quality comprehensively. One commonly used method for measuring steel hardness is the Vickers hardness test. In this study, the Vickers hardness test results are presented in **Table 2** to provide insights into the changes in mechanical properties after heat treatment.

Table 2 presents the hardness test results obtained using the Vickers method. The highest hardness value was recorded for the sample quenched in a 15% salt water solution, with a hardness of 487.49 ± 10^{-2} HV. Conversely, the lowest hardness value was observed in the sample quenched in a 5% salt water solution, measuring 154.49 ± 10^{-2} HV. To further analyze the effect of saltwater concentration on the hardness of medium carbon steel, the relationship between these variables is illustrated in **Figure 2**.

Table 1. Chemical composition analysis using the Spark-OES method

No	Element	Content (%)
1	C	0.303
2	Si	0.18
3	Mn	0.417
4	P	0.0323
5	S	0.022
6	Cr	0.133
7	Mo	0.0143
8	Ni	0.0927
9	Al	0.0012
10	Co	0.0149
11	Cu	0.194
12	Nb	<0.004
13	Ti	0.017
14	V	<0.0005
15	W	0.0091
16	Pb	<0.002
17	Sn	0.0165
18	Mg	<0.001
19	As	0.0096
20	Zr	0.0042
21	Bi	<0.002
22	Ca	0.0003
23	Ce	0.0038
24	Sb	0.0035
25	Se	0.0013
26	Te	0.0046
27	Ta	0.0294
28	B	0.0012
29	Zn	0.0182
30	La	0.00055
31	Fe	98.5

Table 2. Hardness Test Results

No	Sample	Indentation/Sample H (HV) $\pm 10^{-2}$	Average Indentation H (HV) $\pm 10^{-2}$
1	A ₁₁	173.47	173.47
2	B ₁₁	193.34	172.85
3	B ₁₂	164.18	
4	B ₁₃	161.04	
5	C ₁₁	114.22	154.49
6	C ₁₂	127.92	
7	C ₁₃	221.33	
8	D ₁₁	190.06	209.44
9	D ₁₂	261.52	
10	D ₁₃	176.74	
11	E ₁₁	498.68	487.49
12	E ₁₂	492.74	
13	E ₁₃	471.04	

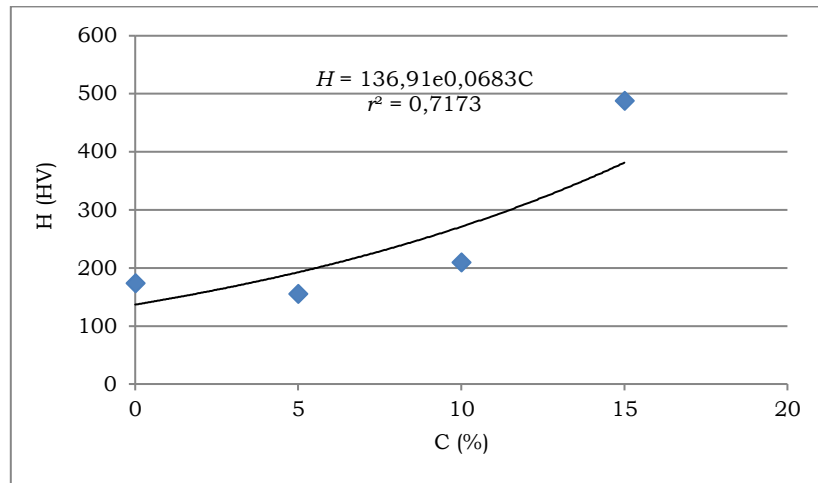


Figure 2. Graph of the Effect of Saltwater Concentration (C) on the Hardness (H) of Medium Carbon Steel

Figure 2 presents the relationship between saltwater concentration (C) and the hardness (H) of medium carbon steel, represented by the exponential regression equation:

$$H = 136.91e^{0.0683C}$$

with a coefficient of determination $r^2 = 0.7173$. The influence of saltwater concentration on steel hardness can be quantified by converting the coefficient of determination into a percentage (Agresti & Franklin, 2013). Based on this calculation, saltwater concentration affects the hardness of medium carbon steel by 71.73%.

Furthermore, the relationship between the response variable (hardness) and the explanatory variable (saltwater concentration) can be evaluated using the correlation coefficient (r). The correlation coefficient is obtained by taking the square root of the coefficient of determination (r^2). Given $r^2 = 0.7173$, the correlation coefficient is calculated as $r = 0.8469$, indicating a strong positive correlation between the independent and dependent variables. It suggests that the data exhibits high validity (Abdullah, 2015). Another crucial mechanical property to consider is the toughness of medium carbon steel. The toughness values obtained in this study are presented in **Table 3**.

Table 3. Toughness Values of the Test Specimens

No	Sample Code	Cross-Sectional Dimensions P (mm) $\pm 10^{-3}$ L (mm) $\pm 10^{-3}$		Cross-Sectional Area (mm ²) $\pm \Delta L$	Energy (J) ± 0.5	Toughness J/mm ² $\pm \Delta T$	Toughness average J/mm ² $\pm \Delta T$
1	A ₂₁	9.535	10.255	97.78 ± 0.10	115	1.176 ± 0.006	1.176 ± 0.006
	B ₂₁	9.590	9.845	94.41 ± 0.10	20	0.2118 ± 0.007	
2	B ₂₂	9.050	10.300	93.22 ± 0.10	17	0.1823 ± 0.005	0.2169 ± 0.006
	B ₂₃	9.130	9.815	89.61 ± 0.09	23	0.2567 ± 0.005	
	C ₂₁	9.015	9.740	87.81 ± 0.09	23	0.2619 ± 0.005	
3	C ₂₂	9.905	10.420	103.21 ± 0.10	40	0.3875 ± 0.004	0.3720 ± 0.004
	C ₂₃	10.100	10.185	102.87 ± 0.10	48	0.4667 ± 0.004	
	D ₂₁	9.320	10.420	97.11 ± 0.10	30	0.3089 ± 0.005	
4	D ₂₂	9.240	9.860	91.11 ± 0.09	34	0.3731 ± 0.005	0.2709 ± 0.005
	D ₂₃	9.680	10.270	99.41 ± 0.10	13	0.1307 ± 0.005	
	E ₂₁	9.685	10.375	100.48 ± 0.10	58	0.5720 ± 0.004	
5	E ₂₂	9.540	10.385	99.07 ± 0.10	29	0.2927 ± 0.005	0.3895 ± 0.005
	E ₂₃	9.600	10.200	98.69 ± 0.10	30	0.3039 ± 0.005	

Table 3 presents the effect of salt solution concentration (C) on the toughness value of the test specimen (T). The highest toughness value was obtained in the sample treated with a 15% salt solution concentration, measuring 0.3895 ± 0.005 J/mm². Meanwhile, the lowest toughness value was observed in the untreated sample (0% salt solution), with a value of 0.2169 ± 0.006 J/mm². To further analyze the relationship between salt solution concentration and the toughness of medium-carbon steel, the data visualization is presented in **Figure 3**.

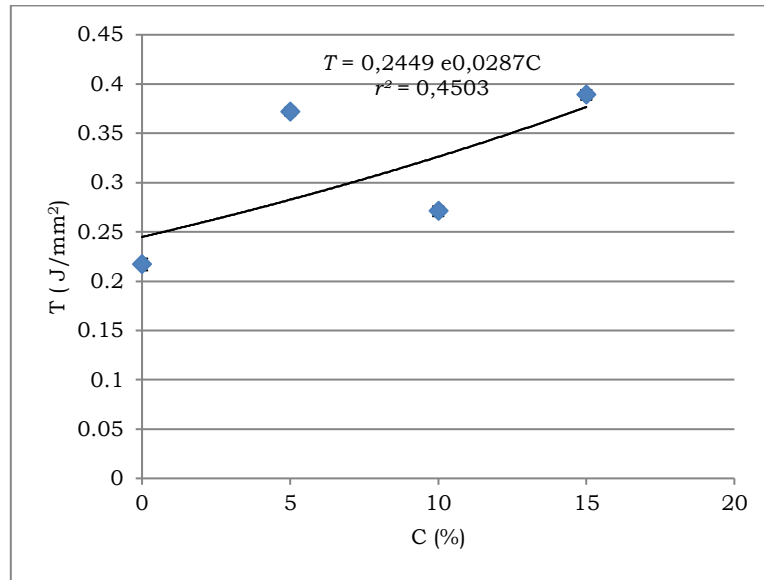


Figure 3. The effect of salt solution concentration (C) on the toughness (T) of medium-carbon steel

The toughness of a material is closely related to its hardness. A specimen with higher hardness tends to be more brittle and prone to fracture. Furthermore, brittle specimens exhibit lower toughness values (Suarsana, 2017). The variation in toughness values does not follow a consistent trend with increasing salt solution concentration. However, the overall trend suggests that toughness (T) increases as the salt solution concentration (C) increases, indicating a positive correlation between these two variables. This observation presents an interesting phenomenon, as the hardness test results indicate that the hardness of medium-carbon steel also increases with higher salt solution concentrations.

In theory, if increasing the salt solution concentration enhances the hardness of a steel specimen, the toughness value should decrease. This assumption is based on the inverse relationship between hardness and toughness in mechanical properties. However, regression analysis reveals that hardness and toughness exhibit a similar positive trend with increasing salt solution concentration. To further understand this behavior, it is necessary to analyze the factors influencing the toughness (T) of the tested specimens.

Three primary factors affect a material's toughness: low temperature, high strain rate (loading speed), and triaxial stress or notch cross-sectional area. In toughness testing, the shape and size of the specimen must be consistent to ensure accurate results (Suarsana, 2017). Referring to **Table 3**, the cross-sectional area of the specimens varies, indicating that the notch dimensions were not uniform. This variation likely contributed to deviations from the expected theoretical toughness values.

The correlation between salt solution concentration and toughness is further illustrated in **Figure 3**. Based on exponential regression analysis, the obtained regression equation is $T = 0.2449 e^{0.0287C}$ with a coefficient of determination $r^2 = 0.4503$, indicating that salt solution concentration influences 45.03% of the toughness variation in medium-carbon steel. The remaining 54.97% (100% - 45.03%) is attributed to other factors. The validity of the results was assessed by calculating the correlation coefficient (r), which yielded a value of 0.6710, suggesting a moderate level of validity (Abdullah, 2015).

One approach to assessing steel's hardness during the hardening process is to analyze the changes in its microstructure. The eutectoid reaction during heat treatment influences carbon steel's microstructure formation and alloys. Microstructural analysis can be performed using metallographic techniques, including optical microscopy. Metallographic analysis visually describes the structure and physical components of metals and their alloys through digital imaging. The following images present the metallographic test results for each sample subjected to different treatments.

Figure 4 presents the microstructure of specimens subjected to various quenching media treatments. The microstructural analysis was conducted using a Nikon Eclipse MA 100 optical microscope with a magnification of 500×. Before microscopic analysis, the samples underwent several preparation stages to ensure optimal image quality, including mounting, polishing, and etching.

Despite optical microscopy's advantages, digital image analysis remains limited in providing a comprehensive quantitative description of microstructural characteristics. Parameters such as grain size, compactness, and other physical attributes are challenging to quantify solely based on optical micrographs.

Figure 4(a) illustrates the microstructure of the untreated specimen, consisting of ferrite (a) and pearlite (P) phases. The ferrite phase appears as the brighter region, whereas pearlite, which contains higher carbon content, appears darker. In contrast, **Figures 4(b)–(e)** depict specimens' microstructures after the hardening process. A significant transformation is observed where the original ferrite and pearlite structures are no longer present. This transformation occurs because, during the hardening process, ferrite and pearlite first transition into austenite and subsequently transform into martensite. Martensitic structures are characterized by a lath or needle-like morphology (Callister & Rethwisch, 2009).

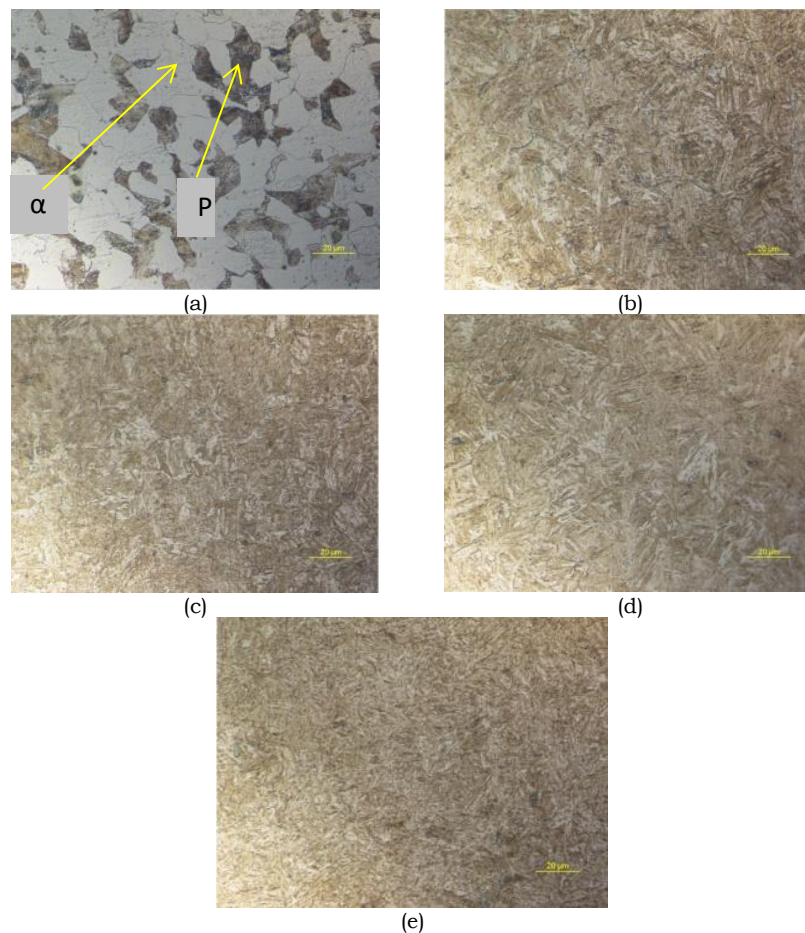


Figure 4. The microstructure of specimens subjected to various quenching media treatments a) untreated b) 0% c) 5% d) 10% e) 15%

The presence of martensitic structures directly influences the hardness of the specimens. One of the key factors affecting hardness is the concentration of the salt solution used as the quenching medium. In **Figure 4(b)**, the martensitic structure appears relatively coarse. As the salt concentration increases, notable changes in the martensitic structure can be observed. In **Figure 4(c)**, the martensitic phase becomes finer compared to **Figure 4(b)**, while **Figure 4(d)** shows an even finer and more uniform structure. However, structural variations are still present, with the specimen's right side exhibiting finer martensite than the left. This non-uniformity may result in hardness variations across different specimen regions. Finally, **Figure 4(e)** exhibits the finest and most homogeneous martensitic structure, indicating the highest hardness among all the tested specimens.

Metallographic testing using an optical microscope inherently has several limitations, particularly in quantitative analysis. This method cannot precisely determine the percentage of various constituent phases in metal alloys, grain size, aspect ratio, and the distribution of grain sizes within the metal. Consequently, performing a comprehensive microstructural analysis solely through optical microscopy remains challenging.

Image processing techniques can mitigate these limitations by applying them to metallographic images obtained from optical microscopy. One practical approach is using *Wolfram Mathematica* for image processing. This method enhances the analysis by providing visual interpretation and delivering quantitative data on phase composition, grain size, aspect ratio, and grain size distribution within the metal. The integration of image processing techniques enables a more accurate and detailed microstructure characterization, facilitating a more robust analysis of metallographic data (Thiando et al., 2018).

4. Conclusions

Based on the experiments conducted to investigate the effect of salt solution concentration on the mechanical properties and microstructure of the specimens, the following conclusions were obtained:

1. The chemical composition test using the *Spark-OES Spectromaxx* method revealed that the test specimen's carbon content was 0.303%, classifying it as medium-carbon steel.
2. The concentration of the salt solution significantly influenced the hardness of medium carbon steel. The highest hardness value recorded was 487.49 ± 10^{-2} HV at a salt solution concentration of 15%.
3. Increasing the salt solution concentration affected the microstructure of the specimens. After the hardening process, the initial ferrite and pearlite phases in the untreated specimens transformed into a martensitic structure.
4. The relationship between salt solution concentration (C) and hardness (H) followed an exponential regression equation:

$$H = 136.91e^{0.0683C}$$

with a coefficient of determination of 71.73%, indicating a strong correlation.

5. The concentration of the salt solution (C) also influenced the toughness (T) of the steel, following an exponential regression equation:

$$T = 0.2449e^{0.0287C}$$

with a coefficient of determination of 45.03%, suggesting that additional factors contribute to toughness.

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