



ADSORPTIVE EFFECTS AND SYNERGISTIC STUDIES ON AQUEOUS EXTRACT OF *DATURA STRAMONIUM* LEAVES AS GREEN CORROSION INHIBITOR FOR MILD STEEL IN WELL WATER

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Abstract:

The synergistic effect of *Datura Stramonium* leaves extract (DSLE) and Zn^{2+} on the corrosion inhibition of mild steel in aqueous medium (100ml well water) was investigated. The corrosion inhibition studies were carried out using weight loss method. The inhibition efficiency was found to increase with increase in extract concentration. The inhibition efficiency of the formulation extract and Zn^{2+} ion combined was found to be 90% at DSLE extract (8ml concentrations) and Zn^{2+} (5ppm) respectively in well water. The synergism factor at the same formulations revealed 1.25 which indicates a synergistic effect exists between the inhibitors. The characteristics behaviour of the adsorption inhibitors were evaluated using the Freundlich isotherm adsorption approach at all concentrations. The synergy of *Datura Stramonium* leaves extract was investigated using the thermodynamic parameters and the phenomenon of physiosorption is proposed from the values of Gibb's free energy of change.

Keywords: mild steel, well water, weight loss, corrosion inhibitor, *Datura stramonium* leaves, physiosorption

1. INTRODUCTION

Corrosion is a phenomenon resulting in materials deterioration through chemical or electrochemical interaction with the environment. This process may compromise the mechanical and physical properties of equipment with serious economic and environmental impact in all infrastructure sectors, such as roads, oil and gas pipelines, construction, water and sewage systems, causing severe damage and threats to public safety and health (Lhaira *et al.*, 2017). Corrosion is an expensive problem in the industrial world according to American Galvanizer's Association. Steel corrosion is a costly problem; in fact the annual direct cost of metallic corrosion worldwide is \$2.5

Trillion USD (AGA, 2024). North America is a large contributor to these annual costs, as the annual cost of corrosion in the US is \$659 Billion, the equivalent of 3.4% of the Gross Domestic Product (GDP). Though steel corrosion, whether in atmospheric, soil, water, or other exposures is a natural phenomenon, it estimates show that 25-30% could be stymied if proper corrosion protection methods were employed (AGA, 2024).

Several methods have been employed to control corrosion, some which includes the use of inorganic compounds such as chromate, phosphates, molybdates among others (Miralrio and Vazquez, 2020). In



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addition a variety of organic compounds containing hetero-atoms such as nitrogen, phosphorus, sulphur and oxygen are being investigated as corrosion inhibitors (Izionworu *et al.*, 2020; Kavitha *et al.*, 2014). Due to the toxic effect of inorganic corrosion inhibitors the search for green corrosion inhibitors has become utterly necessary as they are biodegradable, renewable and free from toxic compounds as well as heavy metals. A number of plant sources such as *Polyalthia Longifolia*, *Eclipta alba*, Red Onion, *Jatropha curcas*, *Crataegus oxyacantha*, *Prunus avium*, *Murraya koenigii* and *Aloe vera* among others have been reported as effective corrosion inhibitors (Rahal *et al.*, 2018; Singh *et al.*, 2014; Olusegun *et al.*, 2013).

Despite the extensive spectrum of naturally and organic occurring compounds as metallic corrosion inhibitors, their effective performance is predicated on the phytochemical constituents that are present in the extracts (Umoren *et al.*, 2012). In recent times, studies have shown that the vast variety of possible corrosion systems coupled with the specificity of action of most acid inhibitors often necessitates the use of combined extracts to provide synergistic effects for effective metallic corrosion inhibition. The results of such studies are the basis for the investigation of synergism among corrosion inhibiting compounds (Nasikin *et al.*, 2019; Huaihang Zhang *et al.*, 2017).

According to Eduok *et al.*, (2010), synergism is an effective method to optimize the performance of organic inhibitors knowing well that their potency is as result of the presence of the polar functional groups that assist in the adsorption process. In this regard, the amount of usage is reduced due to cost

and, more importantly, to diversify the application of inhibitors in acidic media (Jingmao Zhao *et al.*, 2015; Li *et al.*, 2009). Some authors have reported on synergistic effect inhibition on organic inhibitors and metallic ion mixture (Prathipa and Raja, 2017; Huaihang Zhang *et al.*, 2017; Kavitha *et al.*, 2014; Li *et al.*, 2008; Cheng *et al.*, 2007; Alagta *et al.*, 2007). However, literature reports on the synergistic effects of inhibition between plant extracts and metallic ions are still very few. Raja *et al.*, (2014) conducted a study on *Acalypha indica* extract and $ZnSO_4$ using gravimetric and electrochemical methods. Kavitha *et al.*, (2014) also evaluated the combined effects of *Carica papaya* leaves extract in the presence of Zn^{2+} ion on mild steel in aqueous medium and found that 2ml of the leaf extract and 50ppm of Zn^{2+} produced inhibition efficiencies of 32% and 59% respectively working alone, but in combination produced an inhibition efficiency of 91%. This suggests an active synergism exists between them. Moreover, the synergistic factors of the combined extracts were greater than 1, which indicates further synergism between the combined inhibitors. The inhibition action of L-Methionine on mild steel and the synergistic effects of Zn^{2+} ions have also been studied (Prathipa and Raja, 2017).

This research focuses on the corrosion inhibition effect of *Datura Stramonium* leave extract – Zn^{2+} on mild steel in aqueous medium using weight loss method. The comparison were made on the separate effect of both the DSLE and the Zn^{2+} ion on the corrosion inhibition of mild steel in the presence of well water and their combined effect as corrosion inhibitors.



2. METHODOLOGY

2.1 Preparation of Specimens

The mild steel sheets utilized for the study has the following composition (wt. %): 78.3% Fe, 0.128% C, 0.010% P, 0.030% S, 0.820% Cr, 0.292% Mn, 11.50% Ni, 1.420% Ar, 0.096% Pb, 0.074% Cu and 0.066% N. The chemical composition of the mild steel was determined via spectrometric analysis. The mild steel specimen was mechanically cut into coupons of dimension 1cm by 1cm, with a thickness of 0.35cm. They were abraded with different grades (400, 600, 800 and 1000) silicon carbide paper, degreased in absolute ethanol, dried in acetone and stored in a moisture-free desiccator prior to use. The aqueous medium employed was well water, and the well water was obtained at Annex Hostel, the Federal Polytechnic Ado-Ekiti, Nigeria. 100ml of the well water was used in each experiment.

Table 1: Parameters of well water

Parameter	Value
Ph	8.6
Conductivity	2620 μ mhos/cm
TDS	1835mg/L
Chloride	450
Sulphate	110
Total hardness	96

2.2. *Datura Stramonium* Leaf Extract, DSLE Preparation

The *Datura Stramonium* leaves were collected nearby Ado-Ekiti golf club along the Federal Polytechnic Ado Ekiti Road, Nigeria. It was ensured that the leaves were used on the day it was collected to ensure that the freshness of the leaves were still intact. An aqueous

extract was prepared with 20g of fresh leaf sample, washed with distilled water, grinded, and then made up to a solution of 100ml using double distilled water. The resulting solution was then filtered and kept in an airtight container.

2.3 Weight Loss Method

2.3.1 Experiment 1

Relevant data on the well water utilized in this study are given in Table 1. The finely polished and dry mild steel specimens of 1cm by 1cm dimensions were weighed and then immersed in 100ml of well water in the absence of the extract as well as in the presence of 2ml, 4ml, 6ml and 8ml concentration of the extract for a period of 24hrs each. Then the specimens were removed from the solutions, washed with acetone, dried and then re-weighed. The weight loss of each specimen after 24hrs in the different solutions was then computed by subtracting the weight after immersion from the weight before immersion.

2.3.2. Experiment 2

The finely polished and dried mild steel specimens were weighed and then immersed in solutions containing 100ml of well water and 10ml of Zn^{2+} solution of 5ppm concentration in the absence of the extract as well as in the presence of 2ml, 4ml, 6ml, and 8ml concentration of the extract. The steel specimens were retrieved from the different solutions after 24hrs immersion, washed with acetone, dried and re-weighed. The weight loss of the steel specimens over 24hrs of immersion in the different solutions were computed by taking the difference between the initial weight of the specimen before immersion and the weight after 24hrs in the solution. From the weight loss values, the following



parameters were computed using the relationship:

$$\text{Corrosion Rates (C. R)} = \frac{W}{DAT} \quad 1$$

Where, W is weight loss in grams, D is density of mild steel (7.86 g/cm²), A is area in cm², T is exposure time in hours.

Inhibition efficiency in percentage was computed using the following relationship;

$$\% \text{ I. E} = \frac{W_u - W_i}{W_u} \% \quad 2$$

Where, W_u is weight loss the in absence of extract, W_i is weight loss in the presence of extract.

The surface coverage (θ) was expressed by the mathematical equation;

$$\theta = \frac{W_u - W_i}{W_u} \quad 3$$

The synergism factor, S_θ was calculated using the relationship;

$$S_{\theta} = (\theta A + \theta B) - \theta AB \quad 4$$

Where θA and θB are the surface coverage of compounds A and B respectively acting alone, and θAB is the observed combined surface coverage of the solution containing both A and B.

3. RESULTS AND DISCUSSION

3.1 Weight loss, corrosion rates and inhibition efficiency

i. Effect of concentration of extract on weight loss

It was observed that there was a reduction in the mild steel weight loss due to corrosion with the increase in concentration of the DSLE. Without inhibition extract, a weight loss of 0.4g was observed by the mild steel in the aqueous medium, and while with the introduction of 2ml of the extract the weight loss was 0.25g. Moreover, further increase in extract concentrations of DSLE to 4ml, 6ml, and 8ml produced 0.2g, 0.15g, and 0.1g in weight loss respectively.

However, with the addition of 10ml concentration of Zn²⁺ (5ppm), a weight loss of 0.2g was observed on the mild steel in the aqueous medium (without the DSLE). When 10ml concentration of

Zn²⁺ (5ppm) was combined with the extract, the weight loss reduced to 0.08g, 0.06g, 0.04g and 0.02g in 2ml, 4ml, 6ml and 8ml concentrations of the extract respectively. This result shows a synergistic effect exists between the inhibitors (Prathipa and Sahaya, 2017).

ii. Effect of concentration of extract on inhibition efficiency

An increase in the concentration of the extract results in an increase in the inhibition efficiency as shown in Table 2. This is due to the fact that as the concentration of the plant extract increases, the fraction of the surface covered by the adsorbed molecule also increases which results in an increase in the inhibition efficiency. As shown in Table 3, an inhibition efficiency of 90% was achieved when 8ml of the extract was combined with 10ml concentration of Zn²⁺ (5ppm). This result emphasizes the synergistic effect that exists between the two inhibitors when combined.



5ppm of Zn^{2+} working alone achieves just 50% inhibition efficiency, while 2ml, 4ml, 6ml and 8ml of the extract acting alone achieves 37.5%, 50%, 62.5% and 75% inhibition efficiencies respectively; however, on combining with 5ppm of Zn^{2+} , the inhibition efficiencies rises to 60%, 70%, 80% and 90% respectively as shown in Tables 2 and 3. It therefore shows that a synergistic effect exists between the inhibitors. This is in tandem with the result obtained by Kavitha *et al.*, (2014).

iii. Effects of concentration on corrosion rate

As shown in the Table 3, it was observed that the corrosion rate of the mild steel specimen decreases with increase in various concentrations of DSLE with 5ppm of Zn^{2+} , indicating that the aqueous extract inhibited the corrosion of mild steel in the aqueous medium (Kavitha *et al.*, 2014)

iv. Effect of concentration on the synergism factor

S/N.	Conc. of DSLE(ml)	α A DSLE	% I.E	wt. loss(g)	C.R Mmpy
1	0	-	-	0.4	0.03170
2	2	0.375	37.5	0.25	0.02035
3	4	0.500	50	0.20	0.01734
4	6	0.625	62.5	0.15	0.01430
5	8	0.750	75	0.10	0.00954

As shown in the Tables 3 and 4, the synergism factor increases with increase in the concentrations of the DSLE and a synergism factor of 1.25 was ultimately achieved on the combination of 8ml of the DSLE with Zn^{2+} (5ppm). When the value of $S_0 < 1$, this indicates an antagonistic effect between the two inhibitors. When $S_0 = 1$, this indicates that the inhibitors act separately and as such has no effect on each other and adsorbed on the metal surface. A value of $S_0 > 1$ indicates that a synergistic effect exists between the inhibitors. Since the synergistic factor increased with increase in concentration of the extract until ultimately a synergistic factor of 1.25 was achieved at 8ml of the extract which is greater than unity, and it shows that synergy exists with the addition of Zn^{2+} (5ppm) to the extract inhibitors (Prathipa and Sahaya, 2017).
 Table 2: Corrosion Rate and Inhibition Efficiency of Mild Steel immersed in 0ppm Zn^{2+} in the presence of DSLE in Well Water using weight loss method



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Table 3: Corrosion Rate and Inhibition Efficiency of Mild Steel immersed in 5ppm Zn^{2+} in the presence of DSLE in Well Water using weight loss method

S/N	Conc. of DSLE(ml)	θ_{AB} (DSLE+ Zn^{2+})	% I.E	wt. loss (g)	θ_B Zn^{2+}	C.R mmpy	S_o
1	0	-	-	0.2	0.5	0.01637	-
2	2	0.6	60	0.08	0.5	0.00912	0.7813
3	4	0.7	70	0.06	0.5	0.00520	0.8333
4	6	0.8	80	0.04	0.5	0.00359	0.9375
5	8	0.9	90	0.02	0.5	0.00169	1.2500

Table 4: Surface Coverage, Synergistic Factor and Adsorption data of Mild Steel immersed in DSLE solution in the absence and presence of Zn^{2+} (5ppm) using weight loss method

S/N	Conc. DSLE (ml)	DSLE Extract θ_A	Zn^{2+} (5ppm) θ_B	DSLE + Zn^{2+} (5ppm) θ_{AB}	S_o	C/ S_o	log C	log S_o
1	0	-	-	-	-	-	-	-
2	2	0.375	0.50	0.60	0.7813	0.01167	-	-
3	4	0.500	0.50	0.70	0.8333	0.00624	-	-
4	6	0.625	0.50	0.80	0.9375	0.00383	-	-
5	8	0.750	0.50	0.90	1.2500	0.00135	-	0.0969

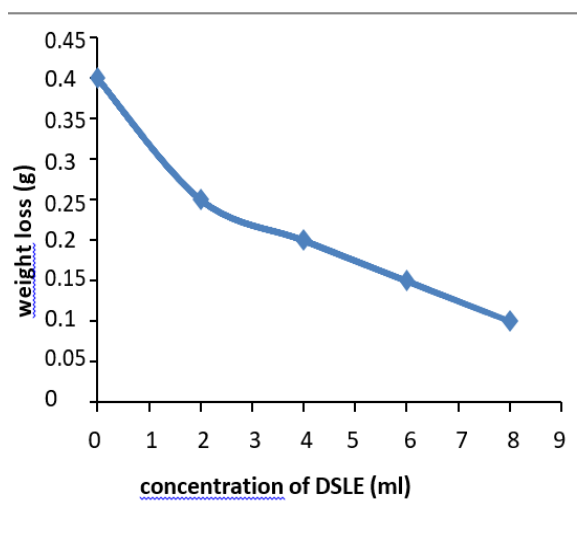


Fig. 1: weight loss against concentration of DSLE

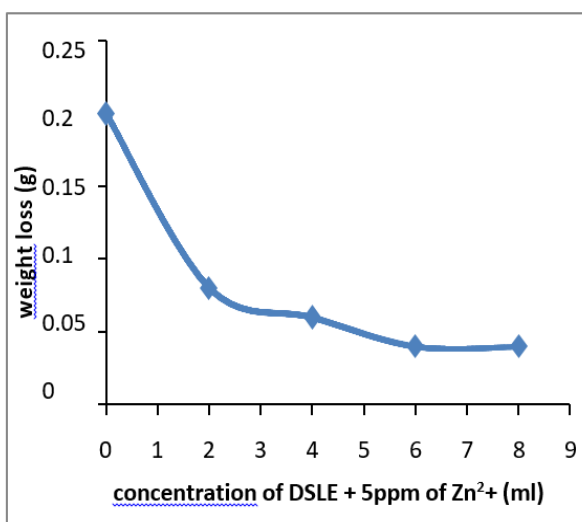


Fig. 2: weight loss against concentration of DSLE + 5ppm of Zn^{2+}

3.2: Adsorption considerations

Adsorption studies, in corrosion science provide further information required to

understand the interaction between the organic inhibitor molecules and the surface of metallic substrate. The



adsorption of the extract components on the metal surface leading to greater surface coverage is the first step towards inhibition mechanism. The various values of the degree of surface coverage, θ , obtained by weight loss method from both the extract alone and the combined inhibitors were used to evaluate the best-fit isotherm. These include Langmuir, Temkin, Freundlich and Flora-Huggins.

However, the correlation coefficient, R^2 , obtained from each isotherm model was used to determine the most suitable model. From the correlation (R^2) obtained, Freundlich isotherm was found to be the best that describes the synergism of DSLE and Zn^{2+} (5ppm) in well water. Hence, the model is suitable to evaluate the adsorption equilibrium constant, K_{ads} .

Table 5: Adsorption parameters for various concentrations of DSLE and Zn^{2+} (0ppm)

S/ N	Conc. (ml)	CR (mmpy)	%IE	θ	C/ θ	log C	log θ
1	Blank	0.03170	-	-	-	-	-
2	2	0.02035	37.5	0.375	0.05427	-1.6914	-0.4260
3	4	0.01734	50.0	0.500	0.03468	-1.7587	-0.3010
4	6	0.01430	62.5	0.625	0.02288	-1.8447	-0.2041
5	8	0.00954	75.0	0.750	0.01272	-2.0205	-0.1249

Table 6: Adsorption and Synergism ($S\theta$) parameters for various conc. of combined DSLE and Zn^{2+} (5ppm)

S/ N	Conc. (ml)	CR (mmpy)	%IE	θ	$S\theta$	C/ θ	log C	log θ
1	Blank	0.01637	-	-	-	-	-	-
2	2	0.00912	60.0	0.60	0.7813	0.01167	-2.0400	-0.1072
3	4	0.00520	70.0	0.70	0.8333	0.00624	-2.2840	-0.0792
4	6	0.00359	80.0	0.80	0.9375	0.00383	-2.4449	-0.0280
5	8	0.00169	90.0	0.90	1.2500	0.00135	-2.7721	0.0969



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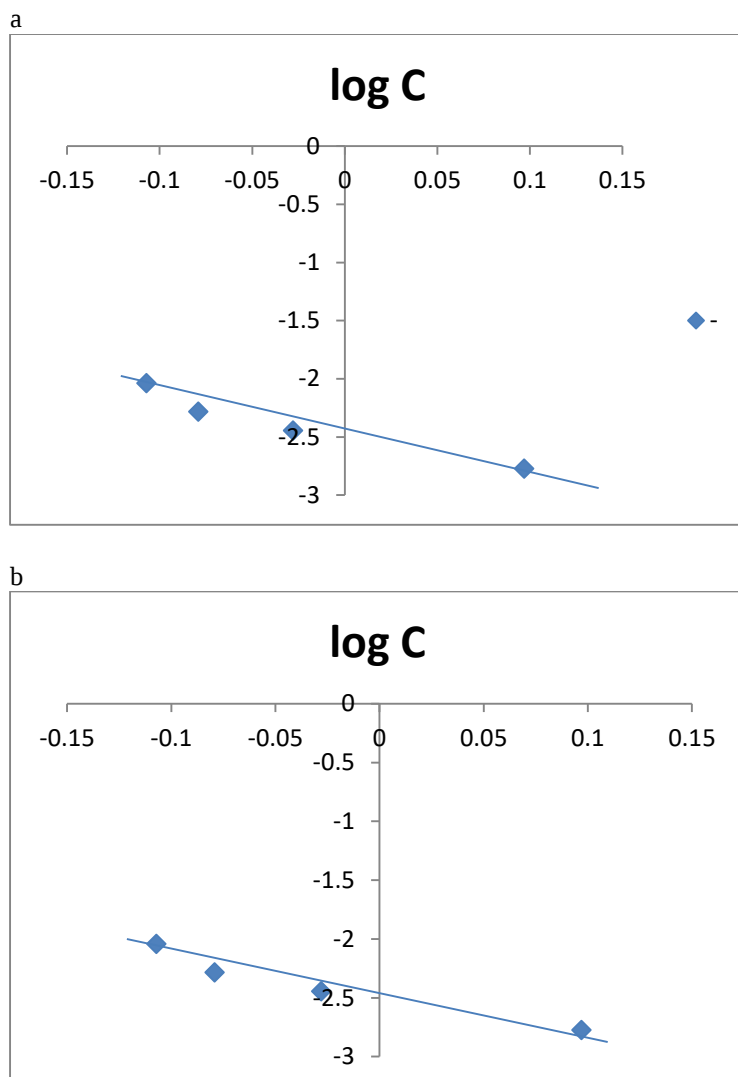


Fig. 3: Freundlich adsorption isotherm plots for mild steel in well water with (a) DSLE alone and (b) DSLE + Zn^{2+} (5ppm) at 30°C

The Freundlich adsorption isotherm equation is as follows (Kliskic *et al.*, 2000):

$$\theta = K_{ads} C^n \quad 5$$

or

$$\log \theta = \log K_{ads} + n \log C \quad 6$$

C is the concentration of the inhibitor (ml), θ is the surface coverage, n is the adsorption intensity, and K_{ads} is the equilibrium constant of the adsorption process obtained from the isotherm and is related to the free energy of adsorption by the equation (Akinbulumo *et al.*, 2020; Ebenso *et al.*, 2008).

$$\Delta G_{ads} = -RT \ln(55.5K_{ads}) \quad 7$$

Where the number 55.5 is the molar concentration of water in solution, R is the molar gas constant (kJ/molK) and T is the absolute temperature (K).



The logarithm of surface coverage plotted against logarithm of concentration are shown in Fig. 3a and 3b for mild steel in well water with DSLE alone and in combination with Zn^{2+} (5ppm) at 30°C. A straight line plots were obtained indicating that the experimental results fit the Freundlich isotherm.

Table 7: Adsorption parameters

Extract formulation	K_{ads} (mol ⁻¹)	ΔG_{ads} (KJ/mol)
DSLE alone	0.011	+1.24
DSLE + Zn^{2+} (5ppm)	0.2107	- 6.20

From Table 7, it is observed that the value of ΔG_{ads} in DSLE alone is positive while the value of ΔG_{ads} in DSLE combined with Zn^{2+} (5ppm) is negative. Also, the value K_{ads} obtained from the linear results of the plot (log θ vs log C) shows the strength between adsorbed species and the metal surface. A greater value of K_{ads} indicates that the adsorption is efficient and effective (Nasikin et al., 2019). Moreover, from the above, the value of K_{ads} in DSLE combined with Zn^{2+} (5ppm) is greater which is in tandem with the previous authors (Akinbulumo et al., 2020; Nasikin et al., 2019). It is therefore noteworthy to mention on this premise that, in well water, the DSLE alone will not be efficient and effective in preventing corrosion; however with the addition of Zn^{2+} (5ppm), a synergistic effect is created which enhances its adsorption inhibitory efficiency (Nasikin et al., 2019). The negative value of ΔG_{ads} , indicate the spontaneous adsorption of the extract/inhibitors on the mild steel surface and stability of the

adsorption layer on the metal. As shown in Table 7, the ΔG_{ads} for the combined inhibitors is negative and less than 20KJ/mol. Therefore, the adsorption mechanism occurred via physical adsorption.

4. CONCLUSION

Datura Stratonium leaves extract, DSLE, alone as an inhibitor will not be efficient and effective to prevent corrosion of mild steel in well water. The adsorption inhibition efficiency increased significantly on the addition of Zn^{2+} ion due to synergistic effect. The adsorption characteristics were approximated by Freundlich adsorption isotherm models. The phenomenon of physisorption is proposed from the values of Gibb's free energy change of adsorption which is negative and less than 20KJ/mol.

Further works

Further works on this study are hereby recommended:

- A detailed FTIR spectra analysis is required to identify the functional groups in the extract.
- The inhibition mechanism should be studied across different ranges of pH conditions.
- The determination of the effect of temperature and immersion time on the inhibition efficiency and corrosion mechanism.
- Effort should be made to develop computational modeling to gain insight into the predominant active constituents in the extract.



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