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Parametric optimization and predictive modelling of the mechanical properties of low carbon steel carburized at different temperatures and holding time using hybrid organic carburizers

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Abstract
The optimal design model of response surface methodology (RSM) was employed to optimize and develop predictive models for hardness, tensile and impact properties of low carbon steel carburized with groundnut and palm kernel shells (60/40 wt%), quenched in water and tempered at 250°C for stress relief. The test pieces machined to ASTM specifications were embedded in these carbonaceous materials, heated to the selected temperature and holding time for carburization. Tests were conducted on the test pieces to determine the effect of carburization on the mechanical properties. The carburization temperature ranges from 800°C to 1000°C at the interval of 50°C and the holding time ranges from 30mins to 150mins at the interval of 30mins. Carburization temperature and holding time were considered as process factors, while hardness, tensile and impact properties were considered as response variables. Numerical, graphical, and other statistical solutions and models that predict responses for carburization operating parameters were generated. From the optimization solutions, carburization at the temperature of 922.98°C and minimum holding time of 80.55 mins with near-unity desirability, corresponding to optimal responses of 216.95 HBN, 731.49 MPa and 31.53 J for hardness, tensile strength and impact energy respectively were obtained. The developed quadratic models showed a significant relationship between the factors and the responses.

Keywords: *Carburization; Response Surface Method; Quenching; Carburization temperature; Holding time.*

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I. INTRODUCTION

The technical criteria for materials selection in any given application demand that such materials should satisfy the required mechanical properties level with regard to hardness, tensile, impact, among others. This is to enable them to withstand the stresses that will likely come into play in the intended service condition. This is very vital and should not be compromised in order to ensure safe and proper functioning of the engineering facility, and to avoid untimely, and catastrophic failure of the service facility. For instance, components like gears, bearings, cams, crankshaft, rock drilling bits, agro farming tools etc., are expected to have a unique balance of properties, such as a hard outer surface and tough inner core, to withstand wear and prevent the initiation and propagation of cracks propagation respectively [1–3]. Low carbon steel being through-hardened and brittle is unsuitable for the above mentioned applications [4]. Local enrichment of the surfaces of low carbon steels with carbonaceous materials, and subsequent quenching in a suitable medium results in a hardened surface and tough inner core [3–6]. This can be achieved through carburization process, which impart certain desirable mechanical properties into a metallic material by adding carbon to enable it function properly in the proposed service condition [7]. The effectiveness of a carburization process is dependent on the diffusion potential of the species in the carburizing material and the operating temperature of the furnace [3]. Thermokinetic treatment is applied to various type of materials in order to enhance their properties and performance. During a thermokinetic process, atomic diffusion takes place, the rate of which can often be improved by adjusting the heat treatment process parameters, such as temperature, time and cooling rate. Using appropriate mathematical modelling, diffusion kinetics and constants, the thermokinetic temperatures, times, and cooling rates can be estimated [8]. It is generally known that diffusion carbon in iron takes place through interstitial mechanism, which is occurs more rapidly in most metals and alloys than vacancy and substitution diffusion mechanisms. This is attributed to the smaller size and quick mobility of interstitial atoms [9,10]. The existence of more empty interstitial sites than vacancies, further increases the probability of interstitial atomic migration than vacancy diffusion. Diffusion coefficient and rates is most profoundly influenced by temperature, as temperature increases the reaction kinetics. This can be practically buttressed using the case of self-diffusion of iron, where the magnitude of coefficient of diffusion increases to approximately six orders as the temperature rises from 500 to 900 °C [8].

When solid particles of carbonaceous material are used for carburization, it is called pack carburizing. In this case, the mild steel is enclosed in a well-sealed box containing suitable carburizer then the temperature is raised to the austenitic region of the steel which specifically depends on the weight per cent composition of carbon traced on the iron-carbon phase diagram. Generally, during the pack-carburization of low carbon steel, the temperature ranges between 850 °C and 950 °C and then held for some time interval at that temperature [10]. Different types of carburizing media and energizers based on synthetic chemical carbonates, such as $BaCO_3$, $CaCO_3$, and Na_2CO_3 are used in the commercial pack carburization of low carbon steel, to improve the carbon potential of the carburizing materials. In recent times, environmental concern and drive for cost reduction have resulted in researches to determine the potential and efficacy of various organic/agricultural wastes as energizers to substitute the chemical/synthetic and commercial carbonates of Ca, Ba, and Na during carburization of low carbon steel. The carburizing potential of some local organic waste materials as substitutes or complements for commercial carbonates of barium, calcium, and sodium in the surface hardness improvement of low carbon steel were variously studied by researchers over the years [11–16]. Most of the investigations had the objectives of cost reduction and minimizing the pollution problems associated with the use of synthetic carbonates. These researches carried out on the application of some household, industrial and agriculturally derived waste materials in the carburization of low carbon steel revealed that the wastes generated from these sources significantly enhanced the surface hardness of low carbon steel at different temperatures and holding times [4,10,16].

Experimental designs, optimization, and statistical analysis tools and techniques have been applied by many researchers for the analysis of the experimental results as well as the mathematical modelling and validation of carburization process parameters for low carbon steels [17–25]. Response surface methodology (RSM) has been well utilized among other experimental design and optimization techniques. RSM is a statistical and experimental design tool which is used to examined and analyze the impact of variation in one or more independent process parameters on selected response variables [26–32]. Hardness and other mechanical properties of carbon steel are influenced by variations in carburization process parameters, such as temperature and holding time [22,23,25]. Response surface optimization and predictive modelling is useful for determining the response of materials' properties to the changes in the process parameters (inputs) and deriving regression equations that can forecast the optimal performance. Therefore, application of RSM in the present study is necessary for predicting the optimal operating parameters (temperature and holding time) for the carburization of low carbon steel with groundnut and palm kernel shells which will result in optimum surface hardness, tensile strength and impact energy. This research aims to study, analyze statistically and optimize the process parameters for the carburization of low carbon steel using local agricultural wastes as hybrid carbonaceous materials (pulverized groundnut shell and palm kernel shell), and develop mathematical models for predicting the mechanical properties for the carburization process.

II. MATERIALS AND METHODS

2.1 Materials

The materials used in this research majorly include 1018 low carbon steel rods, the groundnut and palm kernel shells used as carbonaceous materials for the carburization were sourced locally. The groundnut and palm kernel shells were carbonized at the temperature of 300and 550 °C respectively and quenched with water to avoid turning the carbon into ash. The carbonized groundnut shell (GNS) and palm kernel shell (PKS) was pulverized to fine particles and passed through a sieve size 75 microns. A set of sieves mounted on a motorized vibrator was used for particle size classification. Other equipment used includes a heat treatment furnace, hardness testing machine, tensile testing machine and impact testing machine. The chemical composition of the steel used for this research is as contained in Table SM1.

2. 2 Pack Carburization Process and Test for Mechanical Properties

A box-type resistance furnace with model number SX-5-12 and a temperature capacity of 1200°C was used for the carburization heat treatment. The low carbon steel was machined to standard ASTM specifications of tensile, impact and hardness test pieces before carburization heat treatmentas reported in [2,5]. The prepared test samples were enclosed in a steel pot filled with pulverized carbonized groundnut and palm kernel shells mixed in the ratio of 60/40 wt.%. The steel pot was well sealed to prevent carbon escape and penetration of unwanted furnace gas in the steel pot during heating. The furnace temperature was adjusted to the desired levels within the austenitic temperature of steel (800, 850, 900, 950 and 1000 °C) for each successive heat treatment stage respectively, after which the loaded steel pot was charged into the furnace. The furnace heating at the average rate of 8.89 °C/mins attained 800 °C at about 1hr: 30 mins. It was then held at the temperature for the required time of 30, 60, 90, 120 and 150 minutes respectively to allow for maximum carbon diffusion. After holding for specified time to allow for maximum carbon diffusion and saturation, the steel pot was unloaded from the furnace and the test piece quenched in water and subsequently tempered at 250°C for 60 mins in order to relieve internal stresses due to rapid cooling. After the thermokinetic cycle for other temperature and holding time regimes, the test samples were subjected to hardness, tensile and impact energy tests using the Phase II Model 900-355 Brinell hardness tester, universal tensile testing machine with factory number 130812 (JPL-100KN), Hounsfield balanced impact testing machine and following standard procedures as reported in [10,33,34]. The control sample was also tested for hardness, tensile and impact energy absorbed, the results of which are presented in Table SM2. The average value of the test results conducted in three repeat tests were collated for statistical analysis, optimization, and predictive modelling.

2.3 Optimization of Hardness, Tensile and Impact Properties Using Response Surface Method

The experimental results for hardness, tensile and impact properties were modelled and optimized using the optimal custom design model of the response surface method of the design expert software package version 11. Austenite transformation temperature range of 800-1000 °C at the interval of 50 °C was considered and holding time of 30, 60, 90, 120 and 150 minutes. Carburizing temperature and holding time were set as independent variables (factors X_1 and X_2) while the mechanical properties; hardness, tensile strength, and absorbed impact energy were designated as dependent variables (responses 1, 2 and 3). The actual experimental results of mechanical properties tests at different carburizing temperature and holding time are provided in Table SM2, while the summary of the design settings for the factors and response variables are contained in Tables SM3 and SM4. Twenty-five experimental runs were performed to obtain the results in the design layout given in Table SM5.

III. RESULT AND DISCUSSION

The experimental results of hardness, impact and tensile tests employed in the statistical analysis, modelling and optimization are provided in Table SM2.

3.1 Statistical Analysis, Optimization, and Modelling of Carburization Process Parameters and Properties

The design of experiment enables the statistical analysis, predictive modelling of responses and optimization of the effect of various factors on the response of a process [27,30–32]. The effect of the carburization process factors (temperature and holding time) on the hardness, tensile strength, and impact energy of the pack carburized low carbon steel were evaluated using the optimal design of RSM. The design summary and matrix, showing the report of the design, factors, responses, and build information is presented in Tables SM2, SM3 and SM4. Table SM4 shows the design matrix and experimental results. The results showed that the hardness (Response 1) is directly proportional to the tensile strength (Response 2) but inversely proportional to the absorbed impact energy (Response 3).

Figures 1a-c are predicted vs actual plots for hardness, tensile and impact properties respectively. The predicted vs actual graphs are graphs of the observed (actual) response values versus the predicted response values which help to determine the observations that are not well predicted by the model. From these graphs, the actual responses are well predicted by the model evidence which is point association near-perfect linear graphs. The diagnostics plots and model graphs of predicted responses versus observed responses for all the factors showed a linear graph with the data points split evenly by the 45-degree line, indicating that the design models are sufficient to predict the factors’ responses.

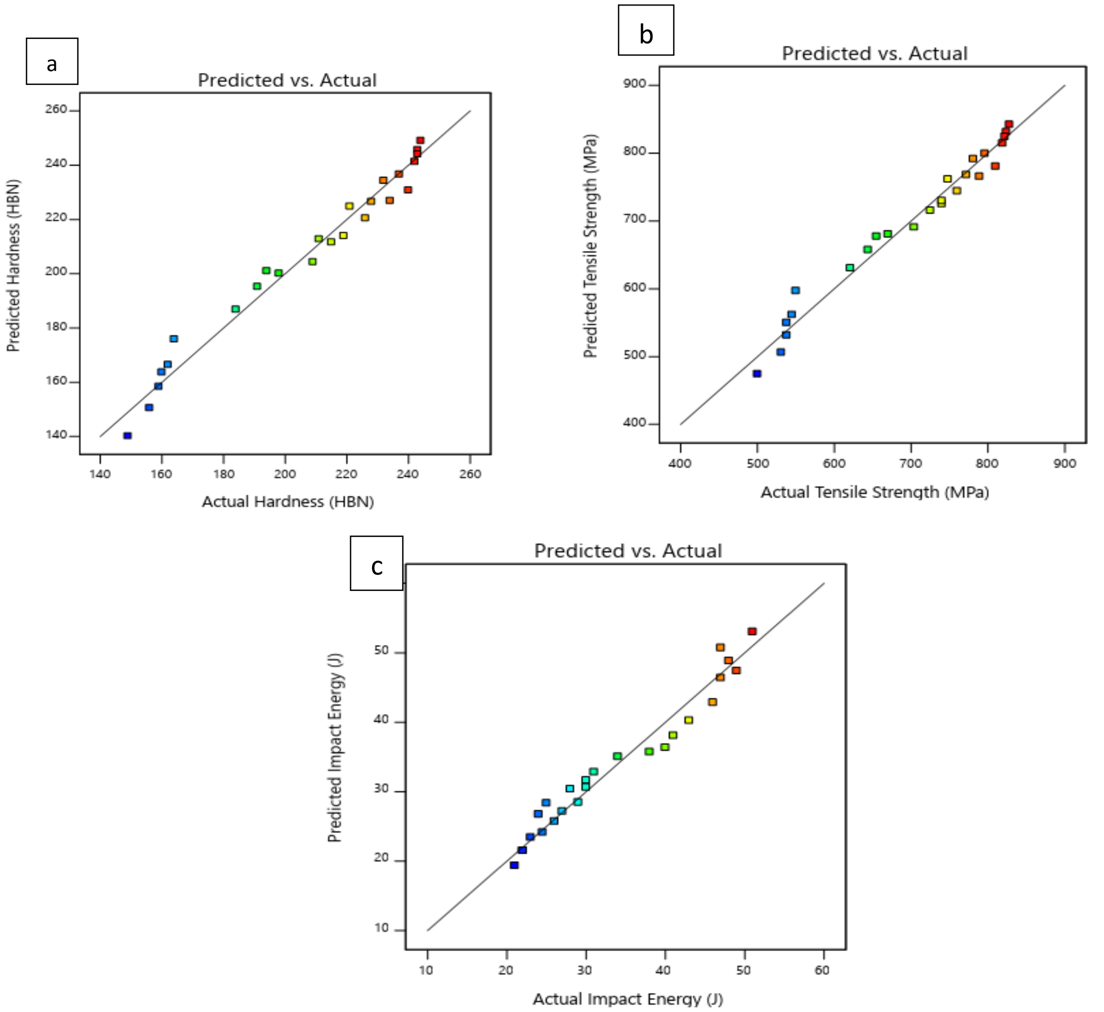

Figure 1: Plots of predicted vs actual; (a) hardness (HBN), (b) tensile strength (MPa), (c) impact energy absorbed (J).

Figures 2a-c, are the interactive 3-D plots for hardness, tensile and impact properties respectively. The 3-D surface plot is the projection of the contour plot in addition to shape and colour. The relative interaction of the factors (carburizing temperature and holding time) and their level of influence on the mechanical properties could be observed in the 3D surface plots. The 3-D showed the immediate effect of temperature as a driving factor in carburization with a noticeable increase in hardness and tensile strength and a proportionate decrease in impact energy. Also, for every increase or decrease in both hardness and tensile properties, there is evidence of a decrease or increase of impact energy respectively. From the temperature of about 850 to 950 °C, there is a steady increase of both hardness and tensile properties accompanied with a steady decrease of impact energy, this temperature range should be the austenite transition temperature for the steel material where interstitial filling of carbon atoms showed increased activities, thus, increasing carburization. At the red colour lies the peak of each property analyzed.

property analyzed.

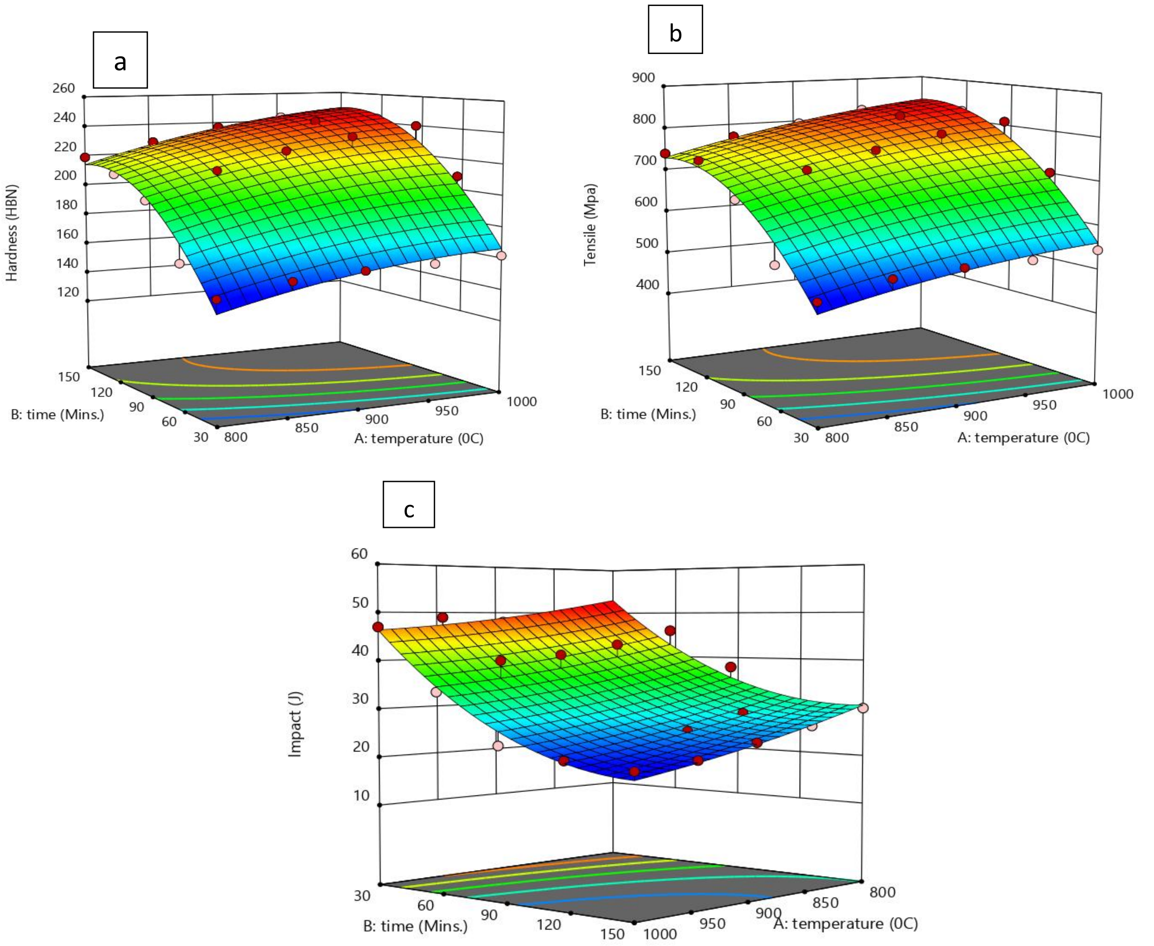

Figure 2: Interactive 3D plots of (a) hardness (HBN), (b) tensile strength (MPa), and (c) absorbed impact energy (J).

Figures SM1a-c, are contour plots for hardness, tensile strength and impact energy respectively. A contour plot is a two-dimensional (2-D) representation of the response plotted against combinations of numeric factors. From the contour plots, the effects of temperature and time influencing the change in properties is more obvious at the temperature around 900 °C. It could therefore be stated, that this temperature is best for carburizing this steel material, the evidence is shown in the optimization report (Table SM9) and overlay plots (Figure 12) to be 922.98 °C and time 80.55 mins. The perturbation plots of Figure 3, bring out succinctly the individual impacts of temperature and holding time as the operating parameters, on the mechanical properties tested. A steep curvature reveals the parameter that has more influence on the responses (hardness, tensile and impact energy) [30]. In this case, it will be stated that the holding time had a more significant effect on the properties of the carburized steel than the carburization temperature.

3.2. ANOVA, Fit Statistics, and Model Equations

Tables 1, 2 and 3 showed the ANOVA and fit Statistics results. The significance of the model for the properties analyzed showed that the model explains a significant portion of the variance. The model probability value (**P-value**) from all the results obtained in the regression for hardness, tensile strength and impact energy was less than 0.05, which is a good indication that the model terms have a significant effect on the response variables, hence good for the design.

The ANOVA and fit statistics tables showed the fit statistics for hardness strength, tensile strength, and impact energy respectively. As a rule, the difference between the **predicted R^2** and the **adjusted R^2** should not be more than 0.2 [26,30,32,35].

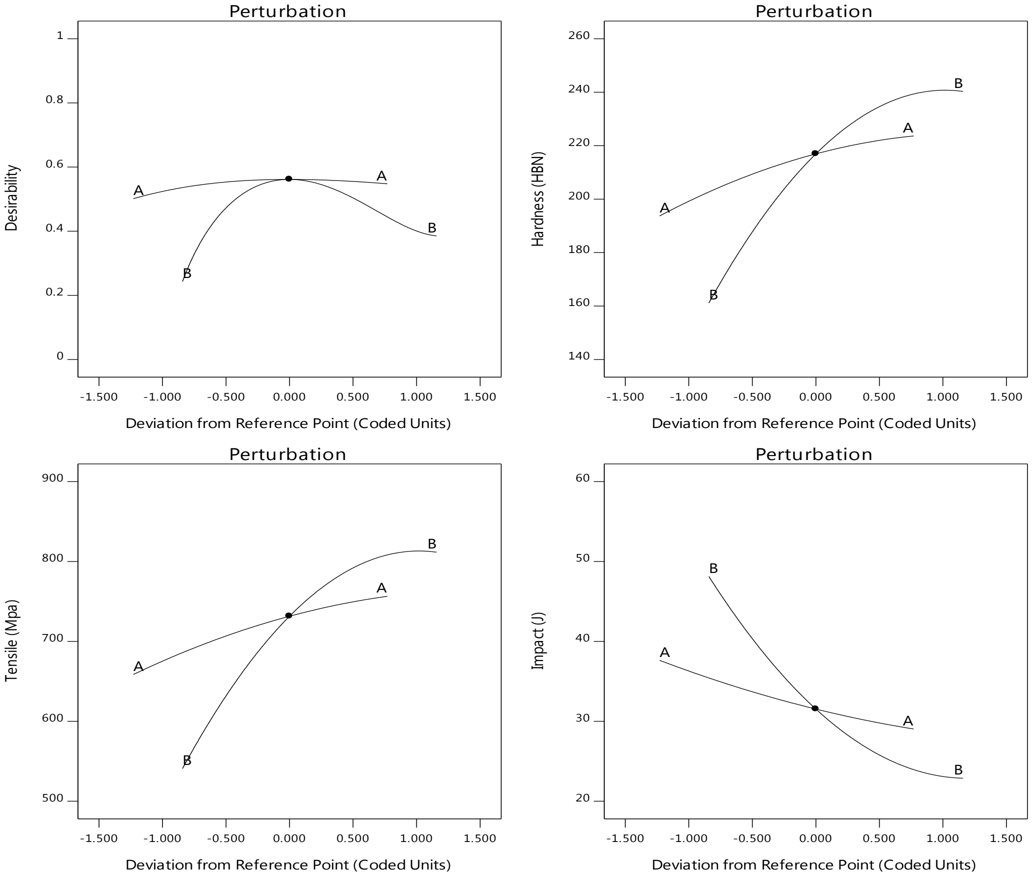

Figure 3: Perturbation plots of the carburization factors.

From Tables 1, 2, and 3, predicted and adjusted R^2 are in reasonable agreement because their difference is less than 0.2. Besides that, adequate precision **(Adeq Precision)** measures the signal to noise ratio. As a rule, a ratio greater than 4 is desirable. The ratio of 38.08, 37.29, and 29.66 in Tables 1, 2, and 3 respectively indicate adequate signal, such that the models can be used to navigate the design space. Also, from the fits statistics, the mean for hardness, tensile and impact energy is 206.44, 698.00, and 33.86 respectively. The standard deviation statistics contained in these tables are 5.83, 20.13, and 2.32 for hardness, tensile and impact energy respectively. The C.V values of 2.83%, 2.88% and 6.85% and the standard deviation contained in the fit statistics for hardness, tensile and impact respectively justifies the capability of the process.

Tables SM6, SM7 and SM8 are coefficient tables for the model terms in terms of coded factors for hardness, tensile and impact properties respectively. It is the table derived from the regression analysis. These tables provide the confidence intervals around the estimated model coefficients. It is from these coefficient tables that the final equations in terms of coded factors were automatically generated. The coefficient estimate represents the expected change in response per unit change in factor value when all remaining factors are held constant. Model terms have either negative or positive coefficients, showing a maximum negative and positive impact on the response/mechanical properties respectively [31,36,37]. The overall average response of all the experimental runs is given by the intercept in an orthogonal design. The coefficients are adjusted around this average response based on the factor settings. Conventionally, orthogonal factors are identified with unit VIFs; while VIFs greater than 1 indicate multi-collinearity, the higher the VIF the more severe the correlation of factors [26]. As a rough rule, VIFs less than 10 are tolerable. From the coefficients tables, the VIFs are 1 for all the responses.

The model equations expressed in factor coding can be used to predict the responses of the pack carburized mild steel properties at specified carburizing temperatures (°C) and holding time (mins). By default, the high levels of the carburization process factors are coded as +1 and the low levels are coded as -1. The coded equations are useful for identifying the relative impact of the factors by comparing the factor coefficients [27,38]. Equations (1), (2) and (3) are final model equations expressed in terms of coded factors for hardness, tensile strength, and impact properties respectively.

Table 1: ANOVA and Fit Statistics for Hardness.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	24487.90	5	4897.58	143.99	< 0.0001	Significant
X_1-temperature	2933.78	1	2933.78	86.25	< 0.0001	
X_2-time	19090.58	1	19090.58	561.26	< 0.0001	
X_1X_2	30.25	1	30.25	0.89	0.36	
X_1^2	113.16	1	113.16	3.33	0.08	
X_2^2	2320.13	1	2320.13	68.21	< 0.0001	
Residual	646.26	19	34.01			
Cor Total	25134.16	24				
Std. Dev.	5.83		**R^2**	0.97		
Mean	206.44		**Adjusted R^2**	0.97		
C.V. %	2.83		**Predicted R^2**	0.95		
			Adeq Precision	38.08		

$$Hardness\ (BHN)\ =\ 220.50 + 15.32X_1 + 39.08X_2 + 2.20X_1X_2 - 5.09{X_1}^2 - 23.03{X_2}^2 \tag{1}$$

Table 2: ANOVA and Fit Statistics for Tensile strength.

zzzzz	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	2.84E+05	5	56721.13	139.92	< 0.0001	Significant
X_1-temperature	31150.08	1	31150.08	76.84	< 0.0001	
X_2-time	2.245E+05	1	2.245E+05	553.67	< 0.0001	
X_1X_2	240.25	1	240.25	0.5926	0.45	
X_1^2	795.66	1	795.66	1.96	0.18	
X_2^2	26969.66	1	26969.66	66.53	< 0.0001	
Residual	7702.36	19	405.39			
Cor Total	2.91E+05	24				
Std. Dev.	20.13		**R^2**	0.97		
Mean	698.00		**Adjusted R^2**	0.97		
C.V. %	2.88		**Predicted R^2**	0.95		
			Adeq Precision	37.29		

$$Tensile\ Strength\ (MPa) = 744.00 + 49.92X_1 + 134.00X_2 + 6.20X_1X_2 - 13.49{X_1}^2 - 78.51{X_2}^2 \tag{2}$$

Table 3: ANOVA and Fit Statistics for Impact Energy Absorbed.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	2335.68	5	467.14	86.95	< 0.0001	Significant
X_1-temperature	250.88	1	250.88	46.70	< 0.0001	
X_2-time	1909.62	1	1909.62	355.44	< 0.0001	
X_1X_2	8.41	1	8.41	1.57	0.23	
X_1^2	3.21	1	3.21	0.60	0.45	
X_2^2	163.56	1	163.56	30.44	< 0.0001	
Residual	102.08	19	5.37			
Cor Total	2437.76	24				
Std. Dev.	2.32		**R^2**	0.96		
Mean	33.86		**Adjusted R^2**	0.95		
C.V. %	6.85		**Predicted R^2**	0.93		
			Adeq Precision	29.66		

$$Impact\ Energy\ (J) = 30.37 - 4.48X_1 - 12.36X_2 - 1.16X_1X_2 + 0.86{X_1}^2 + 6.11{X_2}^2 \tag{3}$$

3.3 Parametric Optimization Report

During numerical optimization, target criteria constraints/goals were set for both the factors and the responses. The carburization temperature and holding time were set to be in range. On the other hand, the responses of hardness, tensile strength, and impact properties were set to maximum. Normally, numerical optimization goal is not to maximize the desirability value, but an acceptable outcome of the numerical optimizationis indicated bythe factor settings that give rise to the highest desirability value. Table SM9 contains the report of numerical optimization solutions. From the table, three solutions were found. Out of the 3 solutions found, one was automatically selected. The solution showed that considering the independent and the dependent variables, at the carburizing temperature of 922.98°C, holding time of 80.55 Mins and desirability of 0.56, the optimal responses of 216.95 HBN, 731.49 MPa and 31.53 J was obtained for the properties tested. These optimal properties obtained after process optimization are higher compared to properties improvement using similar organic carbonaceous materials without optimization of the process parameters [2,5,11,16]. Also, it could be observed that optimal properties were obtained at lower carburization temperatures unlike that obtainable in the un-optimized carburization process [13].

Figure SM2 is the contour plots of numerical optimization solutions for the mechanical properties and their desirability. All response variables and the desirability plots can be used to explore how the factor settings influenced the response. Figure SM3 showed the overlay plot of the graphical optimization solution for the properties tested at the given range of carburization temperatures and holding time respectively. A single spot is produced in the overlay plot which highlights the “sweet spot” where the probability of meeting the target response criteria is high. It is also used to show the limits of failure in a process. The limits specified by the target goalis used to plot thecontours. The bright yellow by default defines the acceptable factor settings, while the grey colour on the other hand defines the unacceptable factor settings. If intervals are included on the criteria, then a blend of the acceptable and unacceptable colours is used to show where the interval limits are unacceptable. The numerical optimization solutions, depicted as flags are carried over and displayed on the overlay graph, showing the optimal values of the selected optimization solutions. Figure SM3 clearly shows that, at the temperature of 922.98 ^{o}C, it is possible to effectively carburize the low carbon steel samples for about 80.55 minutes to have improved surface hardness and tensile strength with a balanced decreased of impact absorption energy.

IV. CONCLUSIONS

Quadratic regression models were generated for hardness, tensile strength, and impact energy of the low carbon steel carburized with groundnut and palm kernel shells with a solution of optimum values. It is clear from the analysis of the results that carburization using these hybrid organic carbonaceous agro wastes increase the hardness and tensile strength of low carbon steel tested but decrease the impact energy absorbed. The models generated are useful for predicting the optimal operating parameters (temperature and holding time) for the carburization of low carbon steel with groundnut and palm kernel shells for optimum hardness, tensile strength and impact energy.

Declarations of competing interests

The authors declare that they have no competing interests.

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