
TRIBOLOGICAL BEHAVIOUR AND MULTI-OBJECTIVE OPTIMIZATION OF AUSTEMPERED HIGH-CARBON STEEL USING SULFATE SALT-MODIFIED SESAME OIL

Hafiz Bashir Aminu^{1,2,*}, Rayyan Mamuda Dodo¹, Terver Ause¹

¹Ahmadu Bello University Zaria

²Federal Polytechnic Idah Kogi State

Email: hafizbashir16@gmail.com, rdmamuda@abu.edu.ng, tause@abu.edu.ng

*Corresponding author: Hafiz Bashir Aminu

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ABSTRACT

This study investigates the tribological behaviour and multi-response optimization of austempered high-carbon steel processed using sulfate salt-modified sesame oil as a bio-based quenching medium. Potassium sulfate (K_2SO_4) and sodium sulfate (Na_2SO_4) concentrations, austempering time and temperature were varied using a Taguchi L9 orthogonal array. Wear rate and coefficient of friction (CoF) were evaluated under dry sliding conditions, while scanning electron microscopy (SEM) was used for qualitative microstructural examination. Grey Relational Analysis (GRA) was employed to simultaneously optimize the two smaller-the-better responses. The nine experimental runs produced wear rates of 0.0003 – 0.0036 mm³/Nm and CoF values of 0.048 – 0.118. Run 4, corresponding to 1.5% K_2SO_4 , 0% Na_2SO_4 , 45 min and 320 °C, produced the highest experimentally observed GRG of 0.8519. The Taguchi response-mean analysis predicted a combined optimum of 3% K_2SO_4 , 0% Na_2SO_4 , 45 min and 320 °C, with an estimated GRG of 0.8721. However, this predicted condition was not included in the L9 matrix and therefore remains experimentally unverified. Pooled ANOVA indicated that austempering temperature contributed 47.74% to GRG variation, followed by Na_2SO_4 concentration at 19.92%, although neither retained factor was statistically significant at the 5% level. SEM observations suggested associations between comparatively refined transformation morphologies and favourable tribological responses, but definitive phase identification requires complementary XRD or EBSD analysis. The findings demonstrate the potential of sulfate salt-modified sesame oil for tailoring the combined wear and friction response of austempered high-carbon steel.

Keywords: Austempering; Sesame oil; Sulfate salts; Tribology; Grey Relational Analysis

1. INTRODUCTION

The increasing demand for engineering components capable of operating under severe service conditions has stimulated interest in heat treatment processes that provide a favourable combination of strength, toughness and tribological performance. Austempering is particularly attractive because isothermal transformation of austenite above the martensite-start temperature produces bainitic ferrite and retained austenite, which can improve hardness, toughness, dimensional stability and wear resistance (Bhadeshia, 2015). Such tribological performance is important in gears, shafts, bearings, railway components, mining equipment and automotive parts, where sliding and abrasive interactions can cause material degradation and premature failure (Stachowiak & Batchelor, 2025).

The wear behaviour of bainitic steels depends not only on hardness but also on the morphology, distribution and stability of retained austenite. During sliding, retained austenite may transform to martensite through a deformation-induced transformation, producing a TRIP effect that enhances strain hardening and resistance to material removal (Chen *et al.*, 2024). Chen *et al.* (2025) associated improved wear resistance with strain partitioning between retained austenite and ferritic phases, while Jiang *et al.* (2026) showed that retained-austenite stability influences crack initiation, propagation and transformation-induced strengthening. Controlling these microstructural features during austempering is therefore important for optimizing tribological behaviour.

Quenching medium characteristics strongly influence heat extraction and subsequent phase transformation. Although mineral oils are widely used, environmental, safety and sustainability

concerns have increased interest in vegetable-oil-based quenchants because of their renewability, biodegradability and favourable cooling characteristics. Sesame oil is a promising candidate owing to its favourable fatty-acid composition, relatively high oxidative stability and flash point. However, previous studies have focused predominantly on the thermal, physicochemical, cooling, mechanical and microstructural characteristics of vegetable-oil quenchants, with comparatively limited attention to their modification for improved tribological performance.

Inorganic salt modification provides a potential means of controlling the heat transfer behaviour of vegetable oils. Aminu *et al.* (2025) reported that sulfate salts can modify the thermal behaviour, cooling characteristics and heat transfer efficiency of vegetable-oil-based quenchants. Such changes may affect bainitic transformation, retained-austenite stability and consequently the tribological behaviour of austempered steels. From an industrial perspective, sulfate salt-modified sesame oil could potentially be prepared using existing oil-quenching systems, while its renewable base fluid offers sustainability advantages. However, practical adoption would depend on raw-material and additive costs, salt dispersion and suspension stability, bath maintenance, thermal degradation, reusability, process consistency and equipment compatibility. Thus, although the approach has commercialization potential, pilot-scale testing, repeated-cycle evaluation, life-cycle assessment and techno-economic comparison with conventional mineral oils are required before industrial implementation can be established.

Despite increasing interest in bio-based quenchants, the tribological behaviour of austempered high-carbon steel processed using sulfate salt-modified sesame oil remains insufficiently documented. This study therefore evaluates the effects of K_2SO_4 concentration, Na_2SO_4 concentration, austempering time and temperature on the wear rate and coefficient of friction using a Taguchi L9 orthogonal array. Grey Relational Analysis (GRA) was employed to simultaneously optimize the tribological responses, while Analysis of Variance (ANOVA) quantified the relative contribution of the process parameters. Microstructural examination was used to relate the observed tribological behaviour to the resulting microstructural features.

The novelty of this study lies in integrating sulfate-salt modification of sesame oil, austempering and Taguchi–GRA multi-response optimization to establish the relationship between quenchant composition, austempering parameters and simultaneous control of wear rate and coefficient of friction. The findings provide an initial basis for evaluating sulfate salt-modified sesame oil as a bio-based alternative to conventional petroleum-derived quenchants and for subsequent pilot-scale and commercialization studies.

2. MATERIALS AND METHODS

2.1 Materials

High-carbon steel and *Sesamum indicum* (sesame) oil were used as the workpiece material and base quenchant, respectively. The steel, supplied in flat-spring form, was machined into samples for tribological and microstructural characterization. K_2SO_4 and Na_2SO_4 were used as quenchant modifiers, while standard metallographic consumables and reagents were used for sample preparation.

The chemical composition of the steel is presented in Table 1.

Table 1: Chemical composition of the selected high-carbon steel sample

Elements	C	Si	Mn	Cr	Ni	Cu	V	W	Fe
Compositions (% wt)	1.26	1.20	2.14	2.35	2.80	0.79	0.74	0.20	balance

2.2 Quenchant Preparation and Experimental Design

Sesame oil was preheated to 100 °C before K_2SO_4 and Na_2SO_4 were added at 0, 1.5 and 3.0 wt.%. The mixtures were magnetically stirred for 10 min to promote uniform salt dispersion. The resulting sulfate-modified sesame oils were used as quenching media for austempering.

A Taguchi L9 orthogonal array was employed with four three-level control factors: K_2SO_4 concentration, Na_2SO_4 concentration, austempering time and temperature. The complete experimental matrix is presented with the experimental results in Table 3.

2.3 Heat Treatment of Samples

The as-received samples were normalized at 850 °C for 1 h and air cooled to obtain a relatively homogeneous initial microstructure. Austempering involved austenitizing at 850 °C for 1 h, followed by transfer into the selected sulfate-modified sesame-oil bath maintained at 260, 290 or 320 °C. The samples were held for 30, 45 or 60 min and subsequently air cooled.

The selected temperature range was within the expected bainitic transformation range of the steel. It was also supported by the thermal behaviour of sulfate-modified sesame oil reported by Aminu *et al.* (2025), who observed increased thermal stability following sulfate modification.

2.4 Tribological Testing

Wear rate and coefficient of friction (CoF) were evaluated using a ball-on-disk tribometer (model 50324) under dry-sliding conditions in accordance with ASTM G99 (2020). Samples were prepared to the required dimensions and tested against a 6 mm diameter stainless-steel ball using a normal load of 10 N, sliding speed of 0.1 m/s, test duration of 5 min and wear-track diameter of 8 mm. All samples were cleaned and dried before testing.

CoF was recorded continuously by the tribometer software. Wear rate was determined from the mass loss before and after testing and expressed as volumetric wear per unit load and sliding distance (mm³/Nm):

$$\text{Wear rate (WR)} = \frac{\Delta m}{\rho \cdot F \cdot L} \quad (1)$$

where Δm is mass loss, ρ is material density, F is applied load and L is sliding distance. The identical test duration and conditions were maintained for all samples to permit comparative evaluation. The relatively short test duration should, however, be regarded as a limitation when extrapolating the results to long-term service wear.

2.5 Microstructural Characterization

For SEM examination, samples were sectioned to approximately 10 × 10 × 5 mm, ground using 400–1200 grit SiC papers, polished with 6 and 1 µm diamond pastes, and etched with 2% nital for approximately 30 s. SEM observations were conducted at 1000× magnification.

The micrographs were used to compare morphological features associated with the different heat treatment conditions and to relate microstructural variations to the observed tribological responses. Phase assignments were made cautiously from morphology and heat treatment history; definitive identification of phases such as retained austenite would require complementary techniques such as X-ray diffraction.

2.6 Taguchi–Grey Relational Analysis

The experimental data were analyzed using Taguchi signal-to-noise (S/N) analysis, Grey Relational Analysis (GRA) and ANOVA in Minitab 17. Since both wear rate and CoF are smaller-the-better characteristics, the corresponding S/N criterion was employed:

$$\eta = -10 \log_{10} \left(\frac{1}{n} \sum_{i=1}^n y_i^2 \right) \quad (2)$$

Where η is the S/N ratio, y_i is the observed response and n is the number of observations. As each L9 condition was tested once, $n=1$.

For simultaneous optimization of wear rate and CoF, the responses were normalized using the smaller-the-better criterion:

$$y_i(s) = \frac{\max x_i(s) - x_i(s)}{\max x_i(s) - \min x_i(s)} \quad (3)$$

The grey relational coefficient (GRC) was subsequently calculated from the normalized responses:

$$\epsilon_i(s) = \frac{\Delta_{min} + \varphi \Delta_{max}}{\Delta_{oi}(s) + \varphi \Delta_{max}} \quad (4)$$

$$\Delta_{oi}(s) = |x_o(s) - x_i(s)| \quad (5)$$

where the identification coefficient was set to 0.5. The grey relational grade (GRG) was calculated as the equally weighted mean of the GRCs for wear rate and CoF:

$$GRGi = \frac{1}{n} \sum_{i=1}^n \epsilon_{ij} \quad (6)$$

The factor level producing the highest mean GRG was selected as the optimum level. The predicted optimum GRG was subsequently obtained using the Taguchi additive model (Roy, 2010):

$$\gamma = \mu + \sum_{i=1}^q (\gamma_{i,opt} - \mu) \quad (7)$$

where μ is the overall mean GRG of all N experimental runs and $\gamma_{i,opt}$ is the mean GRG at the selected optimum level of factor i .

2.7 Analysis of Variance (ANOVA)

Pooled ANOVA was used to estimate the relative contributions of K₂SO₄ concentration, Na₂SO₄ concentration, austempering time and temperature to wear rate, CoF and GRG. The sum of squares (SS), mean square (MS) and percentage contribution were calculated using:

$$SS = \sum_{i=1}^L \frac{T_i^2}{n_i} - \frac{T^2}{N} \quad (8)$$

$$MS = \frac{SS}{DOF} \quad (9)$$

$$P(\%) = \frac{SS_i}{SS_T} \times 100 \quad (10)$$

The percentage contribution of each factor was calculated from its sum of squares relative to the total sum of squares. Because the L9 array contained no experimental replication, the ANOVA results were interpreted primarily as estimates of the relative contribution of the process parameters rather than as definitive tests of statistical significance. The Taguchi–ANOVA procedure followed the established methodology described by Roy (2010).

3. RESULTS AND DISCUSSION

3.1 Tribological Performance

The tribological results (Tables 2 and 3) demonstrate substantial improvement following quenching and austempering compared with the normalized condition. The normalized steel exhibited a wear rate of 0.0096 mm³/Nm and CoF of 0.279, compared with 0.0005 mm³/Nm and 0.220, respectively, after water quenching. Raw sesame-oil quenching produced still lower values of 0.0001 mm³/Nm and 0.127, indicating the favourable tribological response achievable with sesame-oil heat treatment.

Table 2: Wear rate and coefficient of friction tests for normalized, water-quenched and raw sesame oil-quenched samples

Experiments	Wear rate (mm ³ /Nm)	Coefficient of friction
Normalized	0.0096	0.279
Water quenched	0.0005	0.220
Raw sesame oil	0.0001	0.127

3.1.1 Wear test

The wear rate of the nine austempered conditions ranged from 0.0003 to 0.0036 mm³/Nm, with the minimum occurring at Run 1 and the maximum at Run 5. All austempered conditions therefore exhibited lower wear rates than the normalized steel (0.0096 mm³/Nm), although the raw sesame-oil reference produced the lowest value (0.0001 mm³/Nm).

The improved wear resistance is associated with the development of bainitic ferrite and retained-austenite-rich regions during isothermal transformation. Bainitic ferrite provides a strong load-bearing framework, while retained austenite can accommodate deformation and undergo transformation-induced strengthening during sliding (Zhao, 2025). This agrees with Chen *et al.* (2024), who linked improved wear resistance in bainitic steels to the synergistic effects of bainitic ferrite and retained austenite.

3.1.2 Coefficient of friction (CoF) test

The coefficient of friction values obtained from the tribological tests are presented in Table 3. CoF varied from 0.048 to 0.118 among the austempered conditions, with the minimum recorded at Run 5 and the maximum at Run 1 (Table 3). The lower friction observed under some conditions may be associated with retained-austenite-rich regions, which possess greater deformability and can accommodate localized deformation, thereby reducing interfacial stress concentrations during sliding (Chen *et al.*, 2025).

Table 3: L9 orthogonal array layout showing results for tribology tests (wear rate and coefficient of friction) for austempered samples

Run	Factors				Responses	
	A: K ₂ SO ₄ (%)	B: Na ₂ SO ₄ (%)	C: Austemp. Time (min)	D: Austemp. Temp. (°C)	WR (mm ³ /Nm)	CoF
1	0	0	30	260	0.0003	0.118
2	0	1.5	45	290	0.0014	0.088
3	0	3	60	320	0.0007	0.081
4	1.5	0	45	320	0.0004	0.059
5	1.5	1.5	60	260	0.0036	0.048
6	1.5	3	30	290	0.0010	0.107
7	3	0	60	290	0.0005	0.094
8	3	1.5	30	320	0.0008	0.074
9	3	3	45	260	0.0004	0.069

3.2 Taguchi analysis signal-to-noise ratio (S/N) for tribological responses

3.2.1 Wear rate response

The Taguchi S/N analysis (Table 4) identified Na₂SO₄ concentration as the most influential factor on wear rate, followed by K₂SO₄ concentration, austempering time and temperature. The

optimum levels for minimizing wear rate were 1.5% K₂SO₄, 1.5% Na₂SO₄, 60 min and 290 °C, as indicated by the main-effect plot (Figure 1).

Table 4: Response for S/N ratios (wear rate vs K₂SO₄, Na₂SO₄, austempering time and austempering temperature

Level	K ₂ SO ₄ (%)	Na ₂ SO ₄ (%)	Austempering Time (min)	Austempering Temperature (°C)
1	63.54	68.15	64.13	62.43
2	58.94	55.96	64.33	61.03
3	65.31	63.69	59.33	64.33
Delta	6.36	12.18	5.00	3.30
Rank	2	1	3	4

*Bold value signifies to show the level at which the optimum condition is achieved for each process parameter.

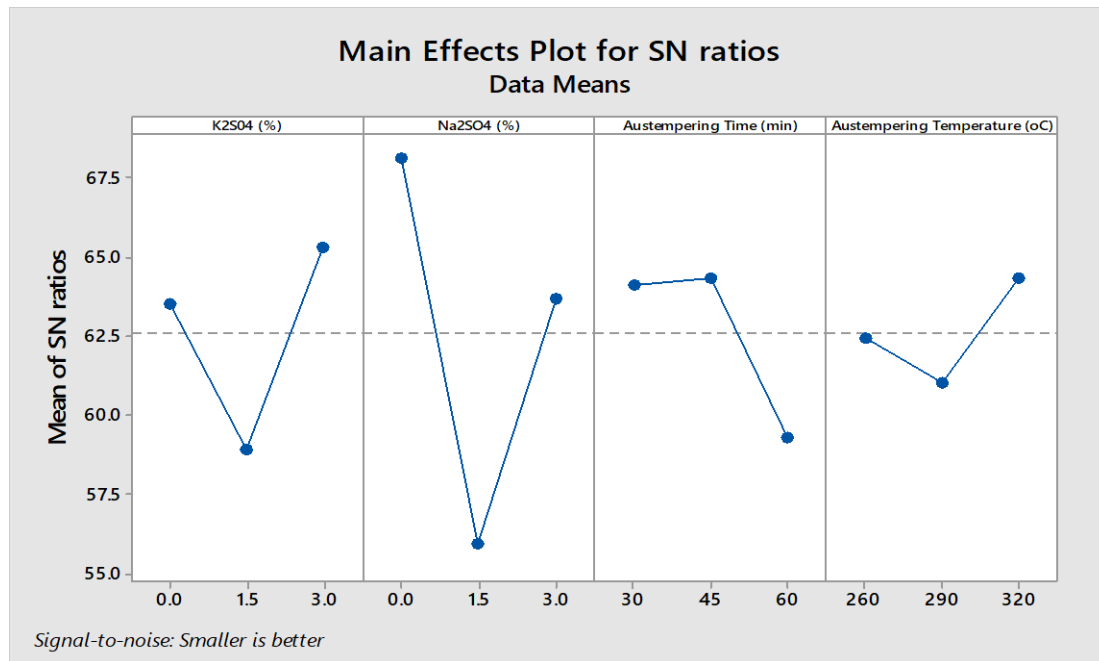


Figure 1: The main effect plots for S/N ratios on wear rate (Taguchi)

3.2.2 Coefficient of friction (CoF) response

For CoF, austempering time was the most influential parameter, followed by temperature, Na₂SO₄ concentration and K₂SO₄ concentration (Table 5). The optimum condition for minimizing CoF was 0% K₂SO₄, 3% Na₂SO₄, 30 min and 290 °C (Figure 2). The stronger influence of holding time suggests that completion and development of the bainitic transformation substantially affect the friction response, while salt concentration modifies the cooling behaviour and consequent microstructural development. Similar time-dependent effects have been reported for austempered niobium-alloyed ductile iron (Garcia *et al.*, 2025).

Table 5: Response for S/N ratios (coefficient of friction vs K_2SO_4 , Na_2SO_4 , austempering time and austempering temperature)

Level	K_2SO_4 (%)	Na_2SO_4 (%)	Austempering Time (min)	Austempering Temperature ($^{\circ}C$)
1	20.50	21.23	18.93	22.72
2	22.19	23.37	22.97	19.08
3	22.13	20.22	22.91	23.01
Delta	1.68	3.15	4.05	3.93
Rank	4	3	1	2

*Bold value signifies to show the level at which the optimum condition is achieved for each process parameter.

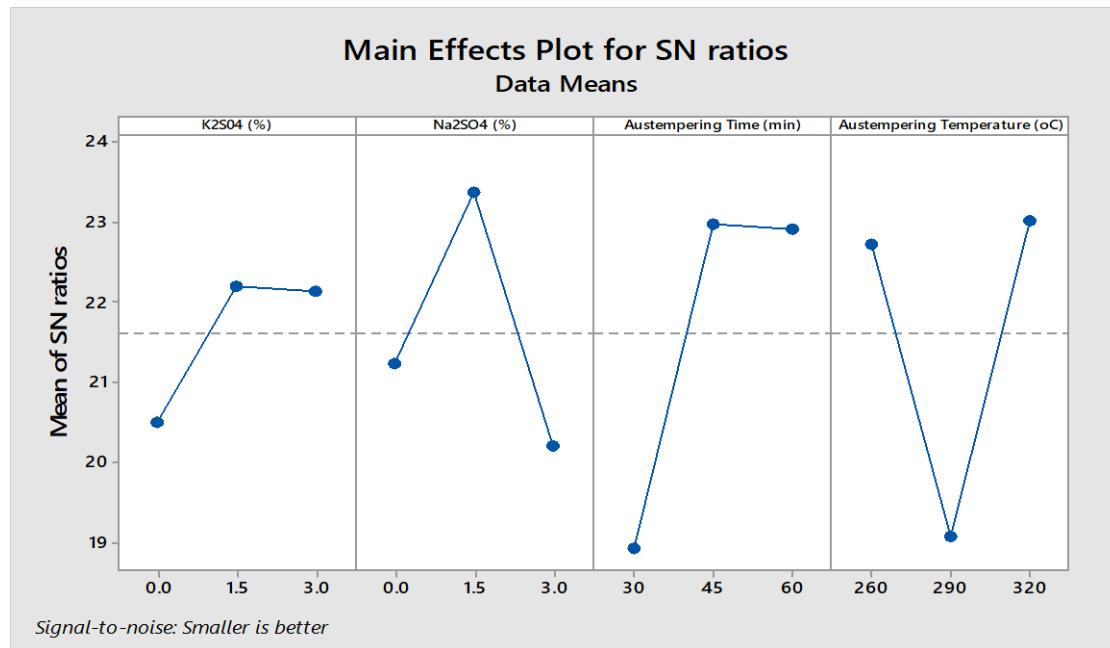


Figure 2: The main effect plots for S/N ratios on coefficient of friction (Taguchi)

3.3 Analysis of Variance (ANOVA) for Tribological Responses

Pooled ANOVA was applied to the L9 experimental results to estimate the relative contributions of the four process parameters to the wear-rate and CoF S/N responses. Because the design was not replicated, the factors with the smallest contributions were pooled to provide an error estimate following the Taguchi approach (Roy, 2010).

Table 6: Pooled ANOVA for S/N ratio of wear rate and coefficient of friction

Factor	Wear contribution (%)	F	p	CoF contribution (%)	F	p
K_2SO_4	18.12	2.01	0.249	26.75	1.26	0.377
Na_2SO_4	63.81	7.06	0.047	Pooled	—	—
Time	Pooled	—	—	30.70	1.44	0.337
Temperature	Pooled	—	—	Pooled	—	—
Error	18.07	—	—	42.55	—	—

3.3.1 Wear rate response

The pooled ANOVA results (Table 6) show that Na_2SO_4 concentration was the dominant contributor to the wear-rate S/N response (63.81%), followed by K_2SO_4 concentration (18.12%). Na_2SO_4 was the only retained factor reaching the adopted 95% significance criterion ($F = 7.06$, $p = 0.047$), whereas K_2SO_4 was not statistically significant ($F = 2.01$, $p = 0.249$). Austempering time and temperature were pooled as error, accounting for 18.07%.

The strong contribution of Na_2SO_4 suggests that quenchant chemistry had a greater influence on wear behaviour than the investigated austempering parameters within the experimental range. This may arise from changes in cooling behaviour and the resulting transformation products; however, the specific microstructural mechanisms require further investigation.

3.3.2 Coefficient of friction response

For CoF (Table 6), austempering time contributed 30.70%, followed by K_2SO_4 concentration (26.75%). However, neither factor was statistically significant at the 95% confidence level (time: $F = 1.44$, $p = 0.337$; K_2SO_4 : $F = 1.26$, $p = 0.377$). Na_2SO_4 concentration and temperature were pooled into the error term, which accounted for 42.55% of the observed variation.

Although statistical significance was not established, the higher percentage contributions of austempering time and K_2SO_4 indicate their greater relative influence within the tested parameter space and may therefore be useful for process control (Roy, 2010). The relatively large pooled error may reflect experimental variability, unaccounted interactions or the limitations of the unreplicated L9 design. Similar limitations have been noted in Taguchi-based tribological investigations (Asha *et al.*, 2024). Future studies using replication or a larger orthogonal array such as L27 could better resolve interactions and experimental variability (Ramalingam *et al.*, 2022).

3.4 Grey Relational Analysis (GRA)

Grey Relational Analysis (GRA) was used to combine wear rate and coefficient of friction (CoF) into a single performance indicator. As shown in Table 7, Run 4 produced the highest grey relational grade (GRG) of 0.8519, indicating the best overall balance between the two tribological responses among the nine experimental conditions. According to Kuo *et al.* (2011), the highest GRG represents the best combined performance across the selected responses.

The need for multi-response optimization is evident from the contrasting individual responses. Run 1 produced the lowest wear rate but relatively high CoF, whereas Run 5 produced the lowest CoF but the highest wear rate. Their identical GRG of 0.6667 despite different individual responses illustrates the trade-off between wear resistance and friction reduction. Thus, optimization of either response independently may not provide the most suitable overall condition. Run 4, corresponding to 1.5% K_2SO_4 , 0% Na_2SO_4 , 45 min and 320 °C, provided the most balanced response. Similar findings have been reported in Taguchi-GRA tribological studies, where the multi-response optimum differs from the optimum of individual responses (Ibrahim *et al.*, 2022; Altas, 2024).

Table 7: GRA integrate multiple responses (wear rate and CoF)

Runs	Norm - WR	Norm - CoF	Δ -WR	Δ -CoF	GRC - WR	GRC - CoF	GRG	Rank
1	1.0000	0.0000	0.0000	1.0000	1.0000	0.3333	0.6667	5
2	0.6667	0.4286	0.3333	0.5714	0.6000	0.4667	0.5333	9
3	0.8788	0.5286	0.1212	0.4714	0.8049	0.5148	0.6598	7
4	0.9697	0.8429	0.0303	0.1571	0.9429	0.7609	0.8519	1
5	0.0000	1.0000	1.0000	0.0000	0.3333	1.0000	0.6667	5
6	0.7879	0.1571	0.2121	0.8429	0.7021	0.3723	0.5372	8
7	0.9394	0.3429	0.0606	0.6571	0.8919	0.4320	0.6619	4
8	0.8485	0.6286	0.1515	0.3714	0.7675	0.5737	0.6706	3
9	0.9697	0.7000	0.0303	0.3000	0.9429	0.6250	0.7839	2

3.4.1 Effect of process parameters on GRG

The mean GRG response (Table 8) ranked austempering temperature as the most influential parameter, followed by Na_2SO_4 concentration, austempering time and K_2SO_4 concentration. Temperature had the largest delta (0.1500), with the optimum level being 320 °C, which produced a mean GRG of 0.7274, compared with 0.7058 at 260 °C and 0.5775 at 290 °C (Figure 3). The superior response at 320 °C may be associated with more favourable transformation conditions and phase development; however, this interpretation remains inferential because phase fractions were not quantitatively determined by techniques such as XRD or EBSD.

Na_2SO_4 had the second-largest influence ($\Delta = 0.1033$), with 0% producing the highest mean GRG (0.7268), followed by 3% (0.6603) and 1.5% (0.6235). The non-monotonic response suggests that salt concentration does not continuously improve the combined tribological performance and may modify quenchant cooling characteristics and subsequent transformation behaviour (Aminu *et al.*, 2025).

Austempering time ranked third ($\Delta = 0.0982$), with 45 min giving the highest mean GRG (0.7230), compared with 0.6248 and 0.6628 at 30 and 60 min, respectively. This intermediate optimum suggests a favourable transformation window within the investigated conditions. K_2SO_4 had the smallest delta (0.0855) but showed a progressive increase in mean GRG from 0.6199 at 0% to 0.6853 at 1.5% and 0.7055 at 3%, making 3% its optimum level.

Table 9: Response for means (GRG vs K_2SO_4 , Na_2SO_4 , austempering time and austempering temperature)

Level	K_2SO_4 (%)	Na_2SO_4 (%)	Austempering Time (min)	Austempering Temperature (°C)
1	0.6199	0.7268	0.6248	0.7058
2	0.6853	0.6235	0.7230	0.5775
3	0.7055	0.6603	0.6628	0.7274
Delta	0.0855	0.1033	0.0982	0.1500
Rank	4	2	3	1

*Bold value signifies to show the level at which the optimum condition is achieved for each process parameter.



Figure 3: The main effect plots for means on GRG (Taguchi)

3.4.2 Predicted Optimum Condition

The best experimental condition and the predicted Taguchi optimum should be distinguished. Run 4 was the best of the nine tested conditions, with a GRG of 0.8519 at 1.5% K₂SO₄, 0% Na₂SO₄, 45 min and 320 °C. However, combining the optimum level of each factor from the GRG response (Table 9) gives the predicted condition 3% K₂SO₄ + 0% Na₂SO₄ + 45 min + 320 °C (A₃B₁C₂D₃).

Using the Taguchi additive model, the predicted GRG was 0.8721, approximately 2.37% higher than the highest experimentally observed GRG (0.8519). Because this combination was not included in the L9 array, the predicted improvement remains an unverified model estimate rather than demonstrated experimental improvement. Confirmation testing is therefore required to establish the predictive adequacy of the model.

Table 9: Mean GRG for optimum levels

Parameter	Optimum level	Mean GRG
K ₂ SO ₄ concentration	3%	0.7055
Na ₂ SO ₄ concentration	0%	0.7268
Austempering time	45 min	0.7230
Austempering temperature	320°C	0.7274

3.4.3 ANOVA for GRG

The pooled ANOVA (Table 10) identified austempering temperature as the largest contributor to GRG (47.74%), followed by Na₂SO₄ concentration (19.92%), while K₂SO₄ concentration and austempering time were pooled as error, accounting for 32.34%. This ranking agrees with the GRG response analysis, which also identified temperature as the most influential factor.

Although temperature had a higher F-value (2.952) than Na₂SO₄ (1.232), neither factor was statistically significant at the 5% level under the pooled-error model ($p = 0.163$ and 0.383 , respectively). The relatively large pooled error and absence of replication limit the strength of statistical inference; therefore, these results should primarily be interpreted as estimates of relative parameter contribution within the investigated experimental range.

Table 10: Pooled ANOVA for grey relational grade

Factor	DOF	Sum of Squares (SS)	Mean Square (MS)	F-value	p-value	Contribution (%)
B: Na ₂ SO ₄ (%)	2	0.01645	0.00823	1.232	0.383	19.92
D: Austemp. temp(°C)	2	0.03942	0.01971	2.952	0.163	47.74
Error (A + C pooled)	4	0.02671	0.00668	----	----	32.34
Total	8	0.08257	----	----	----	100.00

3.5 Microstructure–Property Correlation

SEM micrographs (Plates I – X) obtained at 1000× magnification revealed differences in the morphology, distribution and apparent refinement of the transformation products produced by the various heat treatment conditions. Phase identification from SEM morphology is qualitative; definitive identification and quantification, particularly of retained austenite, would require complementary techniques such as XRD or EBSD.

The normalized sample (Plate I) exhibited a relatively coarse ferrite–pearlite morphology, providing a reference for comparison with the austempered conditions and consistent with its comparatively poor wear resistance (Bhadeshia & Honeycombe, 2017). The austempered samples generally exhibited finer acicular transformation features (Plates II – X), morphologically consistent with bainitic transformation (Bhadeshia, 2015). Differences in feature size, uniformity and distribution were observed among the processing conditions, reflecting variations in transformation behaviour (Wang *et al.*, 2024a, 2024b).

Notably, Plate V (Run 4), which produced the highest experimental GRG (0.8519), exhibited comparatively refined and homogeneous transformation features. Such morphology may promote more uniform load distribution and resistance to localized plastic deformation during sliding, thereby contributing to its favourable combined wear and friction response. In contrast, relatively coarser or heterogeneous morphologies observed in Plates III, VI, VII and X may promote localized deformation and stress concentration, increasing susceptibility to wear damage (Bhadeshia & Honeycombe, 2017; Stachowiak & Batchelor, 2025).

Overall, the SEM observations qualitatively support the tribological results: better-performing conditions generally exhibited comparatively refined and homogeneous transformation morphologies, whereas less favourable responses were associated with coarser or less uniform features. However, this correlation should be regarded as qualitative because the study did not quantitatively determine phase fractions or retained-austenite stability.

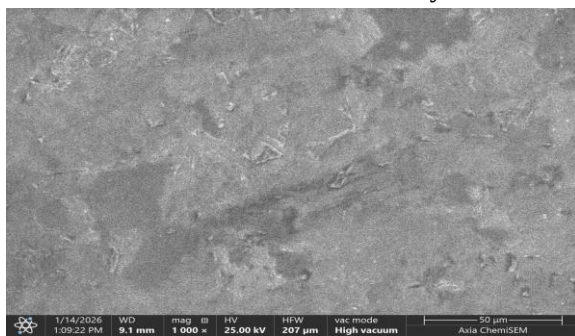


Plate I: SEM micrograph of normalized high-carbon steel at 1000×

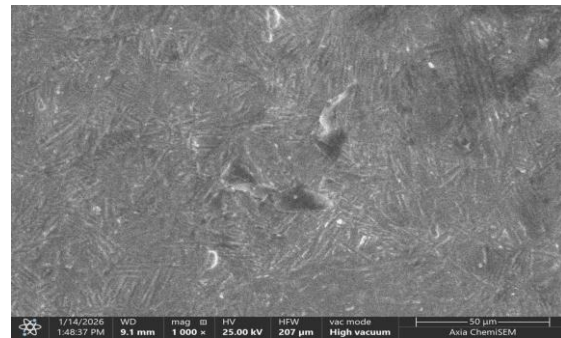


Plate II: SEM micrograph of austempered sample, Run 1, at 1000×

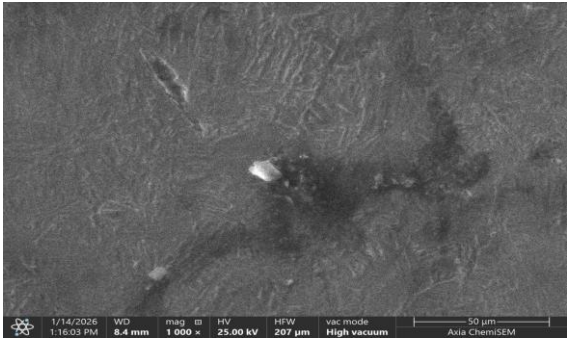


Plate III: SEM micrograph of austempered sample, Run 2, at 1000×

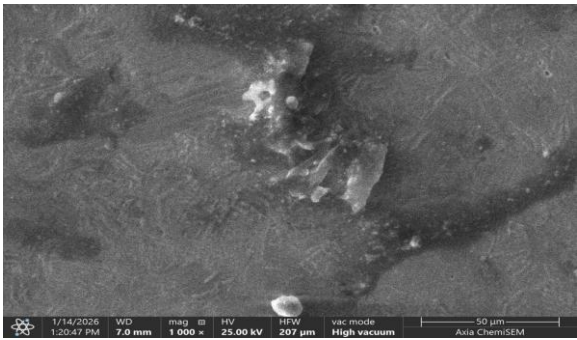


Plate IV: SEM micrograph of austempered sample, Run 3, at 1000×

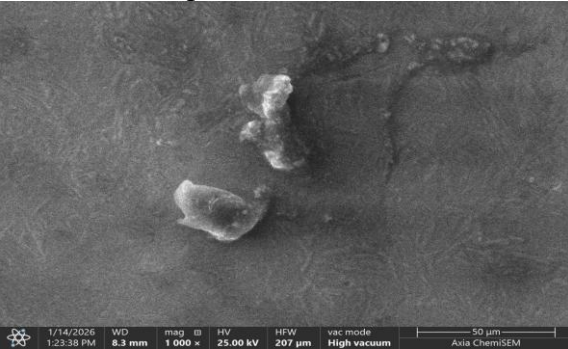


Plate V: SEM micrograph of austempered sample, Run 4, at 1000×



Plate VI: SEM micrograph of austempered sample, Run 5, at 1000×

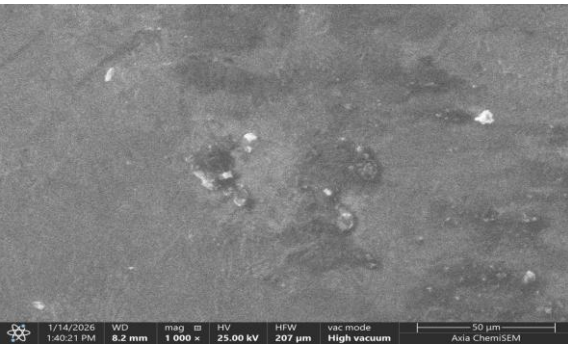


Plate VII: SEM micrograph of austempered sample, Run 6, at 1000×



Plate VIII: SEM micrograph of austempered sample, Run 7, at 1000×



Plate IX: SEM micrograph of austempered sample, Run 8, at 1000×

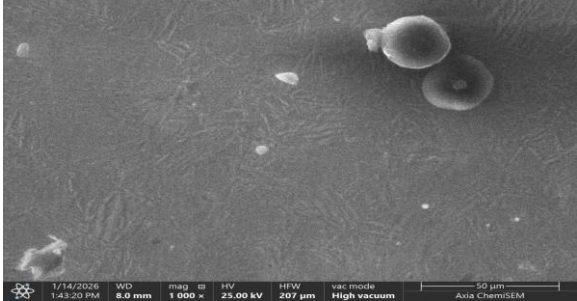


Plate X: SEM micrograph of austempered sample, Run 9, at 1000×

4. CONCLUSION

This study evaluated the tribological behaviour and multi-response optimization of austempered high-carbon steel using K_2SO_4 - and Na_2SO_4 -modified sesame oil. The principal conclusions are:

1. Austempering substantially improved tribological performance relative to the normalized condition, with the investigated sulfate-modified sesame-oil conditions generally producing lower wear rates and coefficients of friction under the specified dry-sliding conditions.
2. Single-response Taguchi optimization produced different parameter combinations. Minimum wear rate was associated with 1.5% K_2SO_4 , 1.5% Na_2SO_4 , 60 min and 290 °C, whereas minimum CoF corresponded to 0% K_2SO_4 , 3% Na_2SO_4 , 30 min and 290 °C, confirming the competing nature of the two tribological responses.
3. GRA provided a balanced multi-response solution. The highest experimentally observed GRG (0.8519) was obtained at 1.5% K_2SO_4 , 0% Na_2SO_4 , 45 min and 320 °C (Run 4). The Taguchi-GRA response analysis predicted 3% K_2SO_4 , 0% Na_2SO_4 , 45 min and 320 °C, with an estimated GRG of 0.8721. Since this combination was not included in the L9 array, experimental confirmation is required.
4. Austempering temperature was the largest contributor to GRG variation (47.74%), followed by Na_2SO_4 concentration (19.92%). However, neither factor was statistically significant at the 5% level under the pooled-error model; therefore, the contribution values should be interpreted as relative influences within the present L9 dataset.
5. SEM observations qualitatively supported the tribological trends, with better-performing conditions generally exhibiting comparatively refined and homogeneous transformation morphologies. Definitive phase identification and retained-austenite quantification would require complementary XRD or EBSD analysis.

5. LIMITATIONS AND FUTURE WORK

The study was limited to a single high-carbon steel, three levels of each sulfate concentration, three austempering temperatures and three holding times using an unreplicated L9 design. The 5-min tribological test duration, absence of independent replication and lack of confirmation testing limit extrapolation to long-term service performance. In addition, SEM provided qualitative rather than quantitative phase characterization.

Future work should prioritize replicated confirmation testing of the predicted condition (3% K_2SO_4 , 0% Na_2SO_4 , 45 min and 320 °C), followed by longer-duration tribological testing under varied loads and sliding speeds. XRD/EBSD and worn-surface SEM/EDS should be employed to clarify phase constitution and wear mechanisms. For industrial implementation, the thermal and suspension stability, quenchant reusability, additive availability, cost and scalability of the modified sesame-oil system should also be evaluated.

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