

A Sustainable Approach to Mitigate CO₂ Corrosion by Leveraging Oxazolone Derivatives with Plant-Derived Extracts

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Received 24 January 2025; revised 15 July 2026; accepted 17 August 2026

Sweet corrosion of pipeline steel remains a persistent operational burden for hydrocarbon producers, and it grows more pressing as reservoirs mature and produced fluids carry ever larger volumes of dissolved CO₂. Three oxazolone derivatives were prepared here by a one-pot, metal-free condensation and each was combined with leaf extracts of *Eclipta alba* and *Moringa oleifera*, furnishing six blended formulations that were screened as inhibitors for API5LX60 steel immersed in “3.5% NaCl saturated with CO₂”. The renewable fraction contributed by the extracts, together with the solvent-lean synthetic route, underpins the sustainability of the approach. Protection was quantified by coupon immersion and by three electrochemical routes — impedance spectroscopy, potentiodynamic polarisation and linear polarisation resistance, which returned mutually consistent efficiencies above 90% for every blend at the optimum dose of 100 ppm, establishing that the compounds examined are capable inhibitors. Surface coverage conformed to the Langmuir description, and values of $\Delta G_{\text{ads}}^{\circ}$ between -24.6 and -25.7 kJ mol⁻¹ identified spontaneous adsorption of mixed physisorption-chemisorption character. The interfacial relaxation time lengthened roughly twenty-fold, from 1.60 s in the blank to 32.64 s at 100 ppm of the most effective blend, *ONEA*. Imaging and X-ray microanalysis of corroded and inhibited coupons (SEM, SEM-EDX) confirmed that a protective inhibitive layer had been laid down over the exposed metal.

Keywords: CO₂ corrosion, *Eclipta alba*, *Moringa oleifera*, Oxazolone, Pipelines

Introduction

Deterioration of steel infrastructure ranks among the costliest recurring problems encountered across the oil and gas sector, and it becomes acute wherever the produced stream carries “a high partial pressure of CO₂”.¹ The penalty is counted in economic and in environmental terms alike, since failed equipment must be replaced and escaped fluids remediated.¹ A problem on this scale has pushed operators and researchers together towards protection strategies that are at once effective, affordable and benign towards the surroundings in which CO₂-rich fluids are handled.²

Heterocyclic molecules carrying extended conjugation have long been recognised as efficient retardants of sweet corrosion.^{3–7} Substances as varied as amino-acid derivatives, “3,4-diaminobenzonitrile”⁴ analogues, “mycophenolic acid”⁴, oxadiazoles, “quinolines, imidazolines^{8,9} and amido-imidazolines, azoles”^{4,10}, pyrimidines^{11,12}, benzene- and dodecylbenzene-sulphonamides^{13–15}, polythiazoles¹⁶,

imidazoline-thiones, thiols^{17,18}, polyaspartic acid, imidazole-pyridine hybrids, polypyrrole and mercaptobenzothiazole¹⁸ have all been placed on trial for this purpose.^{19–23} What they share is a complement of chromophoric and auxochromic centres through which the molecule anchors itself to the metal, so that a continuous organic film comes to separate the substrate from the aggressive electrolyte.

Running parallel to this, the drive towards cleaner chemical manufacture has directed attention to inhibitor sources that are inexpensive, abundant and free of toxicity concerns. Botanical extracts satisfy each of these requirements and have accordingly been examined in some depth on carbon steel exposed to CO₂-bearing brine. Preparations obtained from *Momordica charantia*²⁴, *Calotropis procera*²⁵, *Ficus carica*^{26,27}, *Coptis chinensis*²⁸, *Piper nigrum*²⁹ and *Ginkgo biloba*³⁰ fruit, along with the alkaloid berberine, have each afforded measurable protection under such conditions.³¹ Neither family is free of drawbacks, however: synthetic heterocycles frequently dissolve poorly in brine and may bring a toxicity burden with them,³² whereas crude extracts,

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although rich in the same donor functionalities, tend to lose potency on storage.³² Both classes have nonetheless been endorsed in earlier work as viable candidates for service in CO₂ environments.^{4,33}

Thus, this paper seeks to propose and evaluate a novel corrosion inhibition technique that combines oxazolone derivatives with plant-based extracts to provide effective, sustainable, and ecologically friendly corrosion protection in CO₂ environments. Three derivatives of oxazolones namely, “2-Methyl-4-naphthalen-2-ylmethylene-4H-oxazol-5-one (**ON**)”, 4-(2-Methyl-5-oxo-oxazol-4-ylidenemethyl)-benzaldehyde (**OT**) and 4-(2-Methyl-5-oxo-oxazol-4-ylidenemethyl)-benzene (**OB**)”⁴ were blended with extracts of *Eclipta alba* (**EA**) and *Moringa oleifera* (**MO**) and the performance of the six resulting blends on API5LX60 “steel in CO₂-saturated 3.5% NaCl”⁴ was quantified gravimetrically and by impedance, linear polarisation and potentiodynamic measurement. Relaxation-time and adsorption analyses were applied to the fitted electrochemical parameters, and the exposed surfaces were subsequently imaged and microanalysed by SEM and SEM-EDX. The results establish that these combinations deliver a level of protection in the CO₂-saturated medium that neither constituent reaches on its own.

Materials and Methods

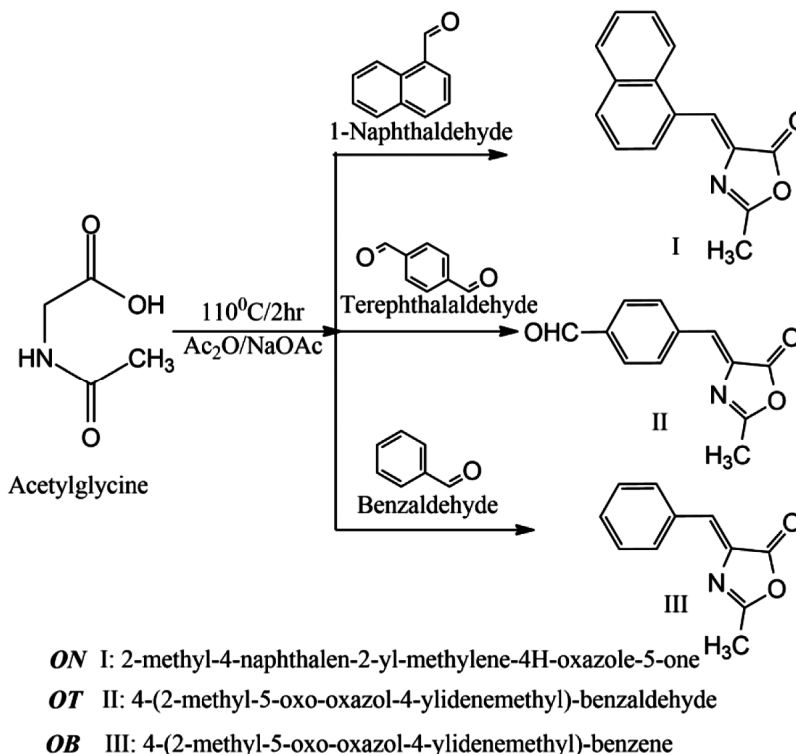
Preparation of Organic Inhibitor

Synthesis of Oxazolone

Scheme 1 sets out the route by which the three oxazolones were obtained.^{19,34} Crude material was recrystallized from ethyl acetate and hexane mixed in a 30:70 ratio, and homogeneity of the products was checked chromatographically on TLC plates developed in hexane containing 20% ethyl acetate. Infrared spectra of the solids were collected on a SHIMADZU IR Affinity-1 spectrophotometer, while ¹H and ¹³C NMR spectra were referenced against internal TMS.

Preparation of Plant Extract

Foliage of the two species “*Eclipta alba* (**EA**)”, and *Moringa oleifera* (**MO**)”³³ was collected locally while fresh, then dried, first under sunlight and afterwards for one night in an oven maintained at 100°C, so that any moisture retained by the tissue was driven off. The dried material was milled to a fine powder, and 10 g portions of each were taken up in 100 mL of ethanol and heated under reflux at 60°C for 6 h in a round-bottomed flask, the mixture then being left to stand overnight at ambient temperature. Working solutions of 40, 60, 80 and 100 ppm were obtained by diluting the filtered stock.



Scheme 1 — ‘Synthesis of oxazolones’^{19,34}

Preparation of Blending Extract

Each oxazolone was combined in equal proportion with the *Eclipta alba* or *Moringa oleifera* extract solution and the combination heated separately under reflux at 60°C for 3 h in a round-bottomed flask. After standing overnight the liquors were filtered, which furnished six blends for evaluation: *ONEA* and *ONMO* from *ON* with *EA* and *MO* respectively, *OTEA* and *OTMO* from *OT*, and *OBEA* and *OBMO* from *OB*. Each was diluted to 40, 60, 80 and 100 ppm for the inhibition study.

Preparation of Steel Specimen and Test Solutions

Coupons measuring $3.8 \times 1.0 \times 0.8 \text{ cm}^3$ and presenting 0.8 cm^2 of exposed metal served for the immersion tests, whereas cubes of $1.0 \times 1.0 \times 1.0 \text{ cm}^3$ were mounted for the electrochemical cell. Before every experiment the working faces were ground on “emery paper, washed with double-distilled water, wiped with acetone to remove grease and dried”³³ between 60-70°C. The API5LX60 carbon steel employed throughout analyzed as “C 0.28%, Mn 1.4%, P 0.3%, S 0.3%, with V, Nb and Ti each $\leq 15\%$ and iron making up the balance”.³³ Test electrolyte was made up to 3.5% NaCl from analytical-reagent salt and double-distilled water, and was sparged to saturation with CO_2 at 700 kPa.

Gravimetric Studies

For the weight loss studies, “the steel coupons were immersed in the test solution in uninhibited and inhibited conditions”³³ with four concentrations (40, 60, 80 and 100 ppm) of the six-blending extract at room temperature (25–28°C) for 24 hours. “Over the 100 ppm, the weight loss outcomes were inconsequential. Henceforth, the optimal concentration was set at 100 ppm. Percentage Inhibition efficiencies (%IE) for different inhibitor concentration for the steel specimen”⁴ was elucidated using Eq. (1).²⁰

$$\%IE = \left[\frac{\Delta W}{W} \right] \times 100 \quad \dots (1)$$

where, $\Delta W = W - W_i$, “ W = average weight loss in uninhibited condition, and W_i = average weight loss in inhibited condition

The corrosion rate (CR) was calculated using Eq. (2).²⁰

$$\text{Corrosion Rate} = \frac{K \times \Delta W}{A \times t \times d} \quad \dots (2)$$

where, K = constant (8.76×10^6) that allows to represent CR in mm/yr; A = surface area of metal

sample (in cm^2); t = immersion time (hours); d = density of the metal (g/cm^3), and ΔW = average weight loss (g).⁴

Electrochemical Studies

All electrochemical runs were performed at laboratory temperature on a “Metrohm Autolab PGSTAT302N potentiostat/galvanostat”⁴ in accordance with “ASTM G-59 and ASTM G-102”^{4,35}. The cell followed the conventional three-electrode design, with Ag/AgCl serving as reference, stainless steel (SS316) as counter and the cold-mounted specimen, 1 cm^2 of which lay exposed, as working electrode. Thirty minutes of immersion preceded each measurement so that a steady Open Circuit Potential (OCP) was established. Impedance spectra were acquired potentiostatically between “100 kHz and 10 mHz using a perturbation of $\pm 10 \text{ mV}$, and were fitted to the equivalent circuit”⁴ reproduced in Fig. 1, whose elements are “the solution resistance (R_s), the polarisation resistance (R_p), a constant phase element (CPE) and the phase exponent (n); values of these elements emerged from non-linear curve fitting”⁴. The CPE Impedance is expressed by Eq. (3).³³

$$Z_{CPE} = q^{-1}(i\omega)^{-n} \quad \dots (3)$$

“where, the CPE’s magnitude is q , ω , the angular frequency, and n , the phase shift.”⁴ The % Inhibition Efficiency from EIS measurements were done using the Eq. (4).³³

$$\%EIS = \left(1 - \frac{R_p}{R_p^I} \right) \times 100\% \quad \dots (4)$$

where, R_p and R_p^I are the polarisation resistances measured without and with inhibitor; R_p itself is the sum of a film resistance R_f and charge-transfer resistance R_{ct} .⁴ “The double layer capacitance values (C_{dl})”⁴ was computed using Eq. (5), at the frequency at which the imaginary component of the spectrum is maximum ($I_{\max}$).^{4,33}

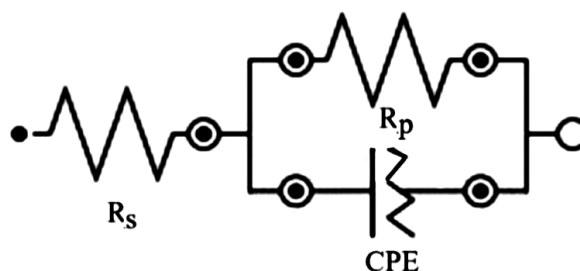


Fig. 1 — Equivalent circuit model

$$C_{dl} = \frac{1}{2\pi I_{max} R_{ct}} \quad \dots (5)$$

“LPR, at a potential range of ± 10 mV from OCP was measured at 0.125 mV/s scan rate. Here, corrosion current and potential linearly correlates by a slight polarization of about ± 10 mV relative to OCP. The LPR parameters were again obtained from curve fitting. Further, PDP was performed at about ± 250 mV potential ranges from OCP and 0.25 mV/s sweep rate. Data reduction and fitting throughout were handled with Nova software.”⁴

The characterizations of the blends as obtained from FTIR and UV-Visible spectroscopy are given below:

OBEA: IR (KBr: γ/cm^{-1}) 3854, 3446.3, 2368.9, 1653.5, 1508.6, 1374.5, 1260.1, 984.56; UV Visible: λ_{max} : 400 nm (abs 0.098), 367.8 nm (1.007), 297.6 (1.563)

OBMO: IR (KBr: γ/cm^{-1}) 3278, 2604, 1652, 1522, 1374, 1263; UV Visible: λ_{max} : 411 nm (abs 0.088), 382.6 nm (2.04), 308.5 (1.78)

OTEA: IR (KBr: γ/cm^{-1}) 3426, 2112, 1650, 1498, 1396, 1213; UV Visible: λ_{max} : 413 nm (abs 0.079), 378.8 nm (1.37)

OTMO: IR (KBr: γ/cm^{-1}) 3426, 2367, 1641, 1489, 1398, 1213; UV-Visible: λ_{max} : 409 nm (abs 0.089), 389 nm

ONEA: IR (KBr: γ/cm^{-1}): 3400, 2949, 2078, 1653, 1489, 1397, 1200; UV-Visible: λ_{max} : 408 nm (abs 0.092), 329.7 nm

ONMO: IR (KBr: γ/cm^{-1}): 3431, 2921, 2198, 1647, 1509, 1375, 1244, 1034; UV-Visible: λ_{max} : 392.7 nm (abs 0.218), 336.6 nm (1.54)

Results and Discussion

Gravimetric Weight Loss Analysis

The gravimetric weight loss data³³ for the six blends are presented in Table 1 and are displayed graphically in Fig. 2, where Fig. 2(a) traces efficiency and Fig. 2(b) corrosion rate against inhibitor concentration. Both plots behave as an adsorptive inhibitor showing all in metal loss as the concentration of the blend inhibitor increased and reaches its minimum at 100 ppm, while efficiency climbs correspondingly. Among the studied extracts **ONEA** extracts, i.e. blending of “2-Methyl-4-naphthalen-2-ylmethylene-4H-oxazol-5-one (**ON**)”⁴

Table 1 — Gravimetric analysis data

Sample	IC (ppm)	Weight loss (in g)	Corrosion rate (mmpy)	% IE ⁴
Blank	0	0.32	14.97	—
OBEA	40	0.156	7.32	51.25
	60	0.113	5.91	64.68
	80	0.083	3.22	74.05
	100	0.056	1.09	82.5
OBMO	40	0.163	7.62	50.93
	60	0.124	5.99	61.25
	80	0.088	3.37	72.5
	100	0.059	1.10	81.56
OTEA	40	0.136	6.03	57.5
	60	0.093	4.01	70.93
	80	0.056	2.20	82.46
	100	0.032	0.99	89.96
OTMO	40	0.141	6.11	55.93
	60	0.099	4.23	69
	80	0.076	2.83	75.96
	100	0.045	0.99	85.93
ONEA	40	0.113	5.11	64.68
	60	0.073	3.89	76.96
	80	0.033	2.00	89.65
	100	0.016	0.71	94.84
ONMO	40	0.128	5.36	60
	60	0.099	3.99	68.8
	80	0.045	2.14	85.87
	100	0.024	0.89	92.43

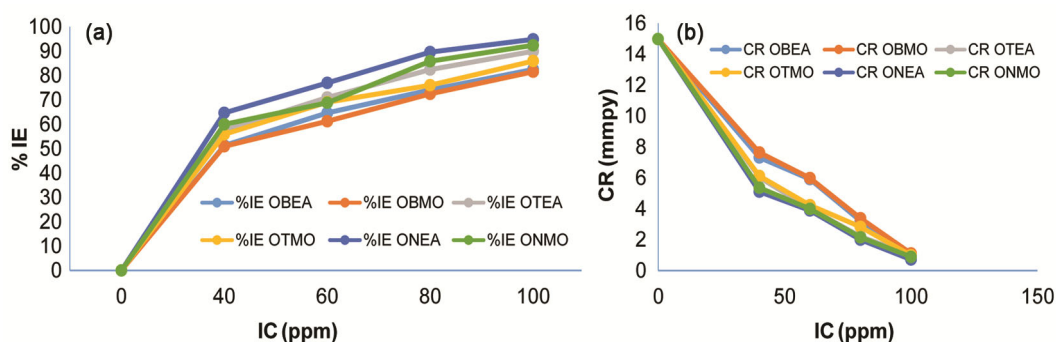


Fig. 2(a) — Plot of % inhibition efficiency Vs inhibitor concentration and (b) Plot of corrosion rate Vs inhibitor concentration for blend samples⁴

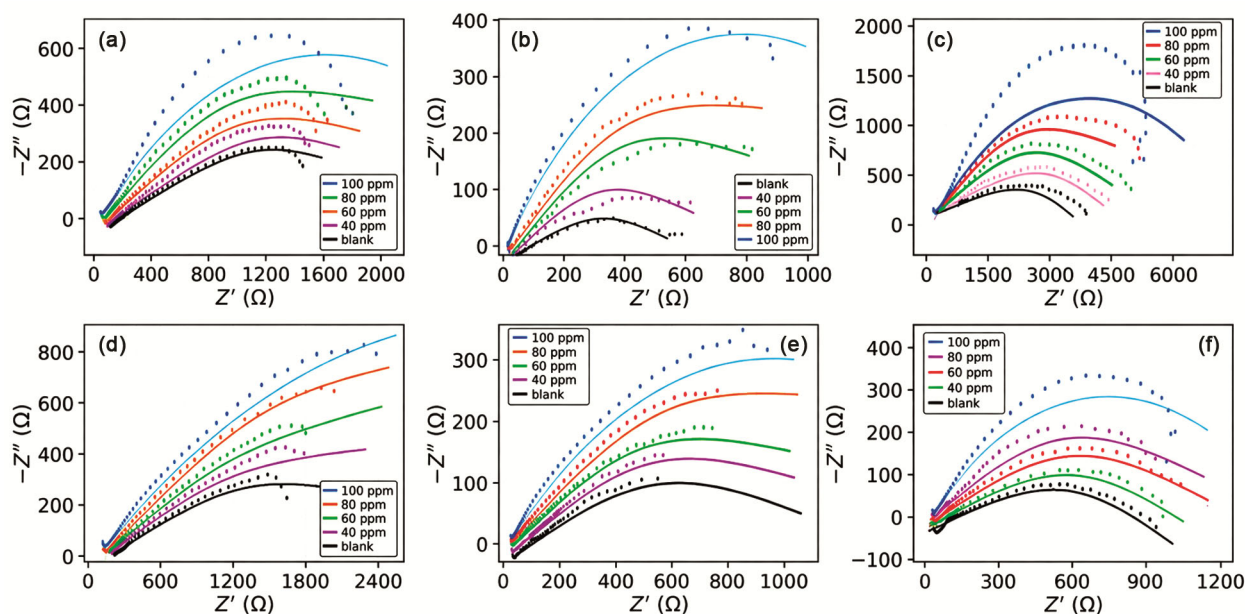


Fig. 3 — Nyquist plots for blends of (a) *OBEA* extracts, (b) *OBMO* extracts, (c) *OTEA* extracts, (d) *OTMO* extracts, (e) *ONEA* extracts, (f) *ONMO* extracts in different concentration

and “*Eclipta alba* (*EA*)”³³ showed some significant results with 94.84% of inhibition efficiency at 100 ppm concentration.

Electrochemical Impedance Spectroscopy

The electrochemical Impedance responses were examined in the “Nyquist, Bode-phase and Bode-modulus”⁴ representations, the first of which is reproduced as Fig. 3(a–f). Attainment of depressed semi-circular arc of Nyquist plots signifies capacitive polarization predominantly. Two-time constants are resolved in the Bode plots: the one lying at higher frequency is assigned to the organic layer adsorbed on the steel, viz. $R_f//CPE_f$, and the one at lower frequency to the “electrical double layer established at the electrode-electrolyte interface”³³, viz. $R_{ct}//CPE_{dl}$. Parameters recovered from the curvefits are

set out in Table 2, where CPE exponents close to unity again point to near-capacitive behaviour.³⁶ R_p rises with each increment of blend concentration, as would follow from progressive blocking of the metal by an adsorbed film and from the consequent hindrance to charge and mass transport across the interface; the maximum in every series falls at 100 ppm, and the solution resistance R_s follows the same pattern. Efficiency from impedance data was computed using Eq. (6).

$$\%IE_{EIS} = \left(\frac{R_p^I - R_p}{R_p^I} \right) \times 100 \quad \dots (6)$$

where, R_p and R_p^I denote the polarization resistance of the uninhibited and the inhibited solution. The gain in efficiency with concentration reflects the larger fraction of surface occupied by inhibitor molecules,

Table 2 — EIS measurements data

Inhibitor name	IC (in ppm)	CPE.Y0 (Ω ⁻¹ s ⁿ) ⁴	R _p .R (Ω) ⁴	R _s .R (Ω) ⁴	CPE.N	%IE	τ(s)
Uninhibited	0	0.00421	377.42	46.77	0.99201	—	1.60
	40	0.00012	802.3	50.96	0.99311	52.96	0.10
OBEA	60	0.00032	1045.8	213.73	0.99769	63.95	0.33
	80	0.00051	1511.3	852.39	0.99489	75.02	0.77
	100	0.00064	1875.6	1875.6	0.98436	79.88	1.20
OBMO	40	0.00019	812.58	19.71	0.4277	53.55	0.01
	60	0.00053	1524.8	76.28	0.62217	75.24	0.71
	80	0.00058	2054.9	105.78	0.97861	81.63	1.20
OTEA	100	0.00061	2776.5	110.86	0.60416	86.40	2.39
	40	0.00008	855.8	25.28	0.51601	55.90	0.01
	60	0.00009	896.9	193.27	0.8043	57.92	0.04
OTMO	80	0.00016	1759.1	275.2	0.99763	78.54	0.28
	100	0.00069	2270.5	291	0.99017	83.38	1.57
OTMO	40	0.00043	921.0	301.9	0.99664	59.02	0.40
	60	0.00045	1498.1	380	0.98976	74.82	0.67
	80	0.00098	2691.7	411.8	0.99611	85.98	2.65
ONEA	100	0.00192	3927	501.61	0.99265	90.39	7.65
	40	0.00023	874.2	375.05	0.99043	56.82	0.20
	60	0.00169	1411.4	425.1	0.94252	73.26	2.52
ONMO	80	0.00395	4813.6	717.87	0.98976	92.16	19.60
	100	0.00469	6765.9	896.9	0.99195	94.42	32.64
	40	0.000126	1065.1	340.62	0.99432	64.60	0.13
ONMO	60	0.000271	2742.8	451	0.99219	86.24	0.74
	80	0.000301	3871.3	498.2	0.99038	90.25	1.17
	100	0.000457	7659.8	869.5	0.99198	92.07	3.54

and the values so obtained are itemized in Table 2, where efficiency is seen to track R_p throughout. **ONEA** once more heads the ranking, recording 94.42% at 100 ppm, a figure in close accord with the gravimetric estimate.

Observations made from EIS plots suggest that, the semi-circular Nyquist plots diameter is larger for the inhibited solutions of all the six blends than the uninhibited “in 3.5% NaCl solution with CO₂ saturation”⁴ and increases as concentration of blend increases. This is depicted in Fig. 3(a–f). For the Bode plots, the circular portion’s diameter also becomes larger for inhibited solutions. In case of Bode modulus plots, the linear portion became more pronounced for the inhibited solutions.

The characteristic time constant of the electrode-electrolyte interface was further evaluated from the fitted impedance parameters, following the treatment adopted for relaxation phenomena in impedance and dielectric spectroscopy.^{37–39} For a resistance “in parallel with a constant phase element”⁴, the interfacial relaxation time (τ) and the corresponding relaxation frequency (f_{rel}) are given by Eqs (7) & (8):

$$\tau = (Y_0 R_p)^{1/n} \quad \dots (7)$$

$$f_{rel} = 1/(2\pi\tau) \quad \dots (9)$$

where, Y_0 (Ω⁻¹ sⁿ) and n are the magnitude and the exponent of the constant phase element and “ R_p is the polarization resistance”⁴. The computed values are listed in Table 2. The relaxation time for the uninhibited electrolyte was 1.60 s ($f_{rel} \approx 0.10$ Hz) and increased progressively with inhibitor concentration for all six blends, reaching 32.64 s ($f_{rel} \approx 5$ mHz) for **ONEA** at 100 ppm, a twenty-fold retardation relative to the blank. The lengthening of τ reflects a slower exchange of charge across the interface as the adsorbed inhibitor layer becomes more compact and more extensive, and the sequence of τ values across the six blends reproduces the sequence of inhibition efficiencies obtained independently from the gravimetric, LPR and PDP measurements. For the most efficient blends the relaxation frequency approaches the lower limit of the applied frequency window, and the corresponding τ values are therefore reported as estimates derived from the fitted parameters.

Table 3 — LPR measurements data

Inhibitor name	IC (in ppm)	E_{corr} (V)	R_p (Ω)	Corrosion rate (mmpy)	χ^2 ($\times 10^{-10}$)	%IE
Uninhibited	0	-0.49389	205.27	2.1514	0.107	—
	40	-0.47514	420.62	0.7232	0.849	51.19
	60	-0.55891	551.08	0.5354	0.290	62.80
<i>OBEA</i>	80	-0.51367	1094.6	0.4151	0.262	81.26
	100	-0.48988	1184.9	0.2416	0.143	82.67
	40	-0.51834	450.2	0.7423	0.230	54.4
<i>OBMO</i>	60	-0.49261	627.3	0.5082	0.136	72.78
	80	-0.55121	1239.7	0.3710	0.093	83.44
	100	-0.42455	1463.1	0.1621	0.012	85.96
<i>OTEA</i>	40	-0.51943	615.8	0.6239	0.650	66.67
	60	-0.50164	754.24	0.4092	0.132	72.78
	80	-0.54761	1323.8	0.2037	0.123	84.49
<i>OTMO</i>	100	-0.42135	1732.5	0.1147	0.046	88.15
	40	-0.44167	527.8	0.6342	0.860	61.11
	60	-0.52415	667.3	0.4389	0.612	69.24
<i>OTMO</i>	80	-0.48712	1278.9	0.2181	0.112	83.94
	100	-0.43116	1690.5	0.1035	0.054	87.85
<i>ONEA</i>	40	-0.52977	541.14	0.4881	0.348	62.06
	60	-0.53895	804.17	0.2345	0.226	74.47
	80	-0.51281	1239.5	0.1317	0.131	83.44
<i>ONMO</i>	100	-0.49916	2987.1	0.0821	0.093	93.13
	40	-0.55129	623.3	0.4237	0.430	67.07
	60	-0.4616	968.5	0.4018	0.336	78.8
<i>ONMO</i>	80	-0.44318	1412.2	0.2386	0.192	85.48
	100	-0.41724	2334.9	0.1328	0.127	91.28

Linear Polarization Resistance

Corrosion potential (E_{corr}) and polarisation resistance (R_p) obtained from the LPR fits are listed in Table 3, and efficiencies were calculated from these resistances using Eq. (9).

$$\%IE_{LPR} = \left(\frac{R_p^I - R_p}{R_p^I} \right) \times 100 \quad \dots (9)$$

where, R_p and R_p^I again refer to the uninhibited and the inhibited solution

Data in Table 3 affirms that R_p becomes higher for inhibited solution with maximum at 100 ppm inhibitor concentration. E_{corr} conversely, becomes less negative for the inhibited solutions although they were neither increasing nor decreasing suggesting mixed type of behaviour of the studied inhibitor. Also, the % inhibition efficiency surges with rising inhibitor concentration, the highest being 93.13% for *ONEA* blend at 100 ppm.

Potentiodynamic Polarization Study

Tafel polarization plots recorded for the six blends are represented as Fig. 4(a–f). Quantities extracted from

the Tafel fit, “corrosion potential (E_{corr}), corrosion current (I_{corr}), the cathodic and anodic slopes (β_c , β_a) and the corrosion rate in mmpy”⁴ — are presented in Table 4, together with efficiencies obtained from Eq. (10).³³

$$\%IE_{PDP} = \left(\frac{I_{corr} - I_{corr}^I}{I_{corr}} \right) \times 100 \quad \dots (10)$$

“where, I_{corr} and I_{corr}^I are the corrosion current for uninhibited and inhibited solutions respectively”.³³ The corrosion parameters obtained from Tafel fit along with calculated % IE values are tabulated in Table 4.

Analysis of Table 4 reveals that the cathodic current densities decreased drastically for the inhibited solution. This can be explained because of inhibition of cathodic reactions by the inhibitors. Also, corrosion current (I_{corr}) for the inhibited solutions depletes than the uninhibited solutions which reduces steadily as inhibitor concentration raise. Lowest I_{corr} was possessed by extracts of *ONEA* at 100 ppm concentration. Inhibited solutions showed decreased negative E_{corr} values, but

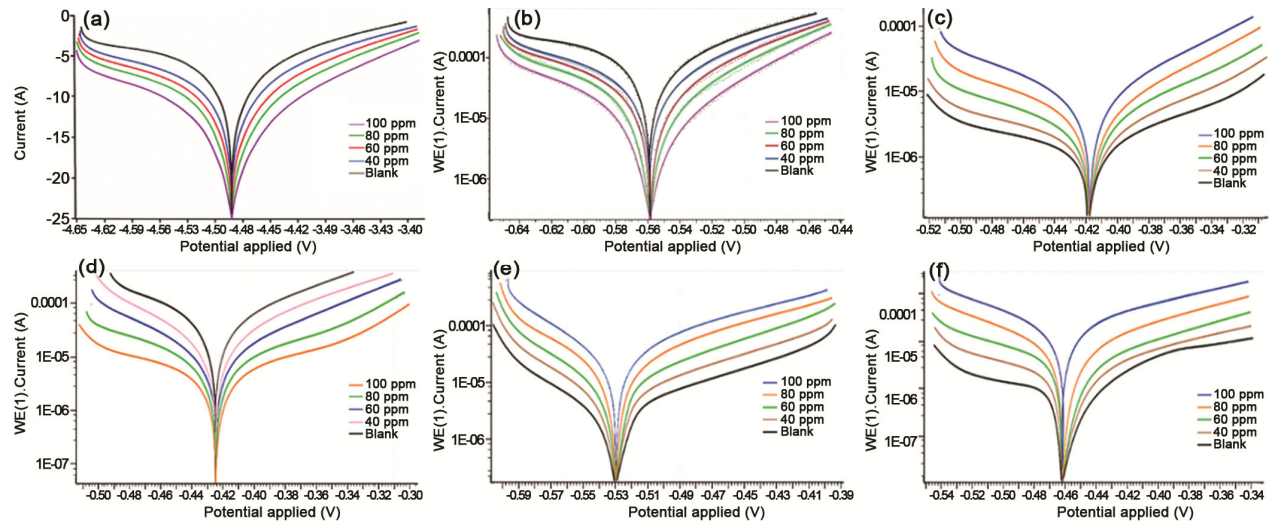


Fig. 4 —Tafel plots for blends of (a)-OBEA extracts, (b)-OBMO extracts, (c)-OTEA extracts, (d)-OTMO extracts, (e)-ONEA extracts, (f)-ONMO extracts in different concentration

Table 4 — PDP measurements data

Inhibitor name	IC (in ppm)	I_{corr} (A)	Corrosion rate (mmpy)	E_{corr} (V)	β_a (V/dec)	β_c (V/dec)	%IE
Uninhibited	0	0.000159	2.1514	-0.49786	0.10578	0.26088	—
OBEA	40	0.0000585	0.8338	-0.5435	0.11297	0.06429	64.9
	60	0.0000435	0.41087	-0.55632	0.11361	0.08345	72.64
	80	0.0000318	0.40751	-0.48794	0.11388	0.11274	80.0
	100	0.0000205	0.23867	-0.44394	0.1141	0.16107	87.1
	100	0.0000205	0.23867	-0.44394	0.1141	0.16107	87.1
OBMO	40	0.0000561	1.0458	-0.50615	0.13837	0.3073	62.64
	60	0.0000430	0.70928	-0.51376	0.13512	0.11351	72.95
	80	0.0000308	0.51512	-0.44231	0.11234	0.12629	80.62
	100	0.0000279	0.21118	-0.48986	0.12986	0.15296	82.45
	100	0.0000279	0.21118	-0.48986	0.12986	0.15296	82.45
OTEA	40	0.0000549	0.83541	-0.56043	0.11684	0.182	65
	60	0.0000408	0.53961	-0.54912	0.10341	0.13487	74.33
	80	0.0000197	0.31298	-0.55478	0.10026	0.12382	87.61
	100	0.0000133	0.23471	-0.4229	0.095985	0.11925	91.63
	100	0.0000133	0.23471	-0.4229	0.095985	0.11925	91.63
OTMO	40	0.0000535	0.89783	-0.51762	0.11458	0.11264	66.35
	60	0.0000428	0.55393	-0.44378	0.094181	0.14519	73.08
	80	0.0000221	0.30657	-0.43561	0.093291	0.097101	86.1
	100	0.000014	0.16213	-0.42637	0.096085	0.092024	91.19
	100	0.000014	0.16213	-0.42637	0.096085	0.092024	91.19
ONEA	40	0.0000456	0.65481	-0.44981	0.10973	0.10645	69.49
	60	0.0000353	0.48818	-0.53181	0.10371	0.076342	77.79
	80	0.0000207	0.27318	-0.51864	0.09145	0.09875	86.98
	100	0.0000111	0.15355	-0.43078	0.076104	0.099952	93.01
	100	0.0000111	0.15355	-0.43078	0.076104	0.099952	93.01
ONMO	40	0.0000485	0.72322	-0.47899	0.14979	0.068528	69.49
	60	0.0000315	0.40188	-0.45995	0.15323	0.12951	80.38
	80	0.0000228	0.28704	-0.54902	0.10251	0.12146	85.66
	100	0.0000114	0.13283	-0.41728	0.10159	0.15554	92.83
	100	0.0000114	0.13283	-0.41728	0.10159	0.15554	92.83

the absence of any systematic displacement in one direction again identifies the blends as inhibitors of hybrid character. Percent IE based on PDP data was obtained for **ONEA** extract at 100 ppm concentration being 93.01%. Corrosion rates

obtained from PDP measurements suggest that inhibited solutions have lower corrosion rates than the uninhibited solutions. **ONEA** extracts demonstrated high inhibitory efficiency and corrosion rates.

Table 5 — Adsorption parameters obtained from the Langmuir Isotherm³³

Blend	K_{ads} (ppm ⁻¹)	R^2	$\Delta G^\circ_{\text{ads}}$ (kJ mol ⁻¹)
<i>OBEA</i>	0.0203	0.999	-24.7
<i>OBMO</i>	0.0193	0.986	-24.6
<i>OTEA</i>	0.0235	0.999	-25.1
<i>OTMO</i>	0.0241	0.992	-25.2
<i>ONEA</i>	0.0295	0.995	-25.7
<i>ONMO</i>	0.0232	0.960	-25.1

Adsorption Isotherm and Thermodynamics of Adsorption

The manner in which the blends engage API5LX60 was probed by testing coverage values derived from the gravimetric efficiencies ($\theta = \%IE/100$) against the Langmuir expression, Eq. (11); the standard free energy of adsorption then follows from Eq. (12).^{14,21}

$$C/\theta = 1/K_{\text{ads}} + C \quad \dots (11)$$

$$\Delta G^\circ_{\text{ads}} = -RT \ln(10^6 K_{\text{ads}}) \quad \dots (12)$$

where, C denotes the blend concentration in ppm and K_{ads} the equilibrium constant of adsorption, while R , T and the factor 10^6 stand respectively for “the gas constant, the absolute temperature (300 K) and the ppm concentration”⁴ of water present in the medium. Plotting C/θ against C yielded straight lines for all six blends, with coefficients of determination lying between 0.960 and 0.999, which places the adsorption within the Langmuir description and implies that coverage is monolayer in nature. The slopes, however, fall between 0.65 and 0.76 and so depart measurably from the ideal value of unity; the departure is attributed to lateral interaction among the adsorbed species and to the multi-component make-up of the blends, and the treatment is accordingly described as a modified Langmuir fit. Parameters derived from the isotherm are compiled in Table 5.

The values of $\Delta G^\circ_{\text{ads}}$ lie between -24.6 and -25.7 kJ mol⁻¹ and are negative for every blend, indicating that adsorption on the steel surface is spontaneous. These magnitudes are more negative than the value of about -20 kJ mol⁻¹ associated with purely electrostatic physisorption, yet appreciably less negative than the value of about -40 kJ mol⁻¹ associated with charge sharing chemisorption, and therefore point to a mixed mode of adsorption in which physical interaction predominates while a chemical contribution is retained. This inference is consistent with the mechanism advanced in the following section on the basis of the π -electron systems and the heteroatoms present in the oxazalone and plant-extract constituents. The most negative

value of $\Delta G^\circ_{\text{ads}}$ and the largest K_{ads} were recorded for **ONEA**, in agreement with its superior inhibition performance in all four techniques employed. It should be noted that the present measurements were carried out under a single ambient condition; the apparent activation energy of the corrosion process and the associated enthalpy and entropy of adsorption require a controlled temperature series and were therefore outside the scope of this study.

Scanning Electron Microscopy (SEM) and Energy Dispersed SEM³⁴

Micrographs of API5LX60 coupons exposed to 100 ppm of *OBEA*, *OBMO*, *OTEA*, *OTMO*, *ONEA* and *ONMO* are assembled in Fig. 5(a), (b), (c), (d), (e) and (f) at 200x magnification, Fig. 5(g), (h), (i), (j), (k) and (l) at 500x magnification and Fig. 5(m), (n), (o), (p), (q) and (r) at 950x magnification respectively. The surface of inhibited X60 appears to be relatively homogeneous which can be inferred due to effective inhibitor coating of active components on the steel.

The SEM-EDX spectrum imaging along with elemental composition is portrayed in Fig. 6 where Fig. 6(a) depicts the untreated surface and Fig. 6(b) images the coupon treated with **ONEA**. Iron dominates the spectrum of the uninhibited specimen but falls away sharply once the blend has been applied, while the carbon and oxygen signals strengthen, precisely the distribution expected where a film built from the organic components of the blend covers the steel.

Mechanism of Inhibition

Conjugated heterocycles carrying π -electron density together with heteroatom lone pairs are generally held to protect metals by assembling a metal-inhibitor complex at the surface.¹⁹ Three modes of attachment may be envisaged for the present system. The first is electrostatic attraction “between the cationic form of the active component³³ and the anions of the aggressive medium already accumulated on the steel. The second is donation from the π cloud of the aromatic rings into vacant d orbitals of surface

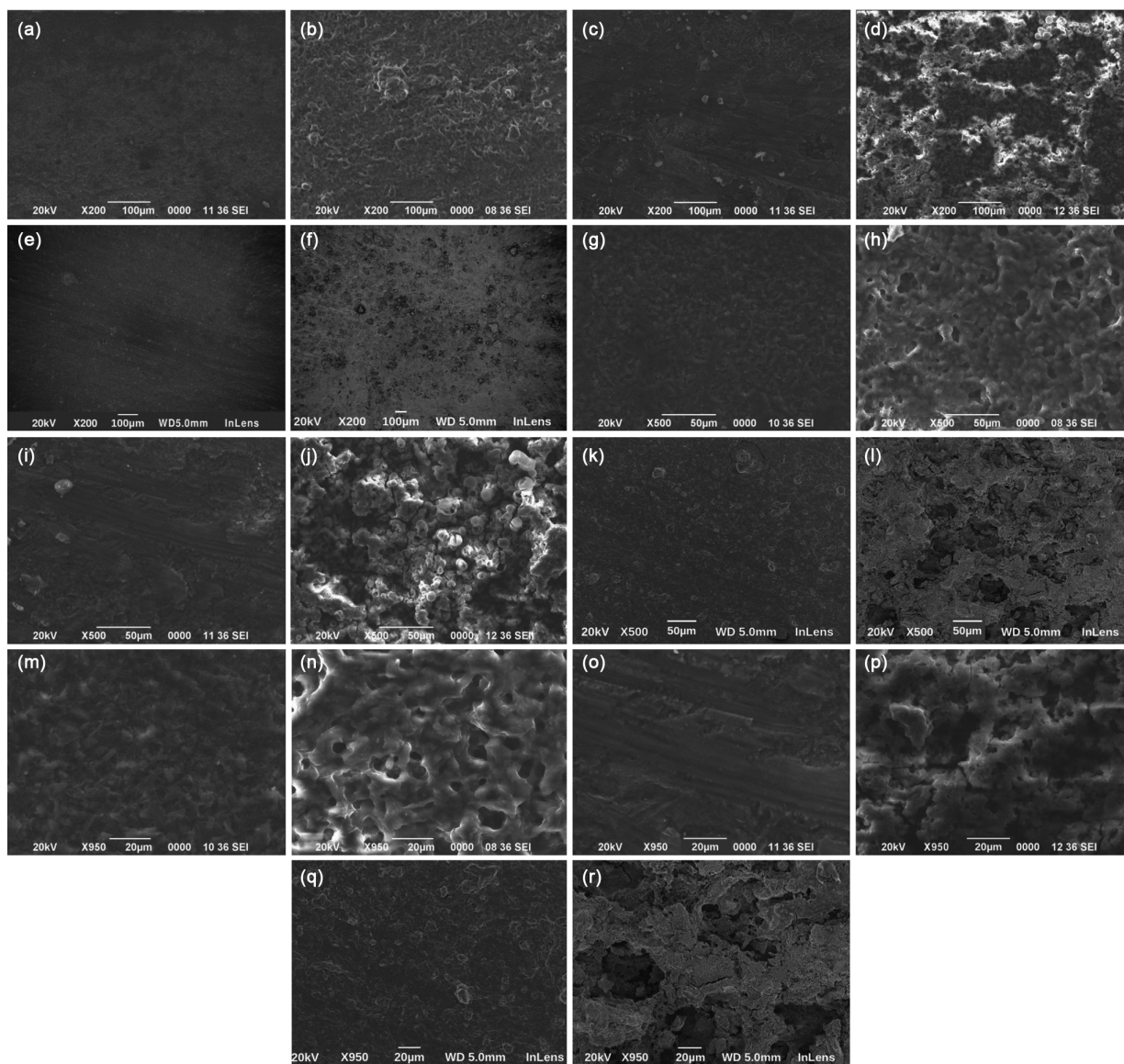


Fig. 5 — SEM Micrograph at 200x magnification for Blends of (a)- *OBEA* Extracts, (b)- *OBMO* Extracts, (c)- *OTEA* Extracts, (d)- *OTMO* Extracts, (e)- *ONEA* Extracts, (f)- *ONMO* Extracts, at 500x magnification for Blends of (g)- *OBEA* Extracts, (h)- *OBMO* Extracts, (i)- *OTEA* Extracts, (j)- *OTMO* Extracts, (k)- *ONEA* Extracts, (l)- *ONMO* Extracts and at 950x magnification for Blends of (m)- *OBEA* Extracts, (n)- *OBMO* Extracts, (o)- *OTEA* Extracts, (p)- *OTMO* Extracts, (q)- *ONEA* Extracts, (r)- *ONMO* Extracts at 100ppm concentration

iron atoms³³. The third is coordination of the unshared electron pairs resident on the heteroatoms to those same vacant orbitals.¹⁹ Physical and chemical binding are therefore both available to the adsorbate, and the barrier so assembled over API5LX60 is what suppresses attack by the electrolyte, through a mixed physisorption-chemisorption mechanism.

Comparative Study of Reported Inhibitor

Performance of the blends measured against that of their individual constituents in CO₂-saturated 3.5% NaCl is summarized in Table 6.

The inhibition efficacy of the ONEA blend was found to be higher than that of ON and EA when the best results were compared. Similarly, ONMO blend gave better inhibition performance than that of ON and MO individually. Consequently, a low in corrosion rates have been observed in case of the blends. A synergistic effect has been observed in the inhibition action of both oxazolone and plant extract when blended. Thus, it may be concluded that the blend possessed better inhibition efficiency in comparison to oxazolone derivatives and plant extracts when studied individually.

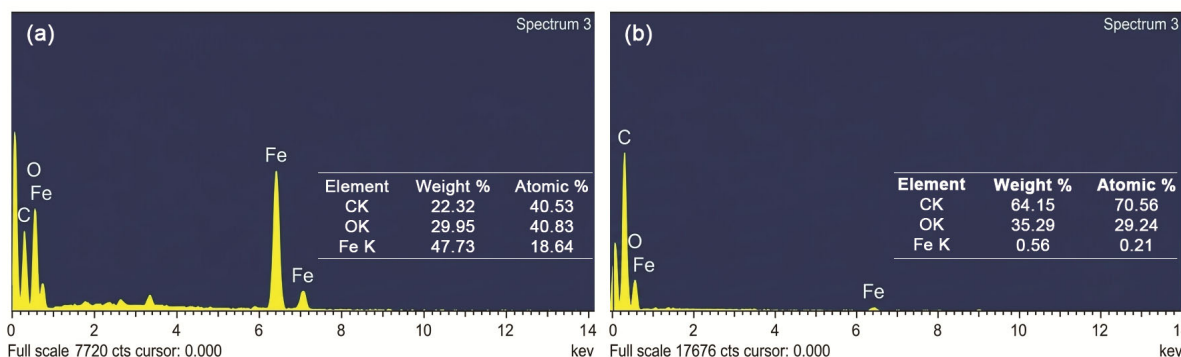


Fig. 6 — (a) 'SEM-EDX Spectrum for Uninhibited Steel, (b) SEM-EDX Spectrum for Inhibited Steel with Extract of *ONEA*'

Table 6 — Comparison of inhibitive properties of some selected studied inhibitor³³

Inhibitor name	Inhibitor concentration (ppm)	Corrosion rate (mmpy)	% Inhibition efficiency
<i>ON</i>	200	0.19	90.70
<i>EA</i>	100	0.1029	92
<i>MO</i>	100	0.2134	91.28
<i>ONEA</i>	100	0.0821	93.1
<i>ONMO</i>	100	0.1328	92.89

Conclusions

Organic molecules and plant-derived extracts have both attracted sustained attention as corrosion inhibitors, yet the protection each affords when used singly is often modest. The present study addressed that limitation by pairing three oxazolone derivatives with extracts of *Eclipta alba* and *Moringa oleifera* and evaluating the six resulting blends on API5LX60 steel in 3.5% NaCl saturated with CO₂. **ONEA** emerged as the most capable formulation, exceeding 93% efficiency at 100 ppm, and the gravimetric data agreed closely with the impedance, linear polarization and potentiodynamic results in respect of both % inhibition efficiency and corrosion rate. Coverage obeyed the Langmuir isotherm and yielded $\Delta G^{\circ}_{\text{ads}}$ values between -24.6 and -25.7 kJ·mol⁻¹, consistent with spontaneous attachment in which physical and chemical contributions are combined, while the relaxation time at the interface, recovered from the impedance fits, lengthened twenty-fold once the most effective blend was present. Examination of the surfaces by SEM and SEM-EDX confirmed the growth of a protective barrier film. The blends therefore offer appreciable protection against CO₂ corrosion of API5LX60 gas pipeline steel. A temperature-resolved investigation of **ONEA**, from which activation energies together with the enthalpy and entropy of adsorption may be derived, is now under way and will form the subject of a separate report.

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.

Acknowledgement

The authors acknowledge the DST PURSE project, Dibrugarh University (SR/PURSE/2022/143 (C)) for funding the project. The authors also acknowledge the Local management (Prankush M. Bujarbaruah, and Uddipana S. Deka) of Oil India Limited, Duliajan, for supporting in initiating the Industry-Academia Collaborative study for the betterment of the two organizations. Acknowledgments are also due to the support provided by the technical staff of various laboratories in the chemical and R&D Departments of OIL and Dibrugarh University. The authors also acknowledge the North East Institute of Science and Technology (NEIST), Jorhat, and Assam for SEM and SEM-EDX analysis.

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