

Enhancing the hardness of low-carbon steel by pack carburizing using shellfish shell powder as a calcium carbonate activator

Junil Adri^{1*}, Durain Parmanoan¹, Refdinal¹, Jasman¹, Bulkia Rahim¹, Meri Marlianna², Lai Chee Sern³

¹ Department of Mechanical Engineering, Faculty of Engineering, Universitas Negeri Padang, **Indonesia**

² Department of Mathematics, Faculty of Mathematics and Natural Sciences Universitas Negeri Padang, **Indonesia**

³ Department of Vocational Education, Faculty of Technical and Vocational Education, Universiti Tun Hussein Onn Malaysia, **Malaysia**

*Corresponding Author: juniladri@ft.unp.ac.id

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Abstract: This study investigates the potential of shellfish shell powder as a natural source of calcium carbonate (CaCO_3) for improving the pack carburizing of AISI 1020 low-carbon steel. AISI 1020 Low-carbon steel is commonly used in engineering applications because of its favorable mechanical properties and machinability; however, its relatively low surface hardness may limit its wear resistance. Carburization was performed at 950 °C for 6 h using three conditions: an untreated control, charcoal only, and a mixture of charcoal shellfish shell powder. Surface characterization was conducted using FE-SEM/EDS, while hardness was evaluated using the Brinell Hardness test. The charcoal-shellfish shell powder specimen achieved the highest average hardness (481.44 BHN), which was significantly higher than those of the charcoal specimen (387.40 BHN) and the control specimen (216.77 BHN) ($p < 0.001$). FE-SEM observations showed that the charcoal-shellfish shell powder specimen exhibited distinct surface morphology, while EDS analysis revealed a higher surface carbon concentration and the presence of calcium, indicating a more effective carburizing environment than the charcoal-only treatment. These findings indicate that the shellfish shell powder effectively enhanced hardness, resulting in the formation of a carbon-enriched surface. The novelty of this study lies in the use of shellfish shell powder derived from shell waste as an alternative natural activator, and in the quantitative validation its effectiveness through statistical analysis, demonstrating its potential as a cost-effective and environmentally sustainable substitute for conventional carburizing activators.

Keywords: hardness; low-carbon steel; pack carburizing; shellfish shell powder

1. Introduction

Materials determine the reliability, performance, and service life of engineering components. Engineers must carefully select materials that meet specific mechanical and industrial requirements. In addition, material selection should consider potential degradation caused by factors such as temperature [1], [2]. The development and modification of materials are crucial for achieving the desired performance under various loading and operating conditions [3], [4]. Low-carbon steel is widely used in various industrial applications because of its low cost, good mechanical properties, and ease of fabrication. However, its relatively low carbon content limits its hardness, making it unsuitable for applications requiring high wear resistance and load-bearing capacity [5]. Surface modification has become an effective approach to enhancing hardness without altering the bulk properties of the material [6].

One of the commonly used surface hardening techniques for low-carbon steel is pack carburizing. In this process, steel is heated in a sealed container filled with a carbon-rich carburizing medium at temperatures ranging from 815 to 1095 °C, producing a hardened case with a depth of approximately 5–60 mils and a surface hardness of 50–60 HRC, depending on the processing conditions [7], [8]. Low-carbon steel is placed in a sealed box or container containing a carbon-rich medium and then heated. During the heating process, carbon diffuses into the steel surface, thereby increasing its hardness. The increased surface hardness enhances the structural stability of the steel, making it more suitable for advanced engineering applications [9].

To accelerate the carburizing process, an activator such as calcium carbonate is commonly added to the carburizing medium. The activator promotes carbon transfer to the steel surface, reducing processing time and increasing the carbon penetration depth. At elevated temperatures, the mobility of carbon atoms increases, allowing them to diffuse from the carburizing medium into the steel surface down the carbon concentration gradient. As carbon atoms penetrate the austenite, the carbon concentration near the surface gradually increases, resulting in the formation of a carbon-enriched case layer [10]. The mechanism involves the reaction between oxygen and carbon inside the sealed container, producing a carbonaceous atmosphere that promotes carbon diffusion into the steel surface during heating. As the carburizing process continues, carbon atoms penetrate deeper into the material, resulting in the formation of a hardened surface layer [11], [12].

After carburization, the specimens underwent quenching. During quenching, this carbon-rich surface transforms into martensite, substantially increasing surface hardness while maintaining a relatively tough core. The diffusion depth and the resulting hardness are strongly influenced by carburizing temperature, holding time, and the carbon potential of the carburizing medium [13]. The pack carburizing process offers significant advantages, particularly in improving the surface hardness of steel. Furthermore, the use of alternative carbon-based materials provides an effective approach to waste utilization while promoting environmental sustainability [14]. Organic materials have also been investigated as biomaterials containing CaCO_3 , which performs the same function as an activator while being more environmentally friendly and cost-effective [15], [16], [17].

Pack carburizing presents several challenges, including prolonged processing time, non-uniform atmospheric conditions within the carburizing container, oxidation during heat treatment, and dimensional distortion of thin or slender components [18]. Heat transfer plays a fundamental role in the pack carburizing process by supplying the thermal energy required to activate carbon transfer reactions and promote atomic diffusion [19]. As the temperature increases, the diffusion coefficient of carbon also increases, allowing carbon atoms to penetrate more effectively into the steel surface. One of the most influential parameters in improving the hardening process during carburization is the carburizing temperature. The application of heat promotes the diffusion of carbon from the carbonaceous medium into the steel, resulting in the formation of a carbon-enriched case with improved hardness and wear resistance. Carbon diffusion is therefore closely associated with the mechanical performance and surface durability of the treated material [20].

This study investigates the influence of shellfish shell powder on the pack carburizing of low-carbon steel and evaluates its potential as a natural activator for enhancing carburization performance. The effect of incorporating shellfish shell powder into a charcoal-based carburizing medium was evaluated through pairwise comparisons of the resulting hardness values among the treatment groups. The use of biomass-derived materials as alternative carburizing media and activators has attracted increasing attention because of their availability, relatively low cost, and potential for waste valorization. In this context, charcoal serves as a carbon-rich material derived from plant biomass, while shellfish shell powder provides a calcium carbonate (CaCO_3)-rich material derived from seafood shell waste. Recent studies have reported the potential of both materials as alternative constituents in surface hardening and carburizing processes [21].

Shellfish are widely cultivated and consumed in many coastal regions, generating substantial quantities of shell waste as a by-product of the seafood industry. Due to their relatively high hardness and low biodegradability, discarded shellfish shells are difficult to recycle and can accumulate as solid waste, raising environmental concerns [22]. However, shellfish shell powder contains approximately 95 wt.% calcium carbonate, highlighting its potential for valorization as a natural activator in pack carburizing processes [23]. Compared with conventional chemical activators, shellfish shell powder derived from seafood waste offers potential advantages, including low cost, abundant availability, and reduced reliance on commercially manufactured chemical additives. The use of shellfish shell powder as a carburizing additive also provides an opportunity to convert an underutilized waste stream into a functional material for surface hardening applications, thereby supporting waste valorization and circular economy practices.

Although CaCO_3 -rich materials, such as eggshells and oyster shells, have been investigated as activators in pack carburizing, the use of shellfish shell powder as a waste-derived has limited attention, particularly for AISI 1020 low-carbon steel. Previous studies have primarily focused on charcoal-based carburizing media or other CaCO_3 -rich waste materials, with limited comparative evaluation of charcoal and shell-derived activators under identical carburizing conditions. In particular, the effectiveness of shellfish shell powder as a natural activator in the pack carburizing of AISI 1020 steel has not been sufficiently evaluated through systematic hardness measurements and statistical validation.

To address this gap, the present study investigates the effectiveness of shellfish shell powder as a natural activator in the pack carburizing of AISI 1020 steel and compares its hardening performance with that of conventional charcoal under identical processing conditions. Charcoal serves as the primary carbon source, while shellfish shell powder provides a CaCO_3 -rich activator that may promote the formation of reactive carbon-bearing gases and facilitate carbon transfer to the steel surface during carburization [24]. The combined use of these biomaterials is hypothesized to enhance carbon transfer into the steel surface and subsequent carbon diffusion, resulting in increased surface hardness after heat treatment [25]. The effects of the carburizing treatments are evaluated through Brinell hardness measurements, FE-SEM/EDS characterization, and statistical analysis to assess the resulting differences in surface properties.

2. Material and methods

2.1 Material

In this study, AISI 1020 steel was selected as the substrate material because of its widespread use in engineering applications. AISI 1020 is classified as a plain carbon steel containing approximately 0.20 wt.%. Its relatively low carbon content results in comparatively low surface hardness and wear resistance, although the steel exhibits favorable ductility, weldability, and machinability. These properties make AISI 1020 suitable for various light-duty engineering components, including shafts, gears, and fasteners; however, its limited surface hardness and wear resistance can restrict its performance under demanding service conditions [26], [27]. Therefore, surface hardening treatments such as pack carburizing can improve surface hardness while retaining the desirable characteristics of the bulk material. The chemical composition of AISI 1020 used in this study is presented in Table 1.

Table 1. Chemical Composition of AISI 1020 Steel (wt%)

Elements	C	Si	Mn	P	S	Ni	Al	Fe
Compositions (wt%)	0.199	0.212	0.52	0.0179	0.009	0.0155	0.0407	98.98

AISI 1020 steel was machined into cylindrical specimens with a diameter of 25.4 mm and a thickness of 12 mm. The specimens were prepared to the specified dimensions to ensure geometric uniformity and consistency throughout the pack carburizing and subsequent hardness testing processes.

The preparation of the carburizing media involved processing the shellfish shells and charcoal prior to the pack carburizing treatment. The shellfish shells were thoroughly washed with clean water to remove adhering impurities, organic residues, and surface contaminants. The cleaned shells were then oven-dried at 120 °C for 4 h until a constant weight was achieved. After drying, the shells were crushed and ground into a fine powder using a grinding machine, followed by sieving to obtain a uniform particle size. The prepared shell powder was subsequently stored in an airtight container to minimize moisture absorption and contamination prior to use. This preparation procedure was intended to ensure a relatively uniform particle size and improve the effectiveness of the shell powder as a calcium carbonate (CaCO_3)-rich natural activator during the pack carburizing process [28]. Meanwhile, charcoal was produced by carbonizing coconut shells under controlled conditions until complete carbonization was achieved. The resulting charcoal was then crushed and ground into a fine powder before being mixed with shellfish shell powder at the predetermined compositions. The prepared carburizing media were then placed in sealed carburizing boxes with the AISI 1020 steel specimens, ensuring that each specimen was completely surrounded by the respective carburizing medium during heat treatment.

To evaluate the effect of the different carburizing media, the specimens were divided into three treatment groups: control, charcoal, and charcoal–shellfish shell powder. The control specimen was not subjected to the pack carburizing process. The mass ratios of the carburizing materials were determined according to the predetermined composition of each treatment. The charcoal-treated specimen was carburized using 20 g of charcoal, while the specimen treated with the charcoal–shellfish shell powder mixture was carburized using 17 g of charcoal and 3 g of shellfish shell powder (85:15 wt.%). Figure 1 shows the carburizing media after weighing and portioning prior to furnace treatment.



Figure 1. Carburizing media prepared according to the specified material ratios; charcoal and shellfish shell powder powder–charcoal mixture in the carburizing boxes

2.2 Pack carburizing procedure

During the pack carburizing process, the carburizing media with the predetermined mass ratios were placed in two carburizing boxes, and the low-carbon steel specimens were subsequently positioned within the media, as shown in Figure 1. The carburizing boxes were then tightly sealed to protect the specimens from direct exposure to the furnace atmosphere during the heating process. The specimens were heated at 950 °C for 6 h. After the heat treatment, the specimens were immediately quenched in water at room temperature to promote surface hardening. Finally, Brinell hardness testing was performed at three different locations on each specimen to evaluate the influence of the shellfish shell powder activator on the hardness of AISI 1020 steel. Figure 2 presents

the sequential stages of the pack carburizing process, including material preparation, allocation of the specified material ratios for each specimen, furnace treatment, quenching, and hardness measurement.

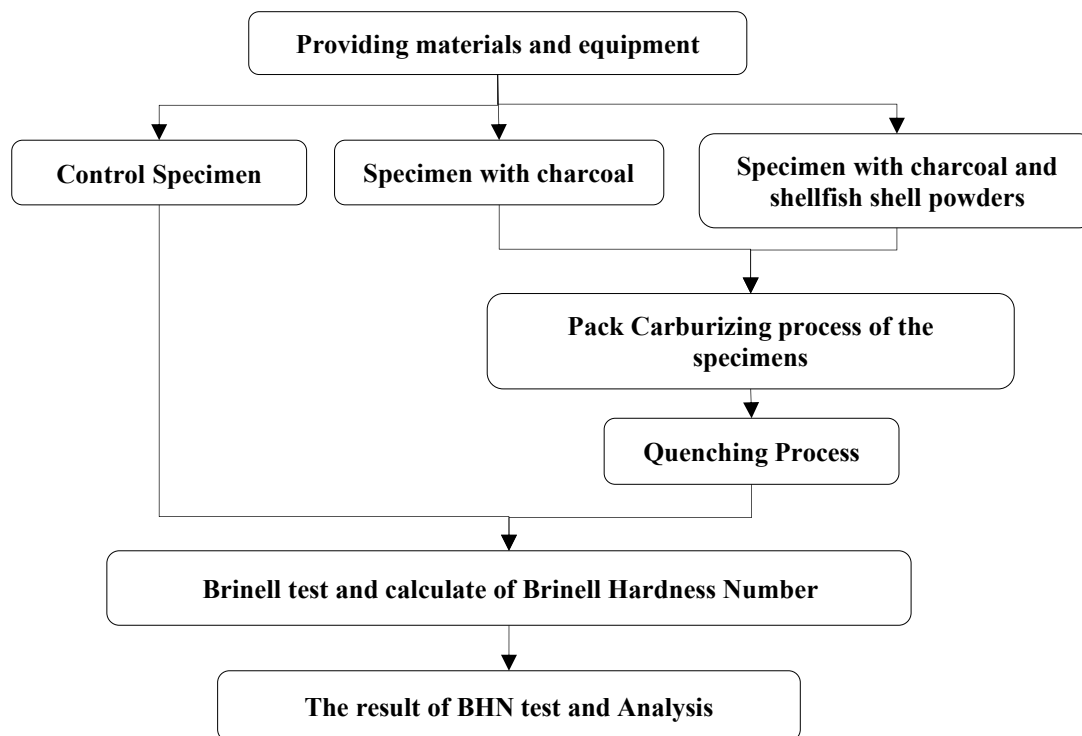


Figure 2. Pack carburizing process diagram of specimen preparation and BHN analysis

2.3 Quenching process

After completion of the pack carburizing process, all specimens were immediately quenched in water at room temperature (25 °C) for 1 h. Following quenching, the specimens were cleaned by grinding and polishing to remove residual carburizing media, oxide scale, and surface contaminants, thereby providing a smooth surface for subsequent hardness testing.

2.4 Determination of brinell hardness number

After completion of the quenching process, each specimen was subjected to Brinell hardness testing at three different positions on the specimen surface to obtain the corresponding dial readings. The dial readings were then substituted into the Brinell Hardness Number (BHN) equation to determine the hardness values of the low-carbon steel specimens. Since the Brinell method was employed in this study, the hardness values were calculated using the following equation. Since this research uses the Brinell method, then the hardness numbers were determined using the following formula BHN [1], [2]. The Brinell hardness test was performed by pressing a 10 mm diameter cemented carbide ball indenter, with a maximum diameter tolerance of ± 0.005 mm, onto the surface of the AISI 1020 steel specimens. A load of 187.5 kgf was applied and maintained for a dwell time of 10–15 s, allowing the indenter to penetrate the specimen surface and produce a permanent indentation. The diameter of the resulting indentation was then measured, and the corresponding dial reading was recorded at each testing position. The recorded dial readings were subsequently substituted into the Brinell Hardness Number (BHN) equation, as shown in Equation (1), to determine the hardness values of the AISI 1020 steel specimens in accordance with ASTM E10.

$$BHN = \frac{2.P}{\pi.D(D - \sqrt{D^2 - d^2})} \quad (1)$$

2.5 FE-SEM and EDS characterization

Following the pack carburizing and quenching processes, the steel specimens were characterized using field-emission scanning electron microscopy (FE-SEM) coupled with energy-dispersive X-ray spectroscopy (EDS) (*Thermo Fisher Scientific Quattro S*) to investigate the surface morphology and elemental composition of the carburized layer. FE-SEM was used to examine morphological changes associated with the carburizing treatment, while EDS was used to determine the elemental composition and distribution on the specimen surface, with particular emphasis on carbon (C), oxygen (O), iron (Fe), and calcium (Ca). Carbon was analyzed as an indicator of changes in surface composition resulting from carburization, whereas calcium was evaluated to assess the contribution of shellfish shell powder as an activator during the pack carburizing process.

Prior to characterization, the carburized specimens were mechanically sectioned into smaller pieces to meet the dimensional requirements of the FE-SEM specimen holder. Each specimen was prepared to an approximate dimension of 20 × 20 mm while preserving the region of interest for subsequent morphological and elemental analyses. Figure 3 shows the control, charcoal-carburized, and charcoal-shellfish shell powder-carburized specimens after Brinell hardness testing. The specimens were then further sectioned to obtain dimensions suitable for mounting on the Fischer instrument for subsequent surface characterization.

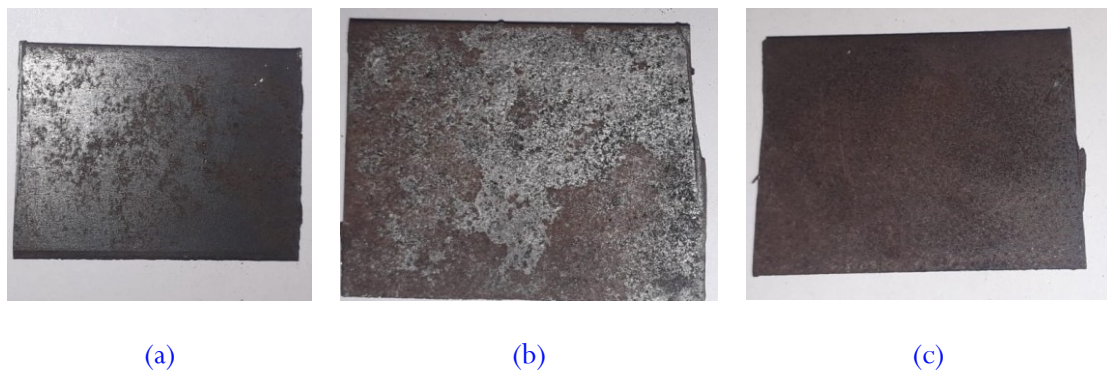


Figure 3. FE-SEM images of the tested specimens: (a) control specimen, (b) shellfish shell powder-charcoal specimen, and (c) charcoal specimen

The sectioned specimens were mounted on aluminum SEM specimen stubs using conductive carbon tape before FE-SEM examination. Surface morphology was examined at magnifications ranging from 400× to 10,000× under conductive operating conditions. Simultaneously, EDS elemental mapping and quantitative analyses were performed in selected observation areas to evaluate the elemental composition and distribution on the specimen surfaces. The FE-SEM and EDS results were subsequently compared among the experimental groups based on surface morphology, carbon content, and elemental distribution to assess the influence of charcoal and charcoal-shellfish shell powder mixtures as carburizing media on the surface characteristics of the AISI 1020 steel.

2.6 Statistical analysis for evaluating the effectiveness of the result

2.6.1 Statistical analysis ANOVA one way

One-way analysis of variance (ANOVA) was used to evaluate differences in the mean hardness values among the treatment groups. The analysis was conducted to determine whether the observed

variation in hardness was statistically significant and could be attributed to differences in the carburizing treatments rather than random variation. A statistically significant ANOVA result indicated that at least one treatment group differed in mean hardness from the others. When a significant difference was detected, Tukey's Honest Significant Difference (HSD) post-hoc test was subsequently performed to identify the specific pairs of treatment groups that differed significantly [29].

Hypothesis formulation for one-way ANOVA analysis

$$\begin{aligned} H_0: \mu_1 &= \mu_2 = \dots = \mu_k \\ H_1: \mu_1 &\neq \mu_2 \neq \dots \neq \mu_k \\ \alpha &= 0.05 \end{aligned} \quad (2)$$

If $p < 0.05$, there is a statistically significant difference among the group means

If $p \geq 0.05$, there is no statistically significant difference among the group means

$$d_{fb} = k - 1 \quad (3)$$

$$d_{fw} = N - k \quad (4)$$

$$SSB = \sum_{i=1}^k n_i (\bar{x}_i - \bar{x})^2 \quad (5)$$

$$SSW = \sum_{i=1}^k \sum_{j=1}^{n_i} (x_{ij} - \bar{x}_i)^2 \quad (6)$$

$$SST = SSB + SSW \quad (7)$$

$$MSB = \frac{SSB}{d_{fb}} \quad (8)$$

$$MSW = \frac{SSW}{d_{fw}} \quad (9)$$

$$F = \frac{MSB}{MSW} \quad (10)$$

2.6.2 Statistical analysis using Tukey's HSD test

Tukey's Honest Significant Difference (HSD) test was employed to compare the hardness values among the different treatment groups. Pairwise comparisons were performed to determine whether statistically significant differences existed between the treatment groups. The specimens were compared in the following pairs:

- A. Control specimen vs. charcoal-carburized specimen
- B. Control specimen vs. charcoal-shellfish shell powder-carburized specimen
- C. Charcoal-carburized specimen vs. charcoal-shellfish shell powder-carburized specimen

The pairwise comparisons identified significant differences in hardness between the treatment groups and provided a basis for evaluating the effect of incorporating shellfish shell powder into the charcoal carburizing medium. In particular, the comparison between the charcoal-carburized and charcoal-shellfish shell powder-carburized specimens was used to assess whether the addition of shellfish shell powder resulted in a significant improvement in the hardness of the carburized steel [34, 35].

$$|\underline{x}_i - \underline{x}_j| > HSD \quad (11)$$

True: There is a statistically significant difference among the treatment group means.

False: There is no statistically significant difference among the treatment group means.

$$q = \frac{|\underline{x}_i - \underline{x}_j|}{SE}, \text{ Studentized Range Distribution} \quad (12)$$

$$q_{kritis} = q_{\alpha}(k, dfw) \quad (13)$$

$$HSD = q_{\alpha}(k, dfw) \sqrt{\frac{MSW}{n}} \quad (14)$$

3. Results and discussion

3.1 Hardness values between specimens with and without shellfish shell powders

After obtaining the dial readings from the three measurement points on each specimen, the recorded values were substituted into the Brinell Hardness Number (BHN) equation to calculate the corresponding hardness values. The BHN Analysis was performed by calculating the Brinell Hardness Number (BHN) for each hardness measurement using the Brinell hardness equation based on the applied load, indenter diameter, and measured indentation diameter. The BHN Mean was then calculated as the arithmetic mean of the individual BHN values obtained from the repeated measurements for each treatment group. The BHN Analysis results and corresponding BHN Mean values are presented in Table 2.

Table 2 presents the experimental hardness data obtained after the pack carburizing process, expressed as the Brinell Hardness Number (BHN). The BHN values were calculated from the dial readings obtained at the three measurement points on each specimen using the Brinell hardness equation. The control specimen exhibited the lowest mean BHN of 216.72, consistent with the experimental design, as it was not subjected to the carburizing treatment. In contrast, the charcoal-treated specimens exhibited a higher mean BHN of 387.40, indicating an increase in hardness following carburization. The specimens treated with a combination of charcoal and shellfish shell powder showed the highest mean BHN, reaching 481.44.

Table 2. The number of dials and Brinell hardness numbers for each specimen and case depth point

Specimens	Hardness Case Depth (points)						BHN Mean	BHN Analysis
	1		2		3			
	Dial	BHN	Dial	BHN	Dial	BHN		
1	57	220	56	215	55	210	216.77	227.5
2	56	215	56	215	56	215		
3	57	220	56	215	57	220		
4	55	210	57	220	55	210		
5	57	220	57	220	56	215		
6	57	220	58	225	56	215		
1	77	328	79	409	80	420	387.4	398.08
2	80	424	79	409	78	395		
3	78	395	77	382	76	370		
4	78	395	76	370	76	370		
5	77	382	77	382	77	382		
6	78	395	78	395	76	370		
1	83	463	85	491	85	491	481.44	503.35
2	85	491	84	476	85	491		
3	86	506	84	476	86	506		
4	84	476	85	491	83	463		
5	83	463	85	491	84	476		
6	83	463	84	476	84	476		

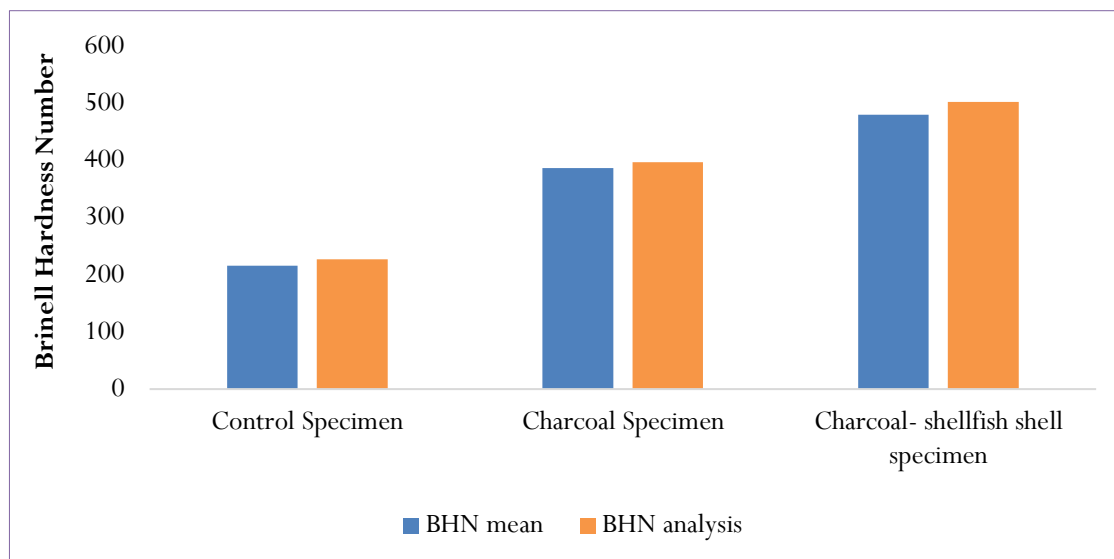


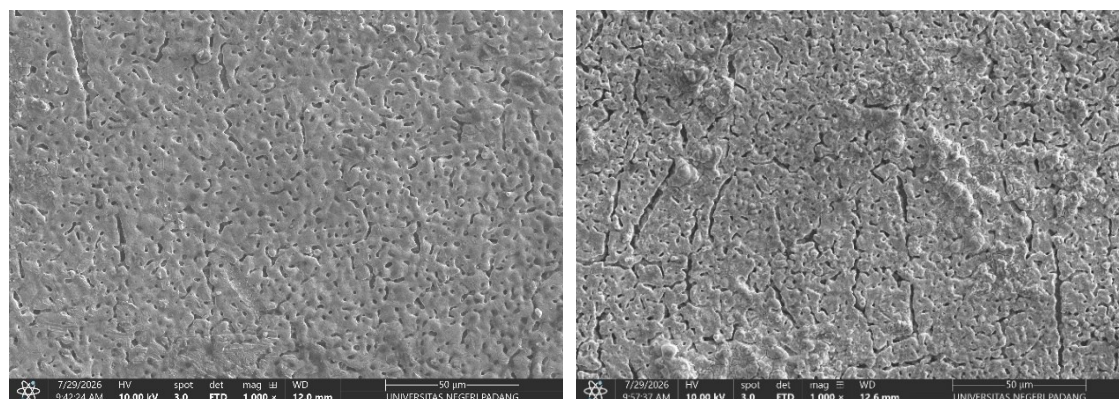
Figure 4. Comparison of Brinell hardness number mean and analysis

Microsoft Excel was used to process the experimental data and visualize the distribution of the BHN values. The calculated hardness values were subsequently compared with the corresponding values obtained from the data analysis. As shown in the diagram, no substantial differences were observed between the mean BHN values obtained from the Brinell hardness equation and those obtained from

the data analysis. The close agreement between the two approaches indicates consistency in the calculated hardness values and supports the reliability of the experimental measurements and subsequent data processing. Figure 4 presents the BHN values obtained from the individual measurement points along with the corresponding mean BHN. The comparison provides a visual representation of the variation in hardness among the control, charcoal-treated, and charcoal-shellfish shell powder-treated specimens.

3.2 Surface morphology of carburized steel surfaces

Following the hardness measurements, FE-SEM characterization was conducted to examine the surface morphology of the specimens after the pack carburizing treatment. The FE-SEM observations examined the surface features of the specimens treated with charcoal and charcoal-shellfish shell powder mixtures to identify morphological changes associated with the different carburizing media. Representative FE-SEM micrographs of the treated specimens are presented in Figure 5. The FE-SEM characterization revealed distinct surface morphological characteristics among the specimens following the carburizing process. Figure 5 presents the FE-SEM micrographs of the specimens treated with (a) charcoal and (b) a charcoal-shellfish shell powder mixture at different magnifications. The micrographs provide a comparative view of the surface morphology resulting from the two carburizing media.



(a)

(b)

Figure 5. FE-SEM images showing the surface morphology of steel carburized using (a) charcoal-shellfish shell powder (b) charcoal as carburizing media at a magnification of 1000×

The effect of incorporating shellfish shell powder as an activator into the pack carburizing medium was reflected in changes in the surface morphology and hardness of the steel specimens. As shown in Figure 5(a), the specimen treated with a charcoal-shellfish shell powder mixture exhibited a relatively dense and uniform surface morphology, with a more homogeneous surface distribution and minimal visible microporosity. The presence of shellfish shell powder, which is rich in calcium carbonate (CaCO_3), may have promoted the generation of reactive carbon-bearing gases during the carburizing process, thereby facilitating carbon transfer to the steel surface. The more uniform surface morphology, together with the increased hardness observed for this treatment, suggests more effective carburization and carbon incorporation into the near-surface region. This behavior may be associated with the formation of harder transformation products, such as martensitic structures and iron carbide (Fe_3C), although confirmation of these phases would require complementary microstructural or phase characterization.

In contrast, the specimen treated with charcoal alone, as shown in Figure 5(b), exhibited a comparatively rougher and less uniform surface morphology, with several visible micro-cracks and surface irregularities. These morphological features may be associated with less uniform

carburization and the thermal effects experienced during the treatment in the absence of the shellfish shell powder activator. The presence of surface micro-cracks can also act as local stress-concentration sites and potentially compromise the mechanical integrity of the carburized surface. Consistent with the morphological observations, the charcoal-treated specimen exhibited a lower mean hardness than the specimen treated with the charcoal–shellfish shell powder mixture. Overall, the addition of shellfish shell powder to the charcoal carburizing medium was associated with a denser and more uniform surface morphology and a significant increase in surface hardness, indicating its potential effectiveness as an activator in the pack carburizing of AISI 1020 steel.

3.3 Surface elemental composition analysis

The elemental composition and distribution on the surface of the carburized AISI 1020 steel specimens were analyzed using Energy-Dispersive X-ray Spectroscopy (EDS). A comparison of the normalized mass concentrations of the major detected elements, including carbon (C), oxygen (O), calcium (Ca), and iron (Fe), is presented in Figure 6. Figure 6(a) shows the EDS spectrum of the specimen treated with the charcoal–shellfish shell powder mixture, while Figure 6(b) presents the EDS spectrum of the specimen treated with charcoal.

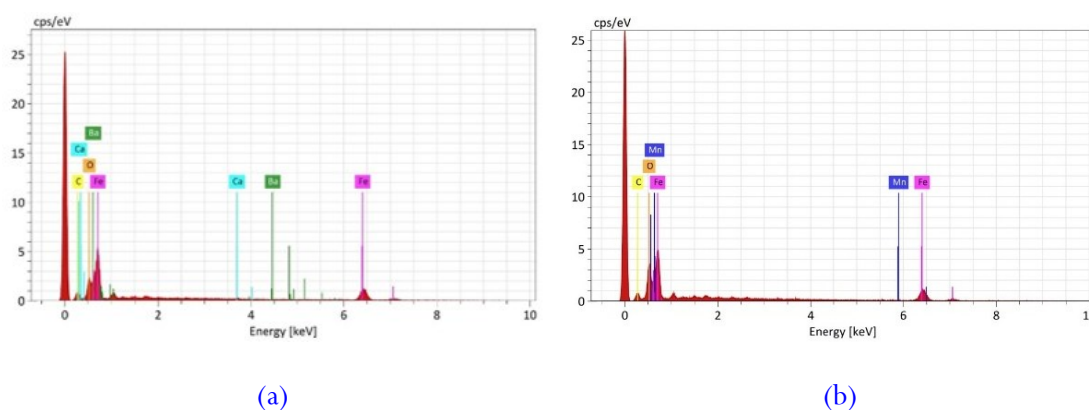


Figure 6. EDS results of carburized AISI 1020 steel: (a) charcoal–shellfish shell powder and (b) charcoal

As shown in Figure 6(a), the specimen treated with the charcoal–shellfish shell powder mixture exhibited a prominent carbon (C) peak, accompanied by a distinct calcium (Ca) peak. The presence of Ca on the specimen surface is consistent with the incorporation or reaction of calcium-containing compounds originating from the shellfish shell powder during the carburizing process. Given that shellfish shell powder is primarily composed of calcium carbonate (CaCO_3), its thermal decomposition may contribute to the generation of CO_2 , which can subsequently react with carbonaceous material in the carburizing medium to promote the formation of CO. This process may enhance the availability of reactive carbon species at the steel surface and facilitate carbon transfer during carburization.

In contrast, the EDS spectrum of the charcoal in Figure 6(b) was dominated by Fe, C, O, and Mn, with no detectable Ca peak. The absence of Ca is consistent with the absence of shellfish shell powder in the carburizing medium. The comparatively lower carbon content observed in the charcoal-treated specimen suggests a lower degree of carbon incorporation at the analyzed surface when compared with the charcoal–shellfish shell specimen. This observation is consistent with the denser surface morphology and higher hardness measured for the specimen treated with the charcoal–shellfish shell mixture. Overall, the EDS results provide compositional evidence supporting the role of shellfish shell powder as an activator in the pack carburizing process and its association with enhanced carbon incorporation at the steel surface.

3.4 Statistical analysis of hardness data

Before comparing the hardness values among the specimens, the assumptions for parametric analysis were evaluated. The Shapiro–Wilk test assessed the normality of the BHN data for each treatment group, while Levene’s test assessed homogeneity of variances among groups. The descriptive statistics and results of the assumption tests are presented in Table 3.

Table 3. Descriptive statistics and results of normality and homogeneity tests for BHN values

Specimens	N	Mean	SD	SE	Shapiro-Wilk (W)	Shapiro-Wilk (p-value)	Levene's Test (F)	Levene's Test (p-value)
Control	6	217	2.59	1.06	0.938	0.648	1.53	0.248
Charcoal	6	387	11.09	4.53	0.804	0.067		
Charcoal–shellfish shell	6	482	8.50	3.47	0.898	0.372		

Note: Levene’s test degrees of freedom: $df1 = 2$, $df2 = 15$

All treatment groups were normally distributed according to the Shapiro–Wilk test ($p > 0.05$), and Levene's test confirmed homogeneity of variances ($F = 1.53$, $p = 0.248$). These results indicate that the assumptions for subsequent one-way ANOVA were met. Since the assumptions of normality and homogeneity of variance were met, a one-way analysis of variance (ANOVA) was subsequently performed to determine whether significant differences in hardness existed among the treatment groups. The results of the one-way ANOVA are presented in Table 4.

Table 4. One-way ANNOVA (Fisher’s)

	F	df1	df2	P
Brinell Hardness Number	1610	2	15	<0.001

The one-way ANOVA revealed a statistically significant effect of the carburizing medium on the Brinell Hardness Number (BHN), ($F(2, 15) = 1610$, $p < 0.001$). This result indicates that the mean hardness differed significantly among the three specimens. Therefore, Tukey’s Honest Significant Difference (HSD) was subsequently performed to identify the specific pairs of treatment groups that differed significantly in hardness. The results of the Tukey’s Honest Significant Difference (HSD) post-hoc test are presented in Table 5.

Table 6. Tukey's Honestly Significant Difference (HSD) post hoc test

		Control	Charcoal	Charcoal & Shellfish
Control	Mean difference	—	-171***	-265.2***
	t-value	—	-36.1	-56.0
	Df	—	15.0	15.0
Charcoal	Mean difference	—	—	-94.3***
	t-value	—	—	-19.9
	Df	—	—	15.0
Charcoal & Shellfish	Mean difference	—	—	—
	t-value	—	—	—
	Df	—	—	—

Note. * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$

Table 5 displays the comparisons obtained from the Tukey's HSD test. The negative values of the mean difference indicate that the first treatment group listed in each comparison exhibited a lower mean hardness than the second group. In contrast, positive values indicate a higher mean hardness in the first group. The magnitude of the t-value reflects the extent to which the difference between two treatment groups exceeds the within-group variation, while the significance level (* $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$) indicates the statistical significance of each comparison. Therefore, larger absolute t-values accompanied by significant p-values provide stronger evidence that the observed differences in hardness among the treatment groups were not due to random variation.

3.5 Pairwise comparisons of treatment group

Following the significant one-way ANOVA result, Tukey's Honest Significant Difference (HSD) post-hoc test was performed to determine the differences between individual treatment groups. Pairwise comparisons were conducted based on the mean BHN values of the three groups to identify the magnitude and statistical significance of the differences in hardness between treatments. The results of the pairwise comparisons, including the mean differences and corresponding significance levels, are presented in Table 6.

Table 6. Differences of Couple Group Specimens

Pairwise mean differences	Range
Specimen control vs. Specimen with Charcoal	-171
Specimen control vs. Specimen with Charcoal and Shellfish shell powders	-265.2
Specimen with Charcoal vs. Specimen with Charcoal and Shellfish shell powder	-94.3

The Tukey HSD analysis further demonstrated that the charcoal-shellfish shell powder treatment produced significantly higher hardness than both the control group and the charcoal-only treatment. Compared with the control specimen, the charcoal-shellfish shell powder treatment exhibited a mean hardness difference of approximately 265.2 BHN, whereas the charcoal-only treatment showed a difference of approximately 171.0 BHN. Because the addition of shellfish shell powder was the primary difference between the two carburizing treatments, the greater increase in hardness observed for the charcoal-shellfish shell powder treatment suggests that the shellfish shell powder enhanced the effectiveness of the carburizing process.

3.6 Effect of shellfish shell powder on carbon transfer during pack carburizing

Pairwise comparisons were performed to evaluate the effectiveness of shellfish shell powder, which is rich in calcium carbonate (CaCO_3), as a natural activator in promoting carbon transfer and diffusion into the steel surface during pack carburizing. The boxplot presented in Figure 7 illustrates the distribution of the Brinell Hardness Number (BHN) values for each treatment group. In addition to comparing the mean hardness values, the boxplot provides a visual representation of the spread and variability of the hardness measurements within each group, allowing the consistency of the hardness response across the specimens to be assessed.

The control group exhibits the lowest median Brinell Hardness Numbers among the three specimens and overall data distribution. This indicates that the untreated AISI 1020 steel retained its original mechanical properties without the influence of the pack carburizing process. The limited spread of hardness numbers, ranging from approximately 210 to 225 BHN, demonstrates low variability and high measurement consistency within the control group. Such uniformity suggests that the initial material condition was relatively homogeneous before any heat treatment was applied, providing a reliable baseline for evaluating the effectiveness of the subsequent carburizing treatments.

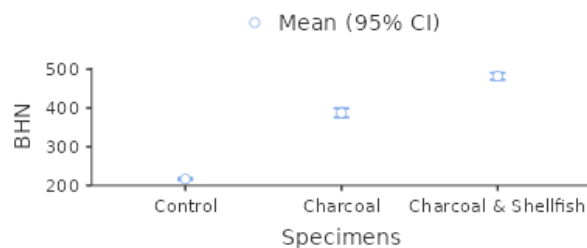


Figure 8. Boxplot of mean distributions for Brinell Hardness Numbers in each specimen

In contrast, both carburized specimens exhibited considerably higher hardness numbers, indicating that the heat treatment and carburizing medium significantly modified the surface properties of AISI 1020 steel. The increase in hardness observed after the pack carburizing process can be explained by the combined effects of heat transfer and carbon diffusion. During heating at 950 °C, thermal energy increases the mobility of carbon atoms in the carburizing medium, allowing carbon to diffuse more effectively toward the surface of the AISI 1020 steel. Maintaining the carburizing temperature for 6 h provides sufficient time for carbon to penetrate the surface layer, resulting in a higher carbon concentration near the steel surface. Consequently, the carbon-enriched surface exhibits greater resistance to indentation, which is reflected by the higher Brinell hardness numbers measured in the treated specimens. These results indicate that the carburizing temperature, holding time, and carburizing medium collectively influence the effectiveness of carbon diffusion and, therefore, the hardness improvement of low-carbon steel.

Previous studies focusing on different temperature treatments state that the concentration of carbon diffused to the surface is strongly influenced by the temperature during the pack carburizing process [30], [31]. This is similar to research that examined the role of carbon in determining hardness depth [32], [33], [34]. This study used carbon-containing additives to observe differences in depth during testing. This can be observed from the explanation that the carbon content in certain materials varies, resulting in different diffusion effects. Similarly, some natural materials such as oyster shells and animal bones used in this study show differences in hardness results. The variation in the depth of the steel material indicates that carbon has an effect on low-carbon steel, but the hardness numbers can vary [35].

Compared with the charcoal, the charcoal and shellfish shell powder exhibited higher and more consistent hardness values. This improvement suggests that the addition of shellfish shell powder enhanced the carburizing atmosphere by providing an activator, thereby promoting more effective carbon transfer during the pack carburizing process. Consequently, a more uniform carbon-enriched surface was produced, resulting in greater resistance to indentation as reflected in the Brinell hardness measurements. The statistical results suggest that the shellfish shell powder contributed to a more effective carburizing process. From an engineering material perspective, $CaCO_3$ acts as a natural activator by promoting the formation of a carbon-rich atmosphere during heating, thereby facilitating carbon transfer to the steel surface. Consequently, the higher hardness observed in the charcoal–shellfish shell powder treatment is consistent with enhanced carbon diffusion during pack carburizing.

4. Conclusion

The addition of shellfish shell powder as a carbon diffusion activator in this study effectively increased the hardness of low-carbon steel. The results of the statistical and Brinell hardness analyses showed a consistent trend, particularly for the charcoal–shellfish shell powder specimen, which exhibited a mean hardness of 481.44 BHN and a BHN analysis value of 503.35. Furthermore, the

selected carburizing temperature of 950 °C effectively promoted carbon diffusion into the steel surface. The heating duration also contributed to the carburization process by maintaining the required temperature within the carburizing container. Therefore, it can be concluded that the treatment conditions applied to the specimens were objectively evaluated based on both statistical and engineering analyses.

The FE-SEM and EDS analyses further showed that the addition of shellfish shell powder was associated with changes in the surface characteristics of the carburized AISI 1020 steel. The charcoal–shellfish shell powder specimen exhibited distinct surface morphology, higher surface carbon content, and detectable calcium compared with the charcoal-only specimen, indicating the contribution of shellfish shell powder as an activator during the pack carburizing process. These findings support the hardness results by suggesting that the modified carburizing environment promoted carbon enrichment at the steel surface, contributing to the increased surface hardness observed after heat treatment.

Furthermore, future research could investigate the development and use of additional biomaterials as environmentally friendly alternatives to conventional chemical activators for improving the surface hardness of steel. Optimizing the use of these biomaterials could contribute to more sustainable production processes and waste reduction strategies. Research exploring alternative materials for the carburization process could have a promising impact. By investigating new materials that provide similar properties with a lower environmental impact, researchers may help the steel industry reduce carbon emissions, decrease reliance on fossil fuels, and minimize its overall environmental impact.

Author's declaration

Author contribution

Junil Adri: Conceptualization, Methodology, Investigation, Formal analysis, Data curation, Writing original draft, Supervision. **Durain Parmanoan:** Investigation, Data curation, Validation, Writing original draft. **Refdinal:** Methodology, Validation, Supervision, Writing – review & editing. **Jasman:** Formal analysis, Validation, Writing – review & editing. **Bulkia Rahim:** Data curation, Visualization, Writing – review & editing. **Meri Marlianna:** Formal analysis, Validation, Writing – review & editing. **Lai Chee Sern:** Supervision, Validation, Writing – review & editing.

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Data availability

Data supporting the findings of this study are available within the article. Additional data related to the experimental measurements and analysis are available from the authors in the form of test materials and research notes.

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Conflict of interest

The authors declare that they have no known financial or personal relationships that could have appeared to influence the work reported in this paper.

Ethical clearance

Not applicable.

AI statement

ChatGPT was utilized to assist with grammatical and linguistic improvements to the manuscript. The revised manuscript was then proofread and verified by a qualified English language expert. The authors retained full responsibility for the content and accuracy of the manuscript and conducted a final review before approving the version submitted for publication.

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Nomenclature

1	Diameter of the ball	d_{fb}	Degree of free between group
F	Test force	d_{fw}	Degree of free within group
d	Mean diameter of the indentation	SSB	Sum of square between group
h	Depth of the indentation	SSW	Sum of square within group
k	The number of different groups	SST	Total sum of squares
n_i	The size of the i group	MSB	Mean of square between group
N	Total sample $\sum n_j$	MSW	Mean of square within group
x_{ij}	Data of $-j$ in group- i	F	Determine of F ratio
$\underline{x_i}$	Mean of group $-i$	q critics	Measuring of Standard Error Tukey
$\underline{x}$	The average of all grand mean	HSD	Measuring of Honest Significant Difference
d_f	Degree of free		