



Comparative assessment and statistical data of admixed rosemary and castor oil on the corrosion inhibition of high carbon and P4 low carbon mold steels

Roland Tolulope Loto*, Cleophas Akintoye Loto, Muyiwa Fajobi, Gabriel Olanrewaju

Department of Mechanical Engineering, Covenant University, Ogun State, Nigeria

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ABSTRACT

Admixed rosemary and castor oil extracts (RSCO) were studied for their corrosion inhibition properties on high carbon steel (HCS) and P4 low carbon mold steel (P4S) in 1 M H₂SO₄ and HCl solution by coupon measurement. Results obtained showed that the admixed oil extract performed effectively on HCS and P4S with mean inhibition efficiency values above 90% for P4S and 80% for HCS in both acid solutions. The inhibition efficiency values of RSCO on P4S and HCS varied only with exposure time in H₂SO₄ with progressive increase in value compared to the effect of RSCO concentration which was negligible. The corresponding effect of RSCO in HCl solution showed no effect of exposure time and RSCO concentration on its performance on P4S while on HCS the effect of exposure time was observed. The standard deviation values obtained for RSCO inhibition efficiency at all concentrations on P4S and HCS in H₂SO₄, and HCS in HCl shows the degree of variation from mean values to be significant compared to the values obtained for P4S in HCl solution which were relatively small. The proportion of inhibition efficiency values above 70% inhibition performance are 39%, 44%, 100% and 39% at margin of error of 13%, 13.5%, 0% and 13% for P4S and HCS in H₂SO₄ and HCl solution. Analysis of variance shows exposure time is the dominant statistically relevant source of variation influencing RSCO inhibition performance in H₂SO₄ and HCl solutions. © 2021 Elsevier Ltd. All rights reserved.

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1. Introduction

Corrosion of carbon steels in industrial operating environments containing certain levels chloride/sulphates anions is a major concern [1]. The resistance of carbon steels to corrosion under these conditions is of huge economic impact and industrial significance due to its versatility, excellent mechanical properties, low cost and recyclability. Carbon steels are extensively used as structural parts and components in machineries, building construction, oil production and refinery plants, radiator tank in automobile, chemical processing plants, high-temperature boilers, energy generation, fertilizer production and mining etc. The corrosion of carbon steels can be alleviated with the use of chemical compounds known as corrosion inhibitors to stifle the oxidation-reduction reactions on the steel surface. These compounds form

a protective barrier over the steel surface often associated with chemical and/or physical adsorption. Some inhibitors modify the corrosive environment forming harmless precipitates or compounds. Inorganic chemical inhibitors containing phosphates, chromates, and other heavy metals are facing strong restriction by various environmental regulation bodies worldwide due to their toxicity [2,3]. Despite the relative advancement in corrosion inhibitor research, production and applications, inhibitor application is still limited due to their toxicity and high production cost [4–6]. Research is still on-going on the use of environmentally sustainable compounds for corrosion inhibition [7–10]. Use of plant extracts for corrosion inhibition has seen increasing use of recent due to their non-toxic nature [11–16]. Previous research on rosemary and castor oil has shown that they are promising with respect to further development and can be improved further [17–22]. This manuscript focusses on the optimal inhibition performance of the combined admixture of rosemary and castor oil on carbon steel in dilute H₂SO₄ and HCl solution.

* Corresponding author.

E-mail address: tolu.loto@gmail.com (R.T. Loto).

2. Experimental methods

Corrosion Rosemary oil and castor oil (abbreviated as RSCO) were combined together in ratio 1:1 prepared in volumetric concentrations of 1.5%, 2.5%, 3.5%, 4.5%, 5.5% and 6.5% respectively per 200 ml of 1 M H₂SO₄ and HCl solution respectively. High carbon steel (HCS) and P4 low carbon mold steel rods were cut and sectioned into 7 test specimens for each experimental study. HCS have an average dimension of 1.6 cm radius and 0.6 cm thickness

while P4S has an average dimension of 0.65 cm thickness and 1.4 cm diameter. The cylindrical surface ends of the steels grades were grinded with silicon carbide abrasive papers of 80, 120 and 220 grits. Weighed steel specimens were individually submerged in 200 ml of H₂SO₄ and HCl solution at specific concentrations RSCO compounds for 216 h. The prepared steel specimens were weighed at 24 h interval with Ohaus analytical weighing balance. The weight loss was calculated from the difference between the initial weight of the steel specimen (kept constant for 216 h) and

Table 1
Data on corrosion rate of P4S from 1 M H₂SO₄/RSCO (0% – 6.5%) solution.

RSCO Conc. (%) Exp. Time (h)	0% RSCO	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	0.0026	0.0008	0.0012	0.0010	0.0008	0.0014	0.0011
48	0.0014	0.0004	0.0007	0.0005	0.0004	0.0008	0.0006
72	0.0009	0.0003	0.0004	0.0003	0.0003	0.0005	0.0004
96	0.0010	0.0003	0.0004	0.0003	0.0002	0.0005	0.0004
120	0.0019	0.0002	0.0004	0.0004	0.0002	0.0004	0.0003
144	0.0023	0.0002	0.0003	0.0003	0.0002	0.0005	0.0004
168	0.0038	0.0002	0.0003	0.0003	0.0001	0.0004	0.0003
192	0.0050	0.0002	0.0002	0.0002	0.0001	0.0004	0.0003
216	0.0066	0.0002	0.0002	0.0002	0.0001	0.0003	0.0002

Table 2
Data on corrosion rate of HCS from 1 M H₂SO₄/RSCO (0% – 6.5%) solution.

RSCO Conc. (%) Exp. Time (h)	0% RSCO	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	0.0122	0.0085	0.0090	0.0079	0.0066	0.0062	0.0098
48	0.0105	0.0057	0.0058	0.0050	0.0044	0.0041	0.0059
72	0.0091	0.0044	0.0046	0.0039	0.0034	0.0030	0.0044
96	0.0080	0.0037	0.0037	0.0031	0.0028	0.0025	0.0036
120	0.0116	0.0032	0.0033	0.0028	0.0025	0.0022	0.0032
144	0.0115	0.0029	0.0029	0.0025	0.0022	0.0020	0.0028
168	0.0119	0.0026	0.0026	0.0023	0.0020	0.0018	0.0026
192	0.0119	0.0024	0.0024	0.0021	0.0018	0.0017	0.0024
216	0.0129	0.0022	0.0022	0.0020	0.0018	0.0016	0.0023

Table 3
Data on corrosion rate of P4S from 1 M HCl/RSCO (0% – 6.5%) solution.

RSCO Conc. (%) Exp. Time (h)	0% RSCO	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	0.00116	0.00004	0.00007	0.00005	0.00003	0.00004	0.00001
48	0.00177	0.00006	0.00008	0.00011	0.00011	0.00011	0.00012
72	0.00219	0.00006	0.00008	0.00008	0.00009	0.00010	0.00011
96	0.00273	0.00024	0.00026	0.00021	0.00023	0.00026	0.00018
120	0.00432	0.00027	0.00023	0.00021	0.00032	0.00025	0.00016
144	0.00627	0.00030	0.00026	0.00021	0.00037	0.00027	0.00020
168	0.00747	0.00031	0.00028	0.00019	0.00042	0.00027	0.00018
192	0.00686	0.00031	0.00031	0.00017	0.00042	0.00026	0.00016
216	0.00648	0.00030	0.00030	0.00020	0.00049	0.00029	0.00016

Table 4
Data on corrosion rate of HCS from 1 M HCl/RSCO (0% – 6.5%) solution.

RSCO Conc. (%) Exp. Time (h)	0% RSCO	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	0.0061	0.0022	0.0031	0.0021	0.0032	0.0042	0.0015
48	0.0051	0.0013	0.0024	0.0015	0.0026	0.0025	0.0014
72	0.0038	0.0011	0.0019	0.0013	0.0019	0.0019	0.0014
96	0.0031	0.0010	0.0016	0.0011	0.0015	0.0017	0.0012
120	0.0047	0.0011	0.0015	0.0010	0.0013	0.0016	0.0011
144	0.0057	0.0011	0.0013	0.0009	0.0012	0.0016	0.0010
168	0.0068	0.0011	0.0011	0.0009	0.0012	0.0015	0.0010
192	0.0075	0.0010	0.0011	0.0009	0.0011	0.0014	0.0009
216	0.0084	0.0007	0.0010	0.0008	0.0014	0.0013	0.0009

the final weight of the steel after measurement every 24 h. Tabulated results of HCS and P4S corrosion at specific RSCO concentrations in both acids are shown from Table 1–4. Corrosion rate was calculated from the equation below;

Table 5
Data on inhibition efficiency of RSCO on P4S in 1 M H₂SO₄ solution.

RSCO Conc. (%) Exp. Time (h)	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	69.12	53.19	62.99	69.61	46.57	57.35
48	69.60	50.12	60.33	69.12	43.23	53.21
72	65.25	57.92	67.61	67.14	44.21	59.81
96	73.64	61.45	70.18	76.28	50.91	62.11
120	86.98	77.71	79.75	89.91	78.60	83.78
144	90.49	86.05	86.33	93.33	79.20	83.16
168	94.35	92.84	93.08	96.33	89.05	91.52
192	95.87	95.70	95.66	97.47	92.41	94.98
216	97.11	97.30	97.03	98.18	94.73	96.76

Table 6
Data on inhibition efficiency of RSCO on HCS in 1 M H₂SO₄ solution.

RSCO Conc. (%) Exp. Time (h)	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	30.84	26.30	35.12	46.19	49.71	19.96
48	46.16	44.41	52.46	58.27	61.40	43.82
72	51.47	49.42	56.60	62.82	66.82	50.98
96	54.09	53.35	60.67	64.65	68.76	54.82
120	72.11	71.85	75.67	78.53	80.72	72.71
144	75.09	74.98	78.64	81.00	82.65	75.36
168	78.05	78.21	81.01	83.23	84.60	78.47
192	80.09	79.75	82.39	84.55	86.14	80.15
216	82.93	82.56	84.55	86.17	87.39	82.19

Table 7
Data on inhibition efficiency of RSCO on P4S in 1 M HCl solution.

RSCO Conc. (%) Exp. Time (h)	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	96.65	93.85	95.53	97.77	96.65	98.88
48	96.69	95.40	93.75	93.75	93.57	93.20
72	97.24	96.35	96.15	95.76	95.46	94.77
96	91.14	90.49	92.45	91.68	90.49	93.58
120	93.78	94.71	95.25	92.48	94.20	96.36
144	95.27	95.89	96.60	94.03	95.72	96.88
168	95.87	96.28	97.52	94.44	96.45	97.62
192	95.52	95.53	97.59	93.89	96.24	97.64
216	95.35	95.44	96.89	92.42	95.59	97.52

Table 8
Data on inhibition efficiency of RSCO on HCS in 1 M HCl solution.

RSCO Conc. (%) Exp. Time (h)	1.5% RSCO	2.5% RSCO	3.5% RSCO	4.5% RSCO	5.5% RSCO	6.5% RSCO
24	63.75	49.49	65.81	48.07	31.23	74.68
48	75.02	52.67	70.07	48.57	51.35	72.39
72	70.15	49.04	66.44	51.10	50.41	64.17
96	68.37	47.60	64.86	52.59	46.01	60.83
120	76.78	68.82	78.62	71.60	66.14	76.55
144	80.58	77.38	83.83	78.65	72.41	81.78
168	83.43	83.23	86.56	82.87	78.60	85.86
192	86.25	85.90	88.57	84.83	81.76	88.00
216	91.77	88.23	90.10	83.00	84.63	89.27

$$R = \left[\frac{87.6W}{DAT} \right] \tag{1}$$

W is the weight loss in grams, D is the density in g/cm², A is the area in

cm², and T is the time of exposure in hours. Tables 5–8 shows the calculated result of inhibition efficiency (IE) as follows;

$$IE = \left[\frac{W_1 - W_2}{W_1} \right] * 100 \tag{2}$$

W₁ and W₂ are the weight-loss of the control (0% RSCO concentration) and inhibited steels in the acid solution with respect to exposure time.

3. Results and discussion

3.1. Coupon measurement

Observation of Tables 1 and 2 shows the corrosion rate of control P4S and HCS specimens at 0% RSCO concentration significantly vary from the inhibited specimens at specific RSCO concentrations due to the electrochemical action of SO₄²⁻ anions in H₂SO₄ solution. It is also observed that the value of corrosion rate for the control HCS specimens is significantly higher than the values obtained for P4S due to the higher corrosion resistance of P4S in H₂SO₄. Comparing the observations with the results in Tables 3 and 4 for corrosion in HCl solution. It can be seen that the rate of deterioration for P4S is much higher compared to values obtained in Table 1 while the rate of deterioration of HCS is relatively lower compared to Table 2 [23,24]. These differences, despite similar RSCO concentration are due to the Cl⁻ anion content in HCl responsible for corrosion of both steels and the competitive adsorption of the anions and inhibitor cations on the steel surface. The inhibition efficiency values of RSCO on P4S and HCS in both acid media is shown from Tables 5–8. Tables 5, 6 and 8 show similar trends in the rate of change of inhibition efficiency with exposure time. Observation shows the inhibition efficiencies on these tables progressively increased to values that signifies effective inhibitor protection at 216 h. This shows the inhibitor protection of RSCO on P4S and HCS in 1 M H₂SO₄, and HCS in 1 M HCl is time dependent [25]. The values presented in Table 7 shows the inhibition efficiency values of RSCO was generally stable at very effective values (above 90%) throughout the exposure hours signifying non-dependence on exposure to reach peak performance. However, RSCO showed total non-dependence on inhibition concentration. Comparative plot of the inhibition efficiency of RSCO on P4S and HCS at highest and lowest RSCO concentration in 1 M H₂SO₄ solution is shown in Fig. 1(a) while the corresponding plot for HCl is shown in Fig. 2(a and b). Fig. 1(a and b) shows the inhibition performance of RSCO on P4S and HCS varies significantly with exposure time and are closely aligned. However, the degree of alignment is much closer in Fig. 1(b) with respect to exposure time. The observation in Fig. 1(a and b) shows the protonated molecular species of RSCO in H₂SO₄ solution react with time gradually inhibiting the electrochemical action of SO₄²⁻ anions in the acid solution. Similar observation occurred in Fig. 2(b). However, Fig. 2(a) shows exposure time has limited influence on the performance of RSCO on P4S in HCl solution. Effective inhibition occurred from the onset of exposure with the inhibitor compounds strongly reacting with the steel to prevent the redox electrochemical processes responsible for corrosion.

3.2. Statistical evaluation

The mean, standard deviation and margin of error for RSCO inhibition efficiency data shown from Tables 5–8 are presented

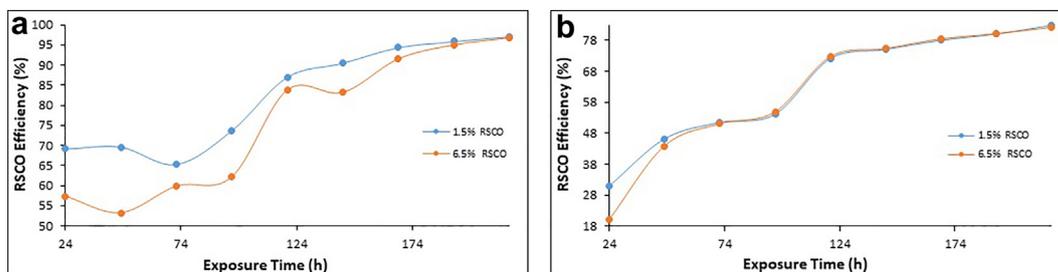


Fig. 1. Comparative plot of the inhibition efficiency of RSCO on (a) P4S and (b) HCS at highest and lowest RSCO concentration versus exposure time in 1 M H₂SO₄ solution.

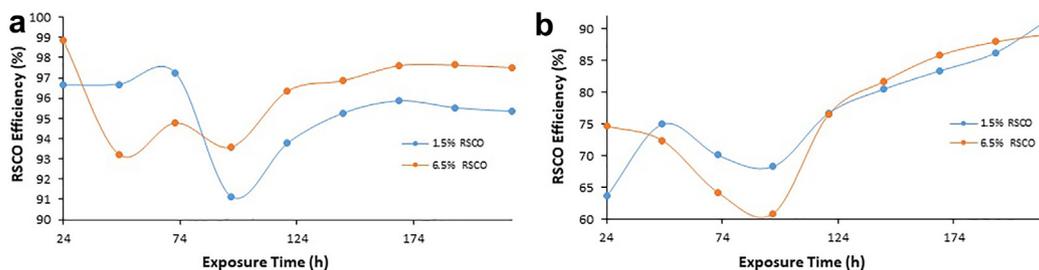


Fig. 2. Comparative plot of the inhibition efficiency of RSCO on (a) P4S and (b) HCS at highest and lowest RSCO concentration versus exposure time in 1 M HCl solution.

Table 9

Mean, standard deviation and margin of error for RSCO inhibition efficiency P4S and HCS in H₂SO₄ solution.

P4S						
ROSC Conc.	1.5%	2.5%	3.5%	4.5%	5.5%	6.5%
Standard Deviation	12.93	19.18	14.44	13.36	22.13	17.56
Mean	82.49	74.70	79.22	84.15	68.77	75.85
Proportion of data above 70% Inhibition Efficiency	39%	Margin of Error	13%			
HCS						
Standard Deviation	18.28	19.63	17.05	14.14	13.16	21.13
Mean	63.43	62.31	67.46	71.71	74.24	62.05
Proportion of data above 70% Inhibition Efficiency	44%	Margin of Error	13.25%			

Table 10

Mean, standard deviation and margin of error for RSCO inhibition efficiency P4S and HCS in HCl solution.

P4S						
ROSC Conc.	1.5%	2.5%	3.5%	4.5%	5.5%	6.5%
Standard Deviation	1.85	1.82	1.73	1.86	1.95	1.98
Mean	95.28	94.88	95.75	94.02	94.93	96.27
Proportion of data above 70% Inhibition Efficiency	100%	Margin of Error	0%			
HCS						
Standard Deviation	9.07	17.30	10.48	16.36	18.55	10.16
Mean	77.34	66.93	77.21	66.81	62.50	77.06
Proportion of data above 70% Inhibition Efficiency	39%	Margin of Error	13%			

in Tables 9 and 10. The standard deviation data in Table 9 for P4S and HCS are comparable with respect to RSCO concentration. Observation of the table shows the standard deviation data varies from 12.93 and 18.28 for P4S and HCS at 1.5% RSCO concentration to 17.56 and 21.13 at 6.5% RSCO concentration. These values show the mean values with respect to RSCO concentration significantly varies with the inhibition efficiency data obtained per exposure time. The results confirm the earlier observation that the effective inhibition performance of RSCO compound is proportional with time. The margin of error shows 39% of RSCO inhibition on P4S and 44% of RSCO inhibition on HCS are above 70% inhibition in

H₂SO₄ solution at margin of error of 13% and 13.25%. The standard deviation value in Table 10 for RSCO inhibition on P4S and HCS are significantly lower than the values obtained in Table 9. The standard deviation value for RSCO inhibition on P4S in HCl solution ranges from 1.85 at 1.5% RSCO concentration to 1.98 at 6.5% RSCO concentration due to the minimal variation of inhibition efficiency values obtained with respect to exposure time. The mean values for inhibition efficiency are generally above 90% while the margin of error is 0% for 100% of the inhibition efficiency data above 70% inhibition efficiency. The corresponding data for HCS in HCl solution shows 39% of RSCO inhibition efficiency is above 70% inhibi-

Table 11
ANOVA data for statistical influence of RSCO concentration and exposure time on RSCO inhibition performance in H₂SO₄ solution.

P4S						
Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	Mean Square Ratio (F)	Threshold Significance Factor	Statistical Relevance Factor (%)
RSCO Conc.	1430.219	5	286.04	16.63	2.45	9.42
Exposure Time	13071.35	8	1633.92	95.01	2.12	86.06
Residual	687.9088	40	17.20			
Total	15189.47	53				
HCS						
RSCO Conc.	1205.966	5	241.19	23.37	2.45	7.61
Exposure Time	14227.5	8	1778.44	172.30	2.12	89.78
Residual	412.8814	40	10.32			
Total	15846.35	53				

Table 12
ANOVA data for statistical influence of RSCO concentration and exposure time on RSCO inhibition performance in H₂SO₄ solution.

P4S						
Source of Variation	Sum of Squares	Degree of Freedom	Mean Square	Mean Square Ratio (F)	Theoretical Significance Factor	Statistical Relevance (%)
RSCO Conc.	27.10	5	5.42	3.96	2.45	13.93
Exposure Time	112.72	8	14.09	10.30	2.12	57.94
Residual	54.74	40	1.37			
Total	194.56	53				
HCS						
RSCO Conc.	1991.04	5	398.21	13.74	2.45	17.11
Exposure Time	8487.84	8	1060.98	36.61	2.12	72.93
Residual	1159.16	40	28.98			
Total	11638.04	53				

tion with margin of error of 13%. The standard deviation values are slightly higher than the corresponding values obtained for P4S in HCl but significantly lower than the corresponding values in H₂SO₄ solution.

Analysis of variance (ANOVA) was applied to study the statistical influence of RSCO concentration and exposure time (sources of variation) on the inhibition performance of RSCO on P4S and HCS in H₂SO₄ and HCl solution [26]. Tables 11 and 12 shows the ANOVA data for RSCO inhibition performance on both steels in the acid solutions. The statistical relevance factor shows the percentage statistical significance of the sources of variation on RSCO inhibition performance. The mean square ratio is the statistical significance value which must be greater than the threshold significance factor to be a statistically relevant factor on the inhibition performance of RSCO in both acids. The statistical relevance factor in Table 11 shows exposure time is the dominant source of variation (influencing factor) responsible for RSCO inhibition performance at 86.06% and 89.78% for P4S and HCS in H₂SO₄ solution. The corresponding value for RSCO concentration is 9.42% and 7.61%. Despite this relatively insignificant value. The mean square ratio in Table 11 is significantly higher than the threshold significance factor making them statistically relevant with minimal influence on RSCO inhibition performance. The ANOVA data in Table 12 for P4S in HCl was observed to be quite similar to the ANOVA data for P4S and HCS in H₂SO₄ (earlier discussed). However, the corresponding data obtained for HCS in HCl shows the statistical relevance factor for RSCO concentration is generally higher than previous values earlier discussed signifying that RSCO concentration is more statistically relevant on HCS in HCl compared to P4S in HCl and H₂SO₄ solution, and HCS in H₂SO₄ solution.

4. Conclusion

The combined admixture of rosemary and castor oil extracts effectively inhibited the corrosion of P4 low carbon mold steel and high carbon steel in H₂SO₄ and HCl solution with optimal inhi-

bition performance generally above 90% at the end of the exposure hours. The effect of inhibitor concentration was negligible as the inhibition efficiency did not vary with concentration compared to exposure time which significantly influenced the performance of the oil extract due to the time dependent action of the inhibitor. The standard deviation values show the degree of variation from mean values varies significantly due to variation with time. Statistical data through analysis of variance shows exposure time is the only statistical relevant variable influencing the performance of the extract with statistical value generally above 80%.

CRedit authorship contribution statement

Roland Tolulope Loto: Supervision, Conceptualization, Writing – original draft. **Cleophas Akintoye Loto:** Supervision, Conceptualization, Writing – original draft. **Muyiwa Fajobi:** Visualization, Investigation, Validation, Methodology, Data curation. **Gabriel Olanrewaju:** Visualization, Investigation, Validation, Methodology, Data curation.

Declaration of Competing Interest

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

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