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British Journal of Contemporary Research

BEXFORD PUBLISHING

Taguchi Analysis of Brinell Hardness in Cow Bone Particle/Cassava Cortex Fibre Epoxy Composites

Original Research Article | Volume 1 | Issue 6 | 2026 | Article Number: 023

Accepted: 19 October 2026 | Published: 10 November 2026 | ISSN: 2979-8582 (Online)

Crossref : **https://doi.org/10.67693/BJCR-74CTZMJN**

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Abstract

This study investigated the Brinell hardness of epoxy composites reinforced with cassava-cortex fibres and cow-bone particles using an 18-run Taguchi experimental design. Reinforcement composition, sodium hydroxide treatment and particle size were varied systematically. Hardness was measured on three specimens per formulation, and the mean values were analysed using factor-response comparisons, analysis of variance and categorical regression. Mean hardness ranged from 21 to 43 BHN, with the highest value recorded for RDT4PS100, containing 20 vol.% cassava cortex and 20 vol.% cow bone. Reinforcement composition produced the largest variation in the observed response. Treatment and particle-size effects were more difficult to separate because their experimental assignments overlapped. The findings demonstrate the potential of the balanced hybrid formulation for further mechanical and application-specific evaluation.

Keywords: Hybrid Composite, Hardness, Taguchi Method, Analysis Of Variance, Cow-Bone Particles, Cassava-Cortex Fibre, Epoxy Composite

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1. Introduction

Lightweight polymer composites have attracted attention in vehicle design because they offer opportunities to tailor mechanical performance while limiting component mass. Natural reinforcements are particularly attractive where locally available resources can be incorporated into manufactured products. Their performance nevertheless depends on processing consistency and the quality of bonding within the polymer matrix (Bledzki and Gassan, 1999; Mohanty et al., 2005; Mallick, 2007). Recent reviews identify interface compatibility, moisture sensitivity and service durability as continuing barriers to automotive deployment (Musa and Onwualu, 2024; Islam et al., 2024; Ullah et al., 2025).

Agricultural and animal-derived residues can serve as reinforcing phases in polymer composites when they are appropriately processed. Their effect on mechanical behaviour is influenced by particle size, dispersion, surface condition and the relative proportions of the constituents (Mohanty et al., 2005; Sapuan, 2017). Contemporary epoxy-composite reviews also emphasise that reinforcement treatment and fabrication route must be reported sufficiently to support reproducibility (Mahmud et al., 2025).

Cassava cortex is a lignocellulosic reinforcement, whereas thermally processed cow bone provides a mineral-rich particulate phase. Their combination permits the relative proportions of fibrous and particulate reinforcement to be varied while holding total reinforcement at 40 vol.%. This creates a useful basis for investigating whether a balanced hybrid mixture exhibits a different macroscopic indentation response from fibre-rich or bone-rich mixtures. Unlike studies that consider natural fibres or mineral fillers separately, the present work compares six reinforcement compositions within an 18-run experimental programme and distinguishes measured hardness from estimates for untested settings. The contribution is a formulation-level comparison of this specific waste-derived hybrid system, rather than proof of independently optimised treatment or particle-size effects (Mohanty et al., 2005; Musa and Onwualu, 2024).

This work examines how the relative proportions of cassava cortex and cow bone, alkaline treatment and particle size influence the Brinell hardness of the resulting epoxy composites. The experimental comparisons provide a basis for identifying the most promising formulation among those tested.

The Taguchi approach was adopted to organise the experimental combinations efficiently. Factor-level responses were examined using the larger-the-better signal-to-noise criterion, alongside analysis of variance and regression modelling. Interpretation of these methods was guided by the structure of the actual experimental assignments (Taguchi, 1986).

The study aimed to compare the hardness of 18 formulations, examine the relative influence of the selected factors and identify the highest-performing formulation. Predicted factor-level combinations were assessed separately from the observed experimental results.

2. Materials and Methods

2.1 Materials

Diglycidyl ether of bisphenol A (DGEBA) epoxy resin and its hardener formed the matrix system. Cassava cortex and cow bone served as reinforcements, while sodium hydroxide was used in the surface-treatment stage. The reported resin-to-hardener mixing ratio was 2:1.

2.2 Preparation of Reinforcements

Cassava cortex collected in Calabar was cleaned to remove soil and extraneous matter, dried, milled and sieved to obtain the required particle fractions. The cassava reinforcement was prepared at sodium hydroxide treatment concentrations of 0, 2 and 4 wt.% and kept in airtight containers before composite

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fabrication. The treatment concentrations were incorporated as experimental factor levels.

Cow bones were cleaned and boiled to remove adhering tissue, dried at 105 °C for 24 h and calcined at 600–800 °C for 2–3 h. Milling and sieving produced the particle fractions used in the experiments.

2.3 Composite Fabrication

Diglycidyl ether of bisphenol A (DGEBA) epoxy resin and hardener were mixed at a resin-to-hardener ratio of 2:1. The prepared cassava-cortex and cow-bone fractions were combined in the prescribed proportions and gradually introduced into the resin system with mechanical stirring to promote dispersion. Reinforced formulations contained 60 vol.% epoxy matrix and 40 vol.% total reinforcement, while the unreinforced control comprised neat epoxy. The mixtures were poured into prepared moulds, degassed and allowed to cure for 24 h, followed by post-curing. The resulting composite specimens were prepared for mechanical characterisation.

Table 1. Composite formulations by volume fraction.

No	Designation	Cassava Cortex (vol.%)	Cow bone (vol. %)	Epoxy (vol. %)
1	A	0	0	100
2	B	0	40	60
3	C	10	30	60
4	D	20	20	60
5	E	30	10	60
6	F	40	0	60

2.4 Experimental Design

The experimental programme comprised 18 formulations arranged using an L18 Taguchi design. Reinforcement composition had six levels, while sodium hydroxide treatment and particle size each had three levels. The investigated particle sizes were 100, 250 and 500 µm. Factor definitions and experimental assignments are presented in Tables 2 and 3.

Table 2. Experimental factors and their corresponding levels in the L18 design.

			Level					
Factor	Abbreviation	Unit	1	2	3	4	5	6
Reinforcement	R	%	A	B	C	D	E	F
Treatment	T	%	0	2	4	-	-	-
Particle Size	PS	µm	100	250	500	-	-	--

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Table 3. Experimental runs, factor settings, sample designations and measured mean Brinell hardness.

Run	Reinforcement (R) %	Treatment (T) %	Particle Size (PS) (μm)	Designation	Hardness (BHN)
1	A (1)	0	100	$R_AT_0PS_{100}$	25
2	A (1)	2	250	$R_AT_2PS_{250}$	28
3	A (1)	4	500	$R_AT_4PS_{500}$	27
4	B (2)	0	100	$R_BT_0PS_{100}$	38
5	B (2)	2	250	$R_BT_2PS_{250}$	37
6	B (2)	4	500	$R_BT_4PS_{500}$	39
7	C (3)	0	100	$R_CT_0PS_{100}$	34
8	C (3)	2	250	$R_CT_2PS_{250}$	36
9	C (3)	4	500	$R_CT_4PS_{500}$	35
10	D (4)	0	500	$R_DT_0PS_{500}$	39
11	D (4)	2	250	$R_DT_2PS_{250}$	41
12	D (4)	4	100	$R_DT_4PS_{100}$	43
13	E (5)	0	100	$R_ET_0PS_{100}$	25
14	E (5)	2	250	$R_ET_2PS_{250}$	26
15	E (5)	4	500	$R_ET_4PS_{500}$	27
16	F (6)	0	100	$R_FT_0PS_{100}$	21
17	F (6)	2	250	$R_FT_2PS_{250}$	22
18	F (6)	4	500	$R_FT_4PS_{500}$	24

2.5 Hardness testing

Brinell hardness testing was performed on prepared composite specimens using a 10 mm tungsten-carbide ball indenter, applied loads within the range 100–250 kgf and a dwell time of 30 s. The relatively large indenter was selected to assess a macroscopic response across the heterogeneous distribution of cassava-cortex reinforcement, cow-bone particles and epoxy-rich regions, rather than a highly localised response from a single constituent. The lower load range was chosen to limit excessive damage during indentation of the polymer composite. A 30 s dwell period provided a consistent loading interval while allowing for time-dependent indentation of the epoxy matrix; it does not imply complete elimination of viscoelastic effects. Three specimens were tested for each formulation, and the reported hardness values are their means, expressed as Brinell hardness numbers (BHN). The applied force is stated as a range because the individual force setting for each formulation is not available in the tabulated results. ASTM E10 describes Brinell testing of metals and is used here only as a reference for the indentation principle,

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not as proof of polymer-specific standard compliance.

The conventional larger-the-better Taguchi statistic is $\eta = -10 \log10[(1/n)\Sigma(1/y_i^2)]$, calculated from individual specimen results y_i. Because only the formulation means were available for this manuscript, the reported descriptive transformation is $\eta_mean = 20 \log10(\text{mean hardness})$. This is not equivalent to the replicate-based Taguchi statistic unless the individual observations are equal. Replicate-based S/N ratios and confidence intervals were not estimated from the available formulation means.

3. Results and Discussion

3.1 Hardness Behaviour Analysis

The mean hardness values of the 18 formulations ranged from 21 to 43 BHN. RDT4PS100 produced the highest measured value, 43 BHN. Its composition comprised 20 vol.% cassava cortex, 20 vol.% cow bone and 60 vol.% epoxy, with 4 wt.% sodium hydroxide treatment and a 100 µm particle-size fraction.

Among the reinforcement compositions, formulation D yielded the highest mean signal-to-noise response, 32.25 dB. This indicates comparatively strong hardness performance for the balanced hybrid formulation within the combinations investigated.

The hardness response may reflect the combined influence of the mineral particulate phase, reinforcement distribution and the fibre–matrix interface. These explanations are consistent with composite-material behaviour, although the hardness data alone do not isolate the contribution of each mechanism (Mallick, 2007; Sapuan, 2017).

The particle-size response did not establish a consistent independent trend across the tested formulations. Its interpretation is further restricted by the way particle size and treatment were assigned in the experimental design.

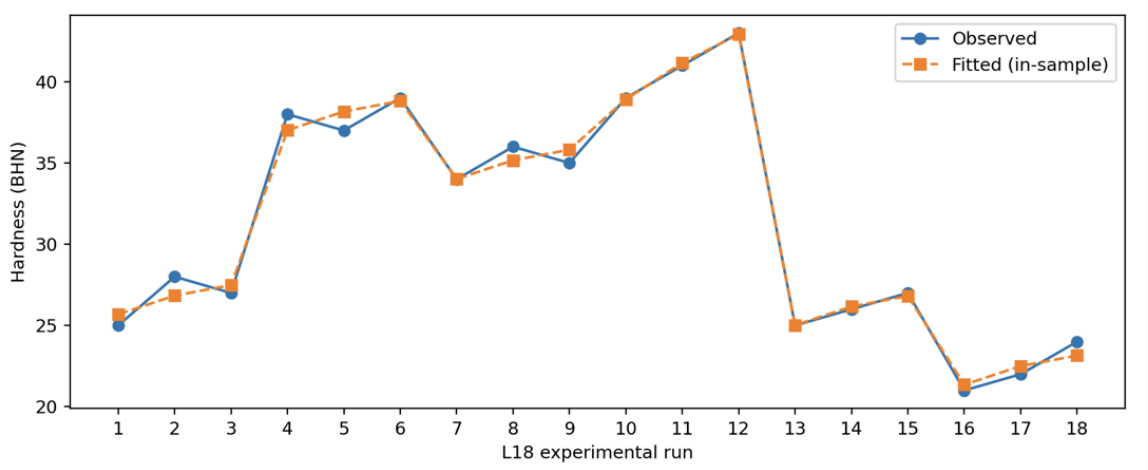


Figure 1. Observed formulation-mean hardness and fitted values for the 18 experimental runs

3.2 Signal-to-Noise Ratio Analysis

A larger-the-better response was adopted because increased hardness was the desired property.

Each formulation was represented by the mean of three specimens. The larger-the-better response was therefore evaluated as 20 log10(mean hardness). This measure allows comparison of the formulation means, although the full replicate-based Taguchi signal-to-noise ratio would also reflect the spread of individual readings.

For the 18 formulation means, the transformation 20 log10(mean hardness) gave factor-level response ranges of 5.28 dB for reinforcement composition, 0.64 dB for treatment and 0.35 dB for particle size. This

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mean-based response differs from the conventional replicate-based Taguchi signal-to-noise statistic. The experimental assignments also couple treatment and particle size, so their marginal contrasts cannot be interpreted as independent causal effects.

Table 4. Mean larger-the-better S/N ratios by factor level.

Level	Reinforcement (R)	Treatment (T)	Particle Size (PS)
1	28.51	29.40	29.54
2	31.59	29.81	29.81
3	30.88	30.04	29.90
4	32.25		
5	28.30		
6	26.97		
Delta	5.28	0.64	0.35
Rank	1	2	3

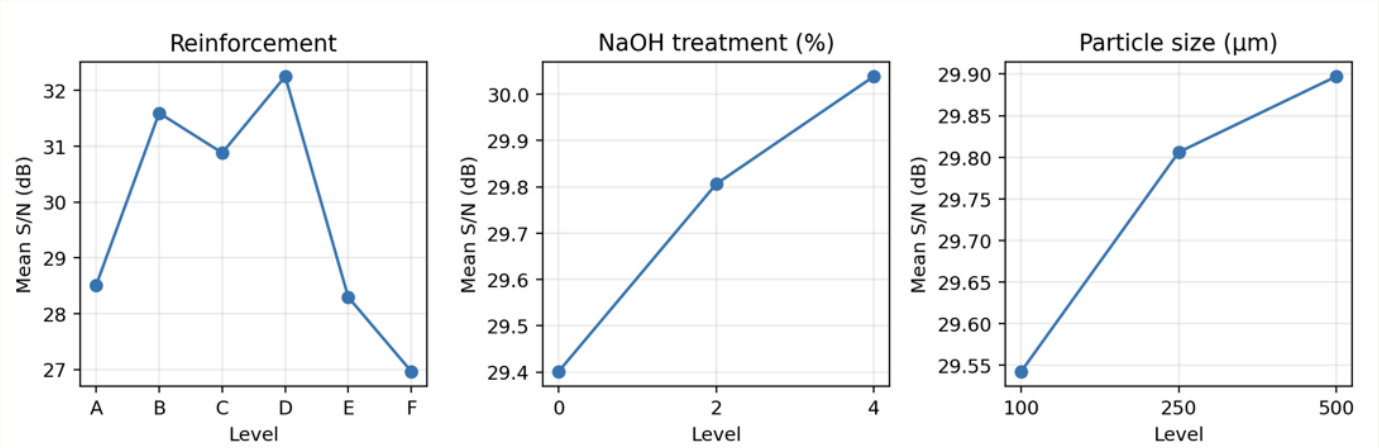


Figure 2. Main-effects plots of the mean-based hardness signal-to-noise response

3.3 Analysis of Variance and Design Limitations

Analysis of variance was undertaken on the 18 formulation-level responses. The resulting sequential sums of squares describe variation attributed to factors in the specified order of entry. Treatment and particle-size levels were not independently distributed across all reinforcement groups, which limits separate attribution of their effects (Montgomery, 2017).

Treatment and particle size were not fully crossed in the design; therefore, their nominal level contrasts are descriptive and cannot establish separate causal effects. The reported ANOVA R^2 of 98.93% concerns the mean-transformed hardness response, whereas the 99.20% R^2 reported later concerns a different model fitted to hardness means on the original BHN scale.

Table 5 presents sequential sums of squares, which depend on the order of entry of the factors. Because treatment and particle size are partly aliased, the percentages describe model allocation of variation rather than independent physical contributions. The residual reflects differences among formulation means rather

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than within-formulation specimen variability.

The analysis of variance was based on the 18 formulation means. It characterises differences between formulations rather than the variation among the three specimens within each formulation.

For five of the six reinforcement groups, treatment and particle-size levels progressed together; the remaining group followed a different assignment. Consequently, only one additional degree of freedom was estimable for particle size after reinforcement and treatment had entered the model. The fitted model had eight factor degrees of freedom, nine residual degrees of freedom and 17 total degrees of freedom.

Table 5. Sequential analysis of variance for mean-based hardness signal-to-noise responses (reinforcement, treatment and particle size entered in that order).

Source	DF	Seq SS	Adjusted SS	Adj MS	F	P	R^2	% PC
Reinforcement (R)	5	66.9742	—	13.3948	163.916	<0.001	—	97.09
Treatment (T)	2	1.2472	—	0.6236	7.631	0.01153	—	1.81
Particle size (PS)	1	0.0267	—	0.0267	0.327	0.58144	—	0.04
Residual error	9	0.7355	—	0.0817	—	—	—	1.07
Total	17	68.9836	—	—	—	—	98.93%	100.00

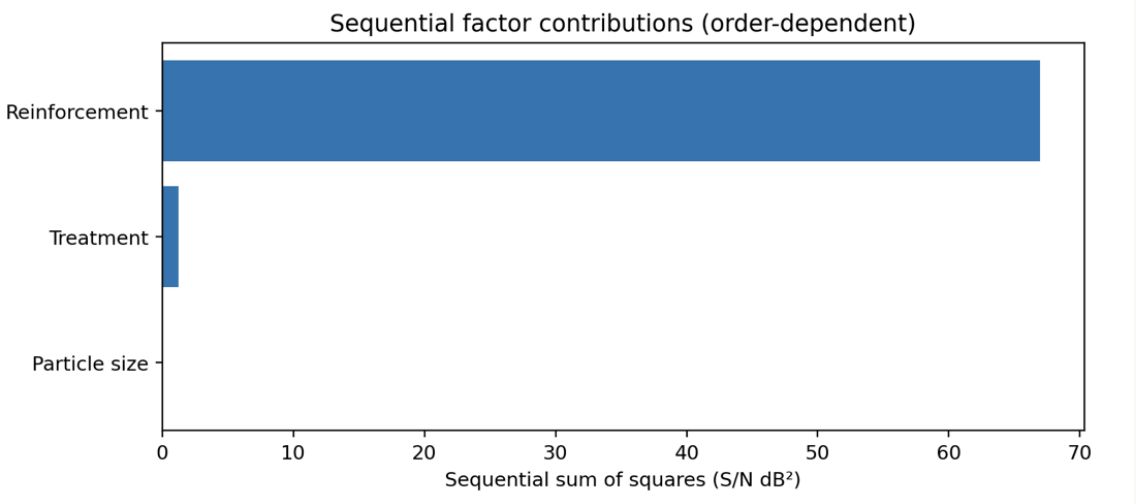


Figure 3. Sequential sums of squares for the hardness signal-to-noise analysis (factor entry: reinforcement, treatment and particle size)

3.4 Categorical Regression and Prediction Limits

A categorical main-effects regression was fitted to the 18 reported mean hardness responses, with reinforcement composition, treatment and particle size treated as factors. The fitted model took the form HB ~ C(R) + C(T) + C(PS) (Montgomery, 2017).

The categorical regression used R=A, T=0% and PS=100 μm as its reference levels.

The fitted equation was $\hat{H}$ (BHN) = 25.6833 + f(R) + g(T) + h(PS), with reference categories R=A, T=0% and PS=100 μm. The coefficients relevant to the proposed RDT4PS500 setting are f(D)=14.3333, g(4%)=2.9000 and h(500 μm)=−1.1000. This yields a model estimate of 41.8167 BHN,

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conditional on the additive main-effects assumptions. The full set of fitted values and residuals is presented in Table 6.

The fitted equation summarises the experimental combinations represented in the dataset. Its use for untested combinations would require additional experimental evidence.

The coefficient of determination (R^2 = 99.20%) refers to the fit of the categorical model to the 18 formulation means. It does not establish external predictive accuracy. Because treatment and particle size were coupled in most runs, their effects are not independently identified by this design; extrapolations to untested combinations are therefore provisional.

Residual diagnostics were examined for the fitted formulation-level model. As the model was based on means, the diagnostics reflect variation among those means and do not quantify differences between specimens within each formulation.

Table 6. Observed hardness, fitted values, residuals, and percentage differences.

Run	Hardness (BHN)	Fitted hardness (BHN)	Residual (BHN)	% Difference
1	25	25.6833	-0.6833	-2.73
2	28	26.8333	1.1667	4.17
3	27	27.4833	-0.4833	-1.79
4	38	37.0167	0.9833	2.59
5	37	38.1667	-1.1667	-3.15
6	39	38.8167	0.1833	0.47
7	34	34.0167	-0.0167	-0.05
8	36	35.1667	0.8333	2.31
9	35	35.8167	-0.8167	-2.33
10	39	38.9167	0.0833	0.21
11	41	41.1667	-0.1667	-0.41
12	43	42.9167	0.0833	0.19
13	25	25.0167	-0.0167	-0.07
14	26	26.1667	-0.1667	-0.64
15	27	26.8167	0.1833	0.68
16	21	21.3500	-0.3500	-1.67
17	22	22.5000	-0.5000	-2.27
18	24	23.1500	0.8500	3.54

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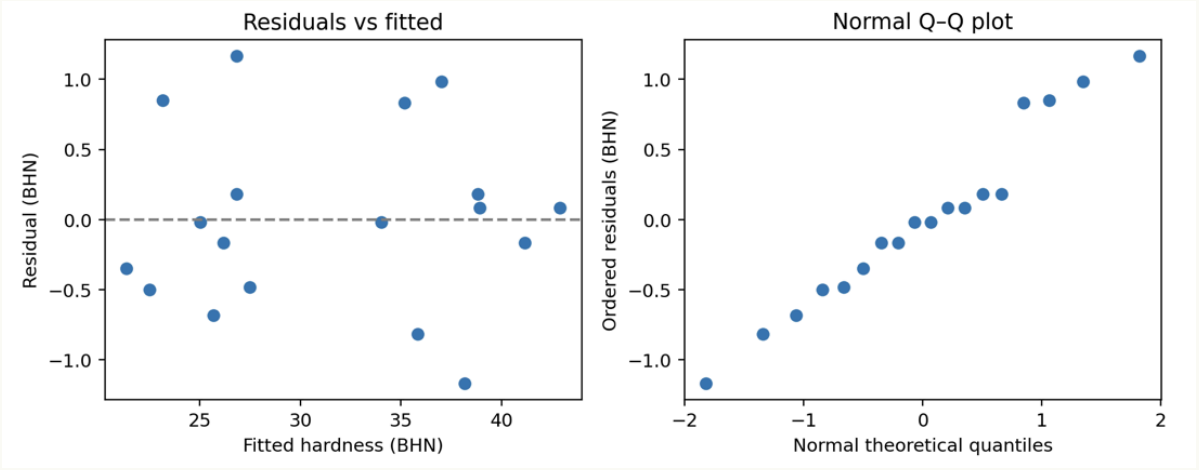


Figure 4. Residual and normal Q–Q plots for the categorical main-effects hardness model

3.5 Comparison of Observed and Model-Estimated Hardness

The measured formulation means were 38 BHN for RBT0PS100 (run 4) and 43 BHN for RDT4PS100 (run 12). The categorical additive model, fitted to the 18 available formulation means, estimates 41.8167 BHN for RDT4PS500, a combination not tested in the experimental programme. This estimate follows from 25.6833 + 14.3333 + 2.9000 − 1.1000 = 41.8166 BHN (41.8167 BHN using unrounded coefficients). The model-derived S/N value, 20 log10(41.8167) = 32.4270 dB, is not a specimen-level Taguchi S/N statistic. The measured RDT4PS100 hardness exceeds the reference by (43 − 38)/38 × 100 = 13.16%; the model estimate exceeds it by (41.8167 − 38)/38 × 100 = 10.04%. No independent confirmation test for RDT4PS500 is claimed.

Table 7. Observed hardness and model estimate for selected formulations (predicted setting not experimentally confirmed).

Measure	Reference RBT0PS100	Observed RDT4PS100	Model-predicted RDT4PS500
Status	Measured (run 4)	Measured (run 12)	Model estimate (untested)
Hardness (BHN)	38.0000	43.0000	41.8167
Mean-based S/N (dB)	31.5957	32.6694	32.4270
Change from reference	—	+13.16%	+10.04%

3.6 Microstructural Analysis

The SEM micrograph of RDT4PS100 reveals reinforcement dispersed within the epoxy matrix and a comparatively continuous composite structure. The observed morphology provides context for the hardness behaviour, although microstructural appearance alone does not establish the strength of the fibre–matrix interface (Mallick, 2007).

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Changes in hardness may be associated with reinforcement distribution and interfacial behaviour. However, the micrograph alone cannot quantify bond strength or establish the individual causes of the measured response.

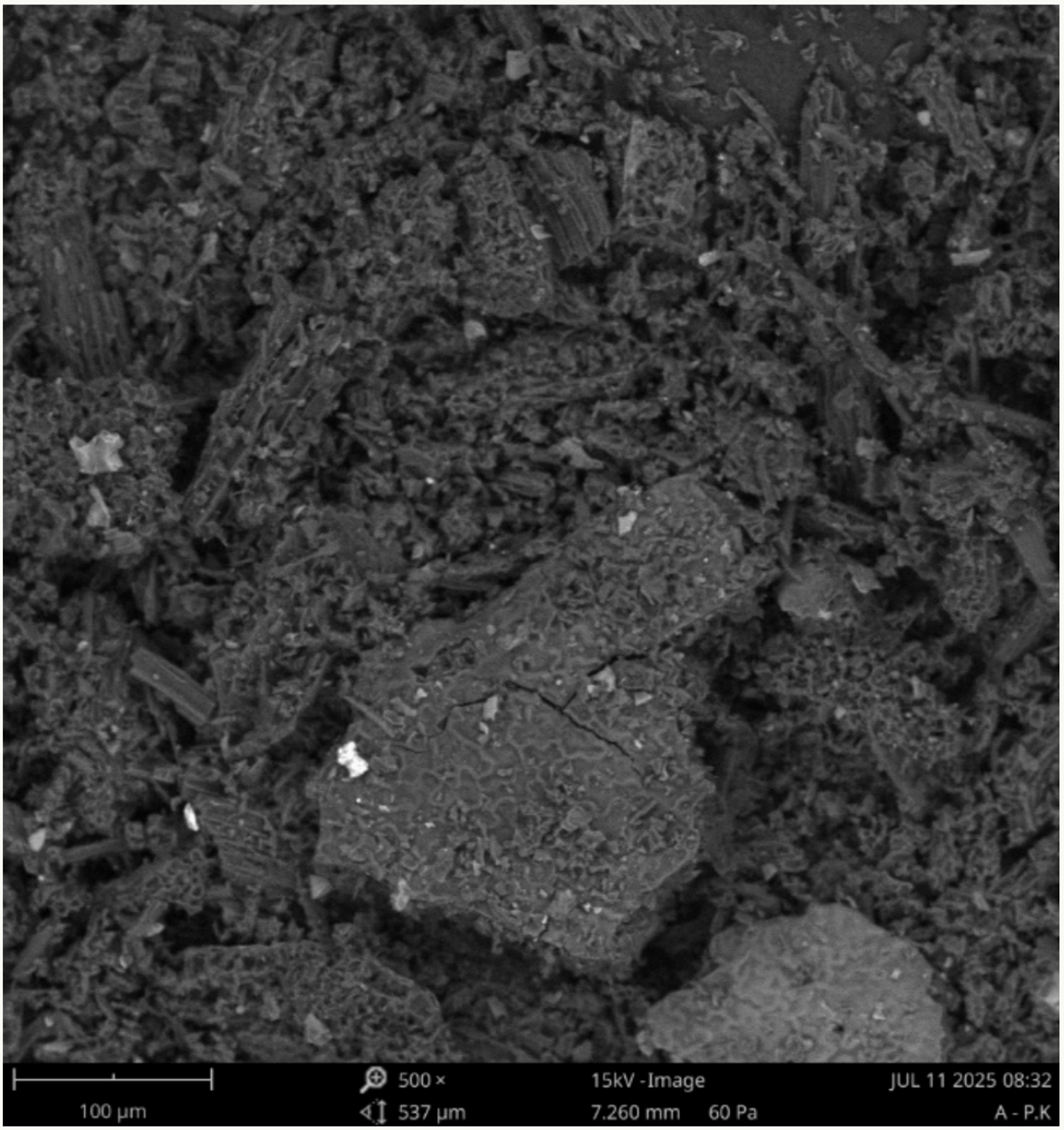


Figure 5. Scanning electron micrograph of composite formulation RDT4PS100

3.7 Automotive Application Potential

The measured hardness suggests that the balanced hybrid composite merits further investigation for non-structural automotive interior components. Hardness is only one aspect of service performance, and component selection must also account for mechanical, thermal and environmental demands (Mallick, 2007; Sapuan, 2017).

Relevant application requirements include tensile and flexural performance, impact resistance, density, moisture response, thermal stability, flammability, wear, ageing and manufacturability. These properties should be evaluated against the requirements of the intended component.

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- Potential applications include dashboards, door panels, interior trims, instrument panels and other non-structural interior components.

Using cassava-processing residue and cow-bone waste as reinforcement offers a potential route for material recovery. The environmental performance of the finished composite would depend on its complete processing and service life (Mohanty et al., 2005).

4. Conclusion

Brinell hardness was evaluated across 18 cassava-cortex/cow-bone epoxy formulations. Three specimens were tested per formulation, and the statistical comparisons in this study use the 18 reported formulation means.

The reported mean hardness values ranged from 21 to 43 BHN, with the highest value obtained for RDT4PS100. This formulation combined 20 vol.% cassava cortex and 20 vol.% cow bone, 4 wt.% sodium hydroxide treatment and 100 μm particles.

Recomputed factor-level mean-based S/N contrasts were 5.28 dB for reinforcement, 0.64 dB for treatment and 0.35 dB for particle size. The latter two are not separately interpretable as causal effects because the experimental factor assignments are partially aliased.

The categorical main-effects regression gave an in-sample R^2 of 99.20%, indicating close agreement between the fitted values and the 18 formulation means. Its predictive accuracy for other compositions and independently prepared specimens remains to be established.

The highest measured mean hardness was 43 BHN for RDT4PS100. The categorical main-effects model yielded 41.8167 BHN for RDT4PS500, an untested combination; this value is therefore a model estimate rather than an experimentally verified optimum. The limited separation of treatment and particle-size settings constrains interpretation of their individual effects.

The comparison identifies a promising balanced hybrid formulation for further investigation in hardness-sensitive, non-structural applications. The principal contribution is the direct comparison of reinforcement composition in this cassava-cortex/cow-bone system, while reliable optimisation of treatment and particle size requires a design with independently varied factors and confirmation tests.

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