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Gravimetric and data analysis of the corrosion resistance behaviour and inhibition of C26000 brass in dilute HNO₃ solution

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Abstract. The corrosion resistance of as-received and heat treated C26000 brass (annealed and quenched) was evaluated by gravimetric analysis in 0.5 M, 1 M, 1.5 M, 2 M, 2.5 M, 3 M and 3.5 M HNO₃ solution. Generally, corrosion rate of C26000 increased with increase in HNO₃ concentration and varies significantly with respect to observation time. Results showed that heat treatment has no influence on the corrosion resistance of the brass heat treated brass compared to the as-received sample. Application of garlic extracts in HNO₃ solution at 0.5 M significantly decreased the corrosion rate of C26000 60% concentration with inhibition value of 79.46%. The garlic extract exhibited significant concentration dependent behaviour and its inhibition performance varies with observation time. Statistical evaluation showed the standard deviation values of as-received and heat treated C26000 varied with respect to mean inhibition data. However, variation increased with HNO₃ concentration due to thermodynamic instability with respect to observation time. In the presence of the garlic extracts, the high standard deviation values at the lowest and highest garlic concentration signify significant variation with observation time and thermodynamic instability. Evaluation through analysis of variance shows HNO₃ concentration and observation are statistically irrelevant to the corrosion rate output of as-received and heat treated C26000. In the presence of garlic extracts only the extract concentration is statistically relevant with value of 90.52%.

1. Introduction

Corrosion is the devolution of the electrochemical, physical, mechanical and metallurgical properties of metallic and non-metallic materials due to intermolecular reactions between the materials and the corrosive anions of their operating environments. Corrosion is more prevalent on metallic alloys due to the high tendency of the metallic surface to form alternating electrochemical cells were oxidation-reduction reactions predominates. The consequences of corrosion are enormous, diverse and in most cases complicated ranging from component damage in mechanical systems to industrial shutdowns and huge economic and financial losses. Significant reduction in metal thickness, evolution of destructive pits and collapse of structures have been the major cause of toxic seepage chemicals, high preservation cost and injury to industry operators. According to Electric Power Research Institute corrosion damage to the power generating establishment totals $17 billion with an estimated 22% of the being avoidable [1]. The foremost corrosion association in America (National Association of Corrosion Engineers) proved in a report that corrosion damage constitutes about 4% of the United States Gross National Product [2]. Globally, corrosion cost the world economy 4% of its Gross

Domestic Product ($2.5 trillion). Alloys of Cu are broadly utilized for the manufacture and construction condenser tubing, desalination units, parts of heat exchangers, hydraulic lines and piping material due to their unique mechanical and aesthetic attributes. The fabricability, thermal, electrical and antiseptic and corrosion resistant of Cu alloys is responsible for their ever increasing applications in most cases replacing ferrous alloys [3-6]. In mildly corrosive conditions the resistance of Cu alloys results from initiation and partial growth of an inert, resilience and transparent film on the alloy. However, in astringent temperature conditions coupled with the presence of corrosive anions the vulnerability of Cu alloys to degradation becomes apparent. Though this is significantly dependent on the concentration of corrosive anions, the relative constituents of Cu alloys, temperature considerations, flow rapidity etc. [7-11]. Cu alloy tends to be reactive in the presence of Cl^- anions resulting in localized deterioration of the alloy [12-17]. The alloys applied in petrochemical refineries and desalination plants tends to fail due to formation of corrosion pits [18-22]. C26000 (alpha brass) Cu alloys are utilized in applications substantially dangerous to ferrous alloys though there are conditions where corrosive anions selectively degrade Cu and Zn in C26000 Cu alloy [23-25]. Application of chemical compounds identified as corrosion inhibitors have been known to significantly reduce corrosion of metallic alloys [26-35]. This research focusses on the effect of metallurgical variation by heat treatment on C26000 corrosion in specific concentrations of HNO_3 solution, while further analysis of C26000 electrochemical behaviour was studied in HNO_3 at specific concentrations of garlic extracts.

2. Experimental methods

2.1 Materials preparation
C26000 brass alloy (C26) rod was acquired in Lagos, Nigeria and analysed in the Materials Characterization Laboratory at Covenant University, Ota, Ogun State, Nigeria. The elemental components of C26 are outlaid in Table 1. C26 was cut and machined into 3 sets of experimental exhibits with average dimension of 1 cm diameter and 2 cm length. The first main set consist of 24 exhibits of C26 which were further subdivided into 3 sets of 8 exhibits each. The first subset of C26 exhibits were subjected to heat-treatment (annealing) in a muffle furnace up to 425°C before gradual cooling to 37 °C ambient temperature. The second subset of C26 exhibits were subjected to heat-treatment (quenching) to 425 °C and instantaneously cooled to 37 °C ambient temperature. The second set main consists of 7 exhibits of C26 while the third set consists of 5 exhibits of C26. All the C26 exhibits were grinded and smoothened with emery papers for gravimetric analysis. Potassium gluconate (PG), a crystalline white powder was secured from Sigma Aldrich, USA. 1Kg of garlic (GLC) was cut into numerous smaller slices and immersed with 2.5 L of C_2H_5OH for 576 h after which the GLC slices were sieved to retain the solution of C_2H_5OH and GLC. The solution was segregated with a rotary evaporator which removed C_2H_5OH from the solution and retaining the garlic extract.

Table 1. Elemental constituents (wt. %) of C26000 Cu alloy

C26000						
Element Symbol	Pb	Zn	Fe	Ni	Mn	Cu
% Composition	0.07	28.38	0.05	-	-	71.5

2.2 Gravimetric analysis
The first main set of C26 exhibits consisting of 3 subsets separately underwent gravimetric analysis in 0.5 M, 1 M, 1.5 M, 2 M, 2.5 M, 3 M and 3.5 M HNO_3 solution for 504 h while measurements were taking every 24 h. The second main set of C26 exhibits underwent gravimetric analysis in 0%, 0.5%, 1%, 1.5%, 2%, 2.5% and 3% PG cubic concentration per 200 ml of 0.5 M HNO_3 solution for 312 h. The third set of C26 exhibits underwent gravimetric analysis in 20%, 40%, 60% and 80% of GLC

extract per 200 ml of 0.5 M HNO_3 solution for 384 h. Corrosion rate, C_R (mm/y) for C26 was enumerated from the equation below;

$$C_R = \left[\frac{87.6W}{DAt}\right] \qquad (1)$$

W illustrates weight loss (g), D illustrates density (g/cm^3), A illustrates total surface area of C26 exhibit (cm^2), 87.6 illustrates corrosion rate constant and t illustrates time (h). The protection performance (P_P) of the PG and GLC were enumerated from the equation below;

$$P_P = \left[\frac{W_1 - W_2}{W_1}\right] \times 100 \qquad (2)$$

W_1 and W_2 illustrates weight loss of C26 at specific PG and GLC concentrations.

2.3 Statistical analysis
Binary-factor single level Anova analysis (F - test) was applied to calculated the statistical importance of HNO_3, PG and GLC concentrates and observation time on PG and GLC performance output, and corrosion rate data in the presence of HNO_3 concentrations (0.5 M to 3.5 M HNO_3). The test was done at confidence level of 95% (significance level of $\alpha = 0.05$) according to the equations below. The summation of column squares (observation time) was enumerated as follows;

$$SS_c = \frac{\sum T_c^2}{nr} - \frac{T^2}{N} \qquad (3)$$

The summation of row squares (HNO_3, PG and GLC concentrations) was enumerated from the equation below;

$$SS_r = \frac{\sum T_r^2}{nc} - \frac{T^2}{N} \qquad (4)$$

Total summation of squares equals

$$SS_{Total} = \sum x^2 - \frac{T^2}{N} \qquad (5)$$

3. Results and discussion

3.1 Gravimetric analysis
Tables 2 and 3 shows the corrosion rate data for untreated, annealed and quenched C26 Cu alloy in 0.5 M – 3.5 M HNO_3 solution with respect to 504 h of observation time. Observation of the corrosion rate data for untreated C26 shows that at 504 h increase in HNO3 concentration results in increase in corrosion of untreated C26 due to oxidation of C26 by NO_3^- ions according to the equations below;
$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 2NO + 4H_2O$
$3Zn + 8HNO_3 \rightarrow 3Zn(NO_3)_2 + 2NO + 4H_2O$
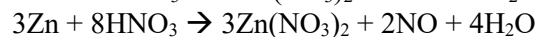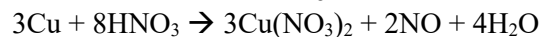
Increase in concentration of HNO_3 accelerated the corrosion of untreated C26 resulting in higher surface oxidation and degradation of the alloy. However, it must be noted that untreated C26 exhibits differential reaction to variation in HNO_3 corrosion with respect to observation time. At 0.5 M HNO_3, corrosion rate of untreated C26 at 24 h is 4.37 mm/y. This value decreased to 0.64 mm/y at 504 h. However, from 1 M to 2 M HNO_3 concentration the corrosion rates of untreated C26 initiated at 2.03 mm/y, 2.78 mm/y and 2.78 mm/y (24 h) and progressively increased to 3.99 mm/y, 4.8 mm/y and 5.92 mm/y at 504 h whereas from 2.5 M to 3.5 M HNO_3 concentration the corrosion rate of untreated C26 initiated at 12.31 mm/y, 21.34 mm/y and 31.23, and decreased to 7.79 mm/y, 8.21 mm/y and 7.57 mm/y at 504h. Annealing C26 altered its metallurgical structure, hence it corrosion reaction behaviour as shown in the corrosion rate data in Table 2. The corrosion rate values of annealed C26 alloy at 24 h of observation time significantly differs from the values obtained for untreated C26 whereas at 504 h variation of annealed corrosion rate values with HNO_3 concentration and observation time are quite similar to the observation of untreated C26 showing that annealing has limited or negligible influence on the corrosion behaviour of annealed C26 compared to untreated C26. The corrosion rate values of

quenched C26 in Table 3 exhibits similar observation showing as earlier confirmed that heat treatment does not influence the corrosion behaviour of C26 Cu alloy.

Further analysis of the corrosion behaviour of C26 Cu alloy was performed by assessing the corrosion protection behaviour of GLC extracts on C26 Cu alloy in 0.5 M HNO_3 solution for 312 h as shown in Table 4. The performance of GLC on C26 at 312 h is marginal with optimum corrosion protection value of 79.46% at 60% GLC concentration. Observing the inhibition performance of GLC with respect to observation time shows that at 24 h of exposure, GLC extract effectively inhibited the corrosion of C26 with values of 99.7%, 106.6%, 103.6% and 102.85% at 20%, 40%, 60% and 80% GLC concentration. The values decreased progressively to 60.41%, 67.20%, 79.46% and 58.63% signifying significant variation with respect to observation time whereas inhibition performance did not vary proportionally with GLC concentration. GLC extract exhibited peak performance from 40% to 60% concentration. Secondly, the progressive decrease in protection performance value shows GLC has weak adsorption properties and its corrosion inhibition behaviour is thermodynamically unstable.

Table 2. Corrosion rate data of untreated and annealed C26 Cu alloy in specific concentrations of HNO_3 solution with respect to observation time

HNO₃ Conc. (M) Observation Time (h)	Untreated C26							Annealed C26						
	0.5 M	1 M	1.5 M	2 M	2.5 M	3 M	3.5 M	0.5 M	1 M	1.5 M	2 M	2.5 M	3 M	3.5 M
24	4.37	2.03	2.78	2.78	12.31	21.34	31.23	0.04	1.89	1.43	8.35	11.39	6.19	29.28
48	2.45	2.3	1.81	3.43	11.05	20.13	34.39	0.04	1.29	1.48	8.56	12.06	9.57	30.42
72	1.66	2.28	1.99	4.61	12.55	21.34	32.19	0.25	1.32	1.57	9.69	12.13	11.74	26.91
96	1.26	2.43	2.29	5.45	12.7	21.55	29.64	0.26	1.06	1.52	10.37	12.66	12.83	25.15
120	1.03	2.31	2.35	7.28	14.15	19.92	26.85	0.35	0.95	1.77	10.32	12.1	12.86	23.17
144	0.87	2.44	2.47	6.84	13.47	20.1	24.35	0.4	0.81	1.76	10.29	11	13.2	21.12
168	0.75	3.09	2.91	7.96	15.37	19.28	22.35	0.42	1.01	2.19	10.37	10.36	13.75	19.53
192	0.66	2.92	3.29	7.53	14.3	17.9	19.88	0.38	1.15	2.39	9.86	10.04	13.33	18.11
216	0.59	3.13	3.78	7.19	13.56	16.85	17.67	0.46	1.21	2.74	9.67	10.76	13.15	17.23
240	0.54	3.17	3.42	7.01	12.74	16.4	15.9	0.44	1.26	3.56	9.57	11.01	12.83	16.5
264	0.52	3.38	3.41	6.8	12.2	15.67	14.46	0.52	1.44	3.78	9.45	10.96	12.52	15
288	0.49	3.64	3.69	6.59	11.56	14.36	13.25	0.52	1.52	4.07	9.06	10.57	12.12	13.75
312	0.46	3.52	3.57	6.21	10.92	13.26	12.23	0.52	1.66	4.37	8.77	10.54	11.7	12.69
336	0.46	3.56	3.77	6.14	10.41	12.31	11.36	0.54	1.75	4.36	8.62	11.43	11.45	11.79
360	0.43	3.75	3.71	6.04	9.95	11.49	10.6	0.54	1.88	4.36	8.51	10.67	10.69	11
384	0.42	3.74	3.8	5.87	9.48	10.77	9.94	0.59	1.91	4.39	8.13	10.01	10.02	10.31
408	0.43	3.69	5.61	5.67	9.08	10.14	9.34	0.59	2.11	4.33	7.82	9.42	9.43	9.71
432	0.41	3.57	4.69	5.78	8.76	9.58	8.83	0.61	2.27	4.29	7.53	8.89	8.91	9.17
456	0.51	3.58	4.61	5.84	8.48	9.07	8.37	0.89	2.55	4.21	7.39	8.43	8.44	8.69
480	0.57	3.8	4.76	5.89	8.18	8.06	7.95	1.18	2.81	4.24	7.19	8	8.02	8.25
504	0.64	3.99	4.8	5.92	7.79	8.21	7.57	1.42	3.08	4.26	7.01	7.62	7.64	7.86

Table 3. Corrosion rate data of quenched C26 Cu alloy in specific concentrations of HNO_3 solution with respect to observation time

Observation Time (h)	Quenched C26 HNO₃ Concentration (M)						
	0.5 M	1 M	1.5 M	2 M	2.5 M	3 M	3.5 M
24	0.29	0.16	0.89	3.6	14.41	20.91	21.27

48	0.21	0.15	0.9	3.86	10.85	19.9	21.24
72	0.36	0.44	0.81	5.43	11.68	19.84	21.74
96	0.48	0.5	0.78	5.7	11.8	19.29	20.42
120	0.5	0.4	1.07	6.95	11.69	18.38	20.47
144	0.52	0.34	1.26	7.53	11.52	17.78	19.41
168	0.55	0.37	1.57	7.31	11.97	16.38	17.84
192	0.53	0.45	1.72	7.51	11.98	15.81	16.65
216	0.48	0.44	1.84	7.9	12.02	15.6	16.26
240	0.45	0.43	2.1	7.72	11.39	14.58	15.38
264	0.43	0.49	2.31	7.57	11.22	13.96	14.65
288	0.41	0.57	2.8	7.71	10.72	13.57	13.43
312	0.39	0.68	3.15	7.72	10.22	12.52	12.4
336	0.37	0.74	3.65	7.55	9.98	11.63	11.51
360	0.38	0.82	3.62	7.63	9.82	10.85	10.74
384	0.38	0.88	3.89	7.47	9.69	10.18	10.07
408	0.57	0.93	4.04	7.23	9.12	9.58	9.48
432	0.7	1.07	3.97	7.15	8.61	9.04	8.95
456	0.77	1.14	3.91	6.95	8.16	8.57	8.48
480	0.85	1.2	3.9	6.71	7.75	8.14	8.06
504	0.96	1.29	4.97	6.6	7.38	7.75	7.68

Table 4. C26 corrosion rate and GLC protection performance data at specific GLC concentrations in 0.5 M HNO_3 solution with respect to observation time

GLC Concentration (%) / Observation Time (h)	Inhibition Efficiency (%)			
	20%	40%	60%	80%
24	99.70	106.60	103.60	102.85
48	98.33	102.3	101.71	103.86
72	91.20	98.30	97.30	100.00
96	87.25	96.71	94.00	93.82
120	81.91	95.09	91.48	88.76
144	75.54	92.35	90.15	82.52
168	72.29	88.48	87.72	78.70
192	70.16	86.57	85.83	74.65
216	68.65	83.84	84.56	71.66
240	64.66	75.47	82.11	67.90
264	63.42	73.36	81.20	61.91
288	61.95	70.69	79.88	59.67
312	60.41	67.20	79.46	58.63

3.2 Statistical analysis

Statistical data showing the mean and standard deviation (SD) for untreated, annealed and quenched C26 corrosion rate data with respect to exposure time and HNO_3 concentration is shown in Table 5 and 6 while Table 7 shows the mean and SD data for GLC protection performance data with respect to exposure time and GLC concentration. The mean corrosion rate data for untreated C26 varied proportionally with increase in HNO_3 concentration. At 0.5 M HNO_3, the SD value is 1.19. Decrease occurred at 1 M HNO_3 before gradual increase with respect to HNO_3 concentration till 3.5 M HNO_3. The higher the SD value, the greater the variation of untreated C26 corrosion rate from mean value signifying significant thermodynamic instability occurring on C26 surface in the presence of NO3- ions. The highest SD value for untreated C26 was obtained at 3.5 M HNO_3 (6.43) while the lowest value was obtained at 1 M HNO_3 (0.42) signifying stable electrochemical behaviour at this

concentration. The mean values for annealed C26 exhibited similar trend compared to untreated C26 Cu alloy though the values for annealed C26 where generally higher. However, the SD values obtained are generally lower. Observation of the corrosion rate data in Tables 2 and 3 shows heat treatment has limited influence on the corrosion reaction behaviour of C26 Cu alloy. However, SD values obtained are relatively lower, as a result annealed C26 alloy are thermodynamically more stable than untreated C26 in dilute HNO_3 solution but similar to quenched C26 alloy (Table 6).

The mean values in Table 7 show effective inhibition performance generally with respect to observation time. The SD values in Table 7 vary with GLC concentration but not linearly. The SD values at 20% and 80% GLC concentration are significantly higher than the values obtained at 40% and 60% GLC. The higher SD values obtained specific to GLC concentration is due to significant variation of GLC protection performance data from mean value signifying thermodynamic instability and relative unstable inhibition behaviour. At 40% and 60% GLC concentration lower SD value is due to stable inhibition behaviour of GLC with respect to observation time. As shown in Table 7, 62.5% of GLC protection performance data are greater than 85% inhibition efficiency at margin of error of $\pm15\%$.

Table 5. Mean and standard deviation data for untreated and annealed C26 Cu alloy with respect to HNO_3 concentration and observation time

C26	Untreated							Annealed						
HNO₃ Conc. (M)	0.5	1	1.5	2	2.5	3	3.5	0.5	1	1.5	2	2.5	3	3.5
SD	1.19	0.42	0.64	1.83	1.22	1.86	6.43	0.16	0.29	0.79	0.73	0.86	2.34	5.04
Mean	1.42	2.61	2.71	6.01	13.22	19.48	25.45	0.30	1.20	2.04	9.71	11.35	11.95	22.74

Table 6. Mean and standard deviation data for quenched C26 Cu alloy with respect to HNO_3 concentration and observation time

C26	Quenched						
HNO₃ Conc. (M)	0.5	1	1.5	2	2.5	3	3.5
SD	1.04	2.37	1.57	1.65	1.65	2.76	7.12
Mean	0.44	0.37	1.29	6.35	11.93	17.85	19.07

Table 7. Mean and standard deviation data for GLC protection performance output on C26 Cu alloy with respect to GLC concentration and observation time

GLC Conc. (%)	20	40	60	80
SD	12.63	9.25	7.26	13.30
Mean	80.97	92.57	91.85	86.47
Margin of Error	$\pm15\%$			
Data above 85% Inhibition	62.5%			

Analysis of variance (ANOVA) was used to evaluate the statistical importance of HNO_3 concentration and observation time on the corrosion rate data of untreated, annealed and quenched C26 alloy. The data are shown in Tables 8 and 9. Table 10 shows the data from analysis of variance for GLC protection performance data with respect to GLC concentration and observation time. The statistical relevance factor shows the percentage value of the statistical importance of the sources of variation on the corrosion rate output of untreated, annealed and quenched C26, and GLC protection performance output. The theoretical significance factor is the threshold value wherewith the mean square ratio is

measured against and must be greater than for the statistical relevance factor to be established. Tables 8 shows observation time overwhelmingly influenced the corrosion rate output of untreated and annealed C26 Cu alloy compared to HNO_3 which is also relatively high. However, the mean square ratio is greater than the theoretical significance factor thus they are statistically irrelevant despite their influence on the corrosion rate output of untreated and annealed C26 Cu alloy. Similar value trend was observed in Table 9 for the statistical data for quenched C26 Cu alloy. Table 10 shows that GLC concentration is the only statistical relevant factor influencing the protection performance output of GLC extract. This is further established from the mean square ration value which is greater than the theoretical significance factor. However, observation time as shown the table is statistical irrelevant for reasons earlier explained.

Table 8. ANOVA data for the corrosion rate output of untreated and annealed C26 Cu alloy with respect to HNO_3 concentration and observation time

| Source of Variation | Untreated C26 | | | Annealed C26 | | |
	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance Factor, F (%)	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance Factor, F (%)
HNO_3 Conc.	-6.70	2.42	97.11	-5.31	2.42	96.47
Observation Time	-6.13	2.1	133.36	-6.13	2.1	167.12

Table 9. ANOVA data for the corrosion rate output of quenched C26 Cu alloy with respect to HNO_3 concentration and observation time

Source of Variation	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance Factor, F (%)
GLC Conc.	-10.44	2.42	97.5
Observation Time	-6.18	2.1	86.54

Table 10. ANOVA data for the GLC protection performance output with respect to GLC concentration and observation time

Source of Variation	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance Factor, F (%)
GLC Conc.	22.95	2.42	90.52
Observation Time	-2.20	2.1	-26.02

4. Conclusion

Gravimetric analysis of the corrosion resistance of as-received and heat treated C26000 Cu alloy specific concentrations of HNO_3 solution showed that heat treatment no influence on the electrochemical characteristics of C26000 alloy. Data showed increase in HNO_3 concentration strongly influenced the corrosion resistance of as-received and heat treated C26000 alloy while the corrosion rate was observed to vary with observation time. Further analysis of as-received C26000 in the presence of garlic extracts at the lowest HNO_3 concentration showed the extract performed marginally at most concentrations with optimal protection performance at 60% extract concentration. Corrosion resistance of C26000 vary minimally from mean values with respect to observation time. However, in the presence of the extracts variation increased significantly. Statistical data showed the

extract concentration was the major determinant factor influencing the corrosion behaviour of C26000 whereas in specific HNO_3 concentration without the extracts corrosion resistance exhibited no statistical relationship with observation time and HNO_3 concentration.

Acknowledgement

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