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Guanidine as a Starting Material for Preparing a Scale Inhibitor

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Abstract

Oilfield scale inhibition, which can obstruct fluid flow in pipelines, valves, and pumps during oil production and processing, is critical for preventing scale formation. Effective scale inhibitors (SIs) minimize downtime and enhance productivity by preventing the build-up of insoluble crystals in water systems. In oil processing, scale formation results from the precipitation of salts due to incompatible aqueous phases, leading to equipment blockage. Phosphonates are highly effective chelating agents for divalent and trivalent metal ions, capable of inhibiting crystal growth and scale formation under extreme conditions, such as high temperatures and fluctuating pH levels. These properties make them valuable in oilfields, cooling systems, desalination, pulp, paper, textiles, and detergents, where they function as SIs, chelating agents, and bleach stabilizers. In this study, ((1-methyl-3,3-bis(phosphonomethyl)guanidino) methyl) phosphonic acid was synthesized, and its effectiveness in reducing scale deposition in pipelines was evaluated. The compound chelates metal ions and inhibits crystal growth. Guanidine was selected as the starting material owing to its potential for phosphonate substitution. Although the initial aim was to introduce five phosphonate groups, steric hindrance and nitrogen bond stability resulted in the successful incorporation of these three groups. The structures and compositions of the synthesized compounds were confirmed by NMR, FT-IR, and mass spectrometry. Further evaluation of its scale inhibition efficiency will provide insights into its potential as an advanced inhibitor in oilfield applications.

Keywords: Desalination, guanidine, oilfield scaling, phosphonates, scale inhibitor

INTRODUCTION

Oilfield scaling is a widespread challenge in the oil and gas sector, leading to substantial financial losses owing to property damage and decreased production. This issue, which ranks among the top production efficiency impediments, is pervasive worldwide, primarily affecting water-related oil extraction operations alongside corrosion and hydrate formation. Scaling can accumulate on various surfaces, particularly around the wellbore, restricting fluid flow and obstructing production equipment from pore throats to processing facilities. The industry typically encounters four major types of scale: calcium carbonate (calcite and aragonite), calcium sulfate salts (such as gypsum), strontium sulfate (celestite), and barium sulfate (barite).

Scale inhibitors (SIs) are the primary solutions used to prevent crystal growth and scale formation, with concentrations ranging from 1 to 500 ppm, to maintain effectiveness. Water-soluble organic SIs are the most common in industry, where it is critical to sustain concentrations above the

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minimum inhibitory concentration (MIC) to ensure efficacy. SIs are commonly applied through downhole squeeze treatments or continuous injections at the wellhead [1]. In squeeze treatments, inhibitors are injected into the subsurface formation, often using seawater to extend their reach, after which the inhibitors are absorbed by the formation rock near the wellbore and released gradually into the produced water, effectively controlling their scale during production.

Phosphoric acids, particularly those with C-$PO(OH)_2$ moieties, are an important class of organophosphorus compounds [2, 3]. These compounds and their derivatives, known as phosphates, are widely used across agricultural, chemical, and pharmaceutical industries [4, 5]. Organophosphonic acids and their salts are essential SIs in the oil industry [6–8]. These inhibitors vary in structure from small non-polymeric molecules with limited phosphonate groups to more complex polymeric compounds with a higher phosphonate concentration. In many cases, phosphonate groups are bonded to amino methylene phosphonate structures, where the amine acts as a Lewis base ligand, further enhancing scale inhibition.

The phosphonate groups in SIs serve a functional role in indicating the inhibitor concentration in the produced water. Their presence aids in determining the timing of the re-squeeze operations to ensure continuous scale inhibition. Phosphonate groups are particularly advantageous in the oilfield industry for mitigating scale issues owing to their durability and biodegradability.

MATERIALS AND METHODS

Previous studies have shown that conjugating amino phosphonates with methylene phosphonates significantly improves the effectiveness of SIs, although biodegradability remains a challenge, as observed in BP-7 and BP-9, which exhibit low biodegradation rates. This study focuses on the synthesis of a new scale inhibitor using guanidine as the starting material. Research suggests that phosphonate groups can effectively replace hydrogen atoms bound to the nitrogen in guanidine, enhancing the ability of the compound to prevent scaling. Furthermore, the inhibitory efficiency is expected to increase with an increase in the number of attached phosphonate groups. Consequently, guanidine, with five hydrogen atoms, provides a promising molecular framework for this investigation, as shown in Figure 1

Guanidine is a hygroscopic organic compound with the chemical formula CH_5N_3 and a molecular weight of approximately 59.07 g/mol. It is a colorless solid, soluble in polar solvents, and possesses strong basic properties. Guanidine is not only found as an independent molecule but also as a constituent of larger organic compounds, including arginine side chains, and occurs naturally in various sources such as urine, turnip juice, mushrooms, rice husks, and muscle tissue. Its melting point is approximately 50°C, and upon heating to 160°C, it is converted to melamine and ammonia. To develop an effective and environmentally friendly inhibitor, certain criteria must be met [9]. These include achieving a minimal effective concentration of 1–100 ppm, with an optimal range of 1–5 ppm, maintaining thermal stability at temperatures up to 100°C and between 130 and 170°C, exhibiting at least 60% biodegradability within 28 days, having a pH compatibility range of 4–9, compatibility with calcium, and cost-efficiency in production and application.

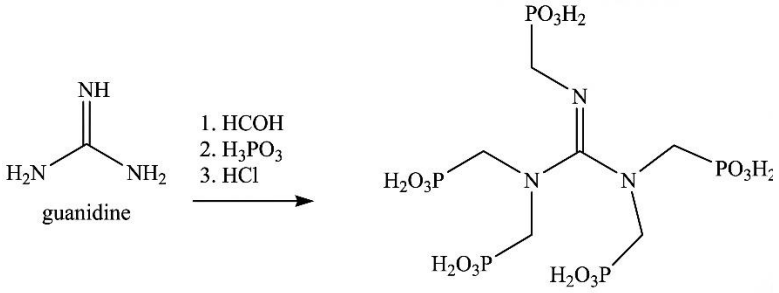

Figure 1. (Dihydroxyphosphaneyl)methyl)-1.3.3-tris(phosphonomethyl)guanidino)methyl) phosphonic acid.

In addition to their potent therapeutic effects, guanidine molecules belong to an indisputable family of chemicals distinguished by their ability to acquire a positive charge through protonation in physiological settings [10]. Guanidine groups play a crucial role in the development and identification of antibiotics because of their characteristics, which enable them to establish hydrogen bonds or electrostatic interactions with possible bacterial targets [11]. A common example of this type of chemical is the antibiotic streptomycin, which has a guanidine group. Guanidine-functionalized groups can be added to polycarbonates as adjuvants, greatly increasing the antibacterial activity of several antibiotics [12].

Guanidine can be isolated from several medications in which it is used; thus, purchasing it is unnecessary. By repurposing old medications, rather than discarding them, this approach guarantees environmental preservation. The ketophane molecule was extracted from medicine and reused in chemical reactions in a scientific investigation [13].

SCALE INHIBITORS

A scale inhibitor is a chemical agent designed to prevent scale formation by reducing the rate of fouling scale development [14]. These inhibitors typically comprise water-soluble compounds that effectively impede the nucleation and crystal growth of inorganic scales by disrupting normal crystal growth patterns, thereby preventing the formation of larger crystals. Certain polymers have been identified as effective inhibitors and dispersants of nucleation. The key characteristics of an effective scale inhibitor include the following.

- *Efficiency:* It must effectively inhibit scale formation, regardless of the mechanisms involved.
- *Stability:* The inhibitor should remain stable at elevated temperatures in oil production environments.
- *Compatibility:* It must not interfere with other oilfield chemicals or change in response to their presence while integrating smoothly with chemical injection systems.

To ensure comprehensive protection against scale formation, maintaining a MIC is crucial. Concentrations below this threshold significantly increase the risk of scale formation. In the oilfield, common scales like carbonates and sulfates contain divalent anions (CO_3^{2-} and SO_4^{2-}) alongside group II metal cations. The scale inhibitor must interact with these anions or cations to effectively anchor to the scale surface and prevent competing molecules from binding to the crystal lattice.

Various organic molecules with anionic groups can interact favorably with group II cations on the scale crystal surfaces. The most prevalent anionic groups are phosphates, phosphonates, phosphinates, carboxylates, and sulfonates. Molecules containing multiple anionic groups or mixtures of these groups have been effective as SIs, particularly in their anionic dissociation form. Common classes of SIs include polyphosphates, phosphates, small non-polymeric phosphonates, aminophosphonates, polyphosphonates, polycarboxylates, phosphino polymers, and polysulfonates.

Phosphate esters are recognized as environmentally friendly SIs, although they may not be the most efficient. Their solubility in water or oil can be adjusted by modifying the alkyl tail length of the alcohol used in their syntheses. Additionally, compact non-polymeric SIs, characterized by limited phosphonate groups, are effective because of their amino methylene phosphonate groups, which can form bonds with divalent cations, enhancing the chelation effect and stabilizing the complexes [9].

Research has highlighted the importance of bisphosphonates (BP), which have been used for decades to treat bone disorders because of their targeting properties [15–17]. These compounds are resistant analogs of pyrophosphates that inhibit mineralization in bones [18,19]. However, phosphonate-based SIs often face criticism for their limited biodegradability, which raises environmental concerns, as both the water treatment industry and oil companies focus on the implications of these compounds [1, 20].

The biodegradability of SIs is categorized by their environmental impact, with "Green" or "Yellow" classifications indicating a minimum biodegradation rate of 20% within 28 days, according to the Norwegian national environmental agency [21]. Conversely, "Red" category chemicals exhibit less than 20% biodegradation. Presently, environmentally friendly SIs lack stability at high reservoir temperatures exceeding 140°C over extended periods, resulting in a scarcity of effective options for near-wellbore regions. Scale squeeze treatments are utilized to address this issue by injecting inhibitors into formations where they adhere to reservoir rocks and are gradually released, necessitating thermal stability for long-term effectiveness (Figure 2).

CHEMICALS AND REAGENTS

Guanidine used in the experiments was obtained from India. It was imported and distributed by the Chang Chun Chan Enterprise Sdn Bhd Company. Phosphorous acid (H_3PO_2), hydrochloric acid (HCl), and formaldehyde (CH_2O) were purchased from Riedel-de Haën. It was purchased from the University of Benghazi.

The reagents used were sodium chloride (NaCl) and calcium chloride ($CaCl_2 \cdot 2H_2O$) supplied by Amazon, sodium sulfate (Na_2SO_4), and sodium bicarbonate ($NaHCO_3$) from India.

INSTRUMENTATION AND APPARATUS

- A magmatic stirrer (FAIC Instrument Laboratory Equipment Supplier, Italy) and a precision balance 440–43 (Shon GmbH, GERMANY) were utilized as mathematical instruments in the experiment.
- A Hamilton micro syringe was used in this preliminary study for samples.
- An OEM digital pH measuring device for a water quality tester was used.
- Used glassware from Witeg Labortechnik GmbH (Germany).
- All previous appliances, tools, and glass were purchased from the Modern Science Company, Benghazi.
- ^1H and C^{13} NMR analyses were performed, and the spectra were recorded using equipment at the National Research Center, Central Laboratory Services, Magnetic Resonance Laboratory, Cairo, Egypt.
- Fourier Transform Infrared (FT-IR) spectroscopy was used to elucidate the molecular structure and functional group composition of the synthesized inhibitor. Analysis was conducted using an Agilent Technologies Cary 630 FT-IR spectrometer at the Libyan Advanced Center for Chemical Analysis in Tajoura, Tripoli.
- A JASCO instrument. The samples were scanned at a resolution of 4 cm^{-1} over the wavenumber range of 550–4000 cm^{-1} at the National Research Center, Central Laboratory Services, Magnetic Resonance Laboratory, Cairo, Egypt.

Experiments and tests were conducted in the laboratory of the Libyan Academy for Postgraduate Studies in Benghazi within the timeframe specified by the academy, which lasted approximately five months. The project began in February 2023, and trial and performance evaluations were conducted in July 2023. This phase marks a crucial step in the experiment before transferring it to external laboratories for further analysis, as outlined in this research.

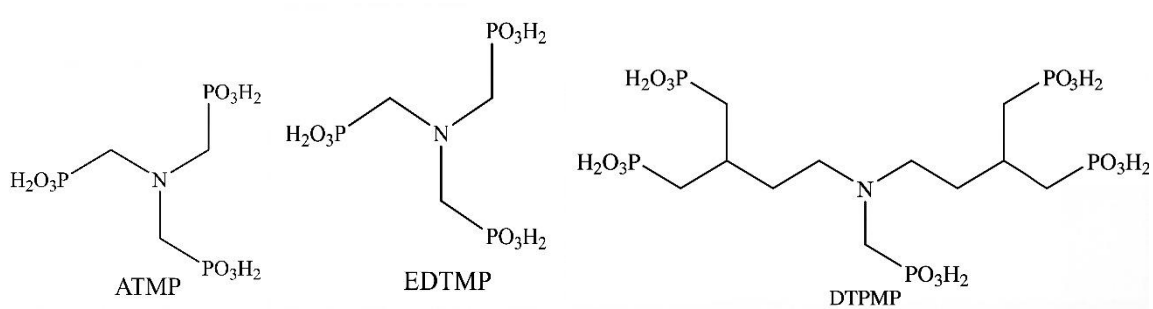

Figure 2. Common oilfield SIs containing phosphonate groups [9].

SYNTHESES OF SCALE INHIBITOR

As illustrated in Figure, guanidine (5 g, 84.6 mmol) was added to a two-neck flask. Phosphorous acid (34.68 g, 423 mmol) was added to the flask while stirring HCl (15. 41 g, 423 mmol) was added dropwise to the mixture and heated in a water bath for 10 min.

At 100°C, formaldehyde (12.7 g, 423 mmol) was added dropwise to the solution with continuous stirring, as described in [22].

The reaction mixture was then incubated overnight at 100°C. The mixture was then filtered, and The Solvent in the liquid phase was removed under reduced pressure, as shown in Figure 3.

RESULTS AND DISCUSSIONS

Results Scale Test Observations

To summarize the findings from the scale tests at various temperatures and concentrations, Tables 1–6 provide detailed observations.

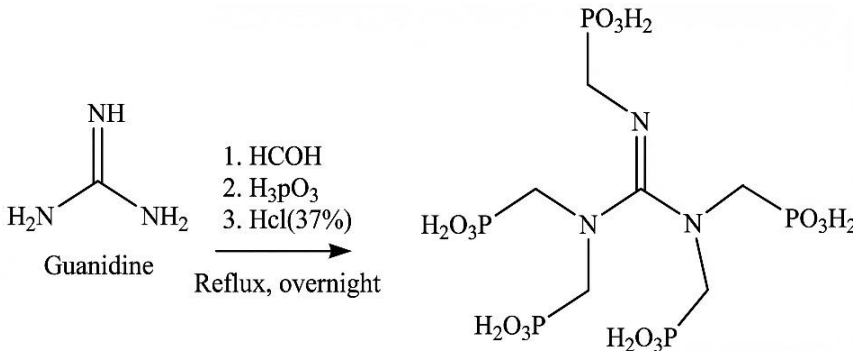

Figure 3. Phosphonation of guanidine diamine with phosphorous acid. formaldehyde and HCl [18].

Table 1. Scale test observations at (40°C) $CaCO_3$.

Product	Dose (ppm)	0 hr.	1 hr.	2 hr.	4 hr.	8 hr.	24 hr.
Plank	0	*C, B	*Sl haze	Sl haze	*Sl CO_3 ppt	*CO_3 ppt	*hvy CO_3 ppt
	5	C, B	C, B	C, B	Sl haze	*Sl CO_3	*CO_3 ppt
	10	C, B	C, B	C, B	Sl haze	Sl CO_3	CO_3 ppt
	15	C, B	C, B	C, B	Sl haze	Sl haze	Sl CO_3 ppt
	20	C, B	C, B	C, B	Sl haze	Sl haze	Sl CO_3 ppt
	30	C, B	C, B	C, B	C, B	C, B	Sl haze

*Note: *C and B = Clear and bright; *Sl haze = Slight haze; *Sl CO_3 ppt = Slight carbonate precipitate; *CO_3 ppt = Moderate carbonate precipitation; *SO_4 ppt = Moderate sulfate precipitate; *Sl SO_4 ppt = Slight sulfate precipitate; *hvy CO_3 ppt = Heavy carbonate precipitate; * Hvy SO_4 ppt = Heavy sulfate precipitate.*

Table 2. Scale test observations at (40°C) $CaSO_4$.

Product	Dose (ppm)	0 hr.	1 hr.	2 hr.	4 hr.	8 hr.	24 hr.
Plank	0	C, B	C, B	C, B	*Sl SO_4 ppt	*SO_4 ppt	*hvy SO_4 ppt
	5	C, B	C, B	C, B	C, B	*Sl SO_4 ppt	*SO_4 ppt
	10	C, B	C, B	C, B	C, B	Sl SO_4 ppt	SO_4 ppt
	15	C, B	C, B	C, B	C, B	Sl SO_4 ppt	SO_4 ppt
	20	C, B	C, B	C, B	C, B	C, B	Sl haze
	30	C, B	C, B	C, B	C, B	C, B	C, B

*Note: *C and B = Clear and bright; *Sl haze = Slight haze; *Sl CO_3 ppt = Slight carbonate precipitate; *CO_3 ppt = Moderate carbonate precipitation; *SO_4 ppt = Moderate sulfate precipitate; *Sl SO_4 ppt = Slight sulfate precipitate; *hvy CO_3 ppt = Heavy carbonate precipitate; * Hvy SO_4 ppt = Heavy sulfate precipitate.*

Table 3. Scale test observations at (60°C) $CaCO_3$.

Product	Dose (ppm)	0 hr.	1 hr.	2 hr.	4 hr.	8 hr.	24 hr.
Plank	0	C, B	Sl haze	Sl haze	Sl CO_3 ppt	CO_3 ppt	hvy CO_3 ppt
	5	C, B	C, B	C, B	Sl haze	Sl CO_3	CO_3 ppt
	10	C, B	C, B	C, B	Sl haze	Sl CO_3	CO_3 ppt
	15	C, B	C, B	C, B	Sl haze	Sl haze	Sl CO_3 ppt
	20	C, B	C, B	C, B	Sl haze	Sl haze	Sl CO_3 ppt
	30	C, B	C, B	C, B	C, B	C, B	Sl haze

*Note: *C and B = Clear and bright; *Sl haze = Slight haze; *Sl CO_3 ppt = Slight carbonate precipitate; *CO_3 ppt = Moderate carbonate precipitation; *SO_4 ppt = Moderate sulfate precipitate; *Sl SO_4 ppt = Slight sulfate precipitate; *hvy CO_3 ppt = Heavy carbonate precipitate; * Hvy SO_4 ppt = Heavy sulfate precipitate.*

Table 4. Scale test observations at (60°C) $CaSO_4$.

Product	Dose (ppm)	0 hr.	1 hr.	2 hr.	4 hr.	8 hr.	24 hr.
Plank	0	C, B	C, B	C, B	Sl SO_4 ppt	SO_4 ppt	hvy SO_4 ppt
	5	C, B	C, B	C, B	C, B	Sl SO_4	SO_4 ppt
	10	C, B	C, B	C, B	C, B	Sl SO_4	SO_4 ppt
	15	C, B	C, B	C, B	C, B	Sl SO_4	SO_4 ppt
	20	C, B	C, B	C, B	C, B	C, B	Sl haze
	30	C, B	C, B	C, B	C, B	C, B	C, B

*Note: *C and B = Clear and bright; *Sl haze = Slight haze; *Sl CO_3 ppt = Slight carbonate precipitate; *CO_3 ppt = Moderate carbonate precipitation; *SO_4 ppt = Moderate sulfate precipitate; *Sl SO_4 ppt = Slight sulfate precipitate; *hvy CO_3 ppt = Heavy carbonate precipitate; * Hvy SO_4 ppt = Heavy sulfate precipitate.*

Table 5. Scale test observations at (70°C) $CaCO_3$.

Product	Dose (ppm)	0 hr.	1 hr.	2 hr.	4 hr.	8 hr.	24 hr.
Plank	0	C, B	Sl haze	Sl haze	Sl CO_3 ppt	CO_3 ppt	hvy CO_3 ppt
	5	C, B	C, B	C, B	Sl haze	Sl CO_3	CO_3 ppt
	10	C, B	C, B	C, B	Sl haze	Sl CO_3	CO_3 ppt
	15	C, B	C, B	C, B	Sl haze	Sl haze	Sl CO_3 ppt
	20	C, B	C, B	C, B	Sl haze	Sl haze	Sl CO_3 ppt
	30	C, B	C, B	C, B	C, B	C, B	Sl haze

*Note: *C and B = Clear and bright; *Sl haze = Slight haze; *Sl CO_3 ppt = Slight carbonate precipitate; *CO_3 ppt = Moderate carbonate precipitation; *SO_4 ppt = Moderate sulfate precipitate; *Sl SO_4 ppt = Slight sulfate precipitate; *hvy CO_3 ppt = Heavy carbonate precipitate; * Hvy SO_4 ppt = Heavy sulfate precipitate.*

Table 6. Scale test observations at (70°C) $CaSO_4$.

Product	Dose (ppm)	0 hr.	1 hr.	2 hr.	4 hr.	8 hr.	24 hr.
Plank	0	C, B	C, B	C, B	Sl SO_4 ppt	SO_4 ppt	hvy SO_4 ppt
	5	C, B	C, B	C, B	C, B	Sl SO_4	SO_4 ppt
	10	C, B	C, B	C, B	C, B	Sl SO_4	SO_4 ppt
	15	C, B	C, B	C, B	C, B	Sl SO_4	SO_4 ppt
	20	C, B	C, B	C, B	C, B	C, B	Sl haze
	30	C, B	C, B	C, B	C, B	C, B	C, B

*Note: *C and B = Clear and bright; *Sl haze = Slight haze; *Sl CO_3 ppt = Slight carbonate precipitate; *CO_3 ppt = Moderate carbonate precipitation; *SO_4 ppt = Moderate sulfate precipitate; *Sl SO_4 ppt = Slight sulfate precipitate; *hvy CO_3 ppt = Heavy carbonate precipitate; * Hvy SO_4 ppt = Heavy sulfate precipitate.*

EFFECT OF THE INHIBITOR ON CRYSTAL MODIFICATION: RESULTS

The presence of PBTC (phosphonobutane-1,2,4-tricarboxylic acid) in solutions containing $CaCO_3$ leads to changes in crystal morphology, as shown in Figure 4, resulting in rounded corners and aggregate formation. However, the number of crystals formed in the presence of PBTC is

significantly lower. The aggregation of $CaCO_3$ crystals can also be observed with certain additives. FT-IR analysis of PBTC-treated $CaCO_3$ crystals indicated the presence of bands associated with the -PO_3 group, suggesting inhibitor incorporation within the $CaCO_3$ lattice or at the crystal edges, as shown in Figure 5.

RESULTS OF THE TITRATION TECHNIQUE

The results of the scale test for $CaCO_3$ are summarized in Table 7.

The results of the scale test for $CaSO_4$ are summarized in Table 8.

Figure 4. Scale test observation for $CaCO_3$ at 70°C.

Figure 5. Scale test observation for $CaSO_4$ at 70°C.

Table 7. Scale test $CaCO_3$.

Product	Dose PPM	Titration reading	mg/L Ca sample	mg/L Ca sample - mg/L Ca blank	Percentage inhibitor %
Blank	0	3.4	272	0.00	0.00
	5	3.8	304	32	11
	10	4.6	368	96	35
	20	5.8	464	192	70
	30	6.8	544	272	100

Table 8. Scale test $CaSO_4$.

Product	Dose PPM	Titration reading	mg/L Ca sample	mg/L Ca sample - mg/L Ca blank	Percentage inhibitor %
Blank	0	4.2	457.2	0.00	0.00
	5	4.8	522.77	65.57	16.3
	10	5.9	642.5	185.3	46
	20	7.1	773.27	316.07	78.4
	30	7.9	860.4	403.2	100

RESULTS OF COMPATIBILITY WITH CALCIUM TEST

In Figure 6, the results of the compatibility test with calcium ions. The bottles in the Figure show the test after 24 h; all bottles had clear solutions.

RESULTS OF HIGH-PRESSURE DYNAMIC TUBE BLOCKAGE TEST

Tables 9 and 10 summarize the chemical composition and preparation details of the sulfate and carbonate brines used in the scale rig experiments, presenting ionic concentrations, corresponding salt masses for different solution volumes, and calculated chloride levels designed to simulate representative formation water chemistries under controlled laboratory conditions.

RESULTS OF SCALE INHIBITOR SEAWATER BIODEGRADABILITY TEST

Oxygen consumption data were recorded over a 28-day period, and all flasks were incubated in the dark at 20°C. At the end of the 28-day period, data were collected, and the results were obtained. The theoretical oxygen demand (ThOD) for each scale inhibitor was calculated in accordance with OECD 306 guidelines, accounting for complete nitrification. Background respiration values (BOD values representing seawater's inherent respiration) were subtracted from the BOD of each test compound prior to determining the biodegradability percentage, as per the OECD.

Figure 6. Compactivity test in 100 ppm Ca^{2+} and 3% NaCl in 2 mL.

Table 9. The composition of sulfate brine 1 and brine 2 used in the scale rig.

Brine 1					
Ion	***ppm***		***g/L***	***g/3L***	***g/5L***
Na	19510	NaCl	38.640	115.93	193.2
Ca	2040	$CaCl_2 * 2H_2O$	5.3100	15.930	26.55
Mg	530	$MgCl_2 * 6H_2O$	13.660	40.980	68.30
K	1090	KCl	1.9200	5.7600	9.600
Ba	570	$BaCl_2 * 2H_2O$	0.5100	1.5300	2.550
Sr	290	$SrCl_2 * 6H_2O$	0.4400	1.3200	2.200
Cl		Actual Cl ppm	31166.40		
Brine 2					
Ion	***ppm***		***g/L***	***g/3L***	***g/5L***
Na	19510	NaCl	35.04	105.12	175.20
SO_4	2960	Na_2SO_4 anhydrous	4.380	13.149	21.900
		Actual Cl ppm	30086.47		

The spectra clearly showed a peak at a wavelength of approximately 3000–3300 nm, which corresponds to the characteristic wavelength of the CH_2-CH group added to guanidine, which does not contain the CH_2 group in its structure (Figure 7).

The presence of a signal at 2117 indicates the presence of a non-exchangeable C=N bond associated with the sp3 nitrogen atom of the NH group, as previously explained. This further supports the facile substitution of hydrogen atoms on the sp3 nitrogen atom compared to the difficulty in substituting hydrogen atoms on the sp3 nitrogen atom in guanidine.

RESULTS OF NMR SPECTROSCOPY

An analysis was performed to confirm the formation of the target compound and elucidate its detailed structure. This involves employing two powerful spectroscopic techniques: Carbon-13 nuclear magnetic resonance C^{13}NMR and proton nuclear magnetic resonance (^{1}H NMR).

Table 10. The composition of carbonate brine 1 and brine 2 used in the scale rig.

Brine 1					
Ion	***ppm***		***g/L***	***g/3L***	***g/5L***
Na	19510	NaCl	49.59	148.77	247.97
Ca	2040	$CaCl_2$ * $2H_2O$	7.48	22.45	37.42
Mg	530	$MgCl_2$ * 6H2O	4.43	13.30	22.16
K	1090	KCl	2.0781	6.23	10.39
Ba	570	$BaCl_2$ * $2H_2O$	1.0138	3.04	5.07
Sr	290	$SrCl_2$ * $6H_2O$	0.8824	2.65	4.4122
Cl	0	Actual Cl ppm	35633.19		
Brine 2					
Ion	***ppm***		***g/L***	***g/3L***	***g/5L***
Na	19510	NaCl	49.59	148.77	247.95
SO_4	2000	Na_2SO_4 anhydrous	2.76	8.26	13.76
		Actual Cl ppm			

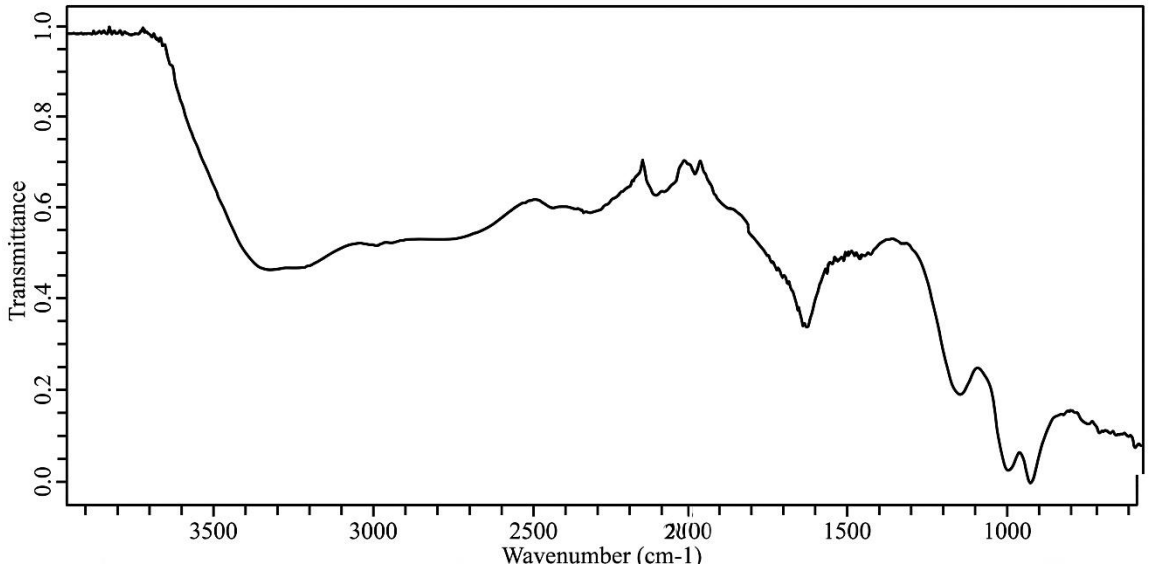

Figure 7. FT-IR Spectra for guanidine.

FT-IR SPECTROSCOPY

These techniques provide complementary information about molecules. C^{13}NMR spectroscopy probes the chemical environment of the carbon atoms within the molecule, revealing the number of carbons and their connectivity. ^{1}H NMR spectroscopy, on the other hand, focuses on hydrogen atoms, providing details about their chemical shifts and coupling patterns.

By analyzing both the C^{13}NMR and ^{1}H NMR spectra, chemists can gain a comprehensive understanding of the structure of the target compound, including the presence of functional groups and the arrangement of atoms within the molecule, as shown in Figure 8.

The strategic substitution of five hydrogen atoms with specific functional groups aims to produce a target compound with tailored electronic properties and enhanced reactivity (Figure 9).

However, the analysis revealed incomplete substitution, with only three hydrogen atoms replaced by phosphonate groups (H_3PO_3), likely due to the combined effects of strong SP^2N bond strength, insufficient reaction time, and steric hindrance in the resulting molecule.

- The substitution of hydrogen on the SP^2N atom is difficult because of the strength of the SP^2N bond.
- The steric hindrance of the resulting compound prevented the substitution of the fourth hydrogen atom on the SP^3 nitrogen atom. The resulting compounds are shown in the following structure in Figure 10.

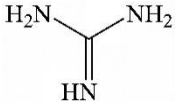

Figure 8. Structure of guanidine.

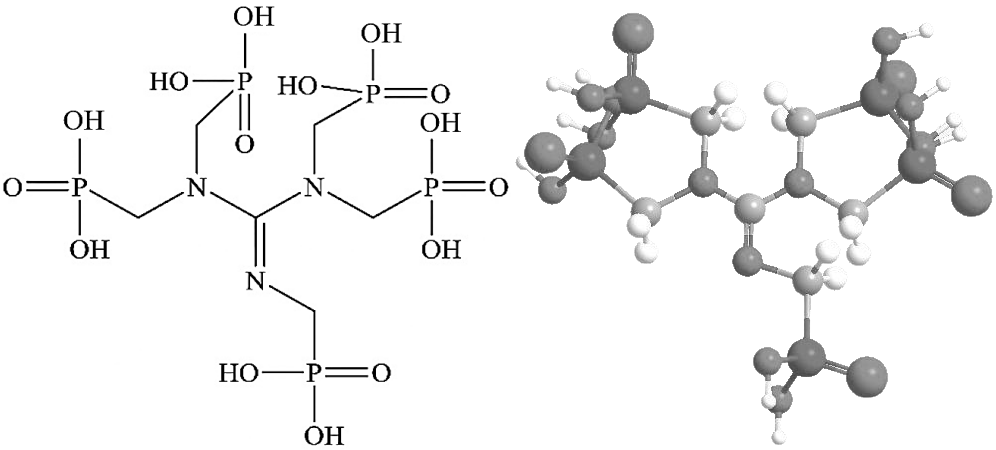

Figure 9. 2D and 3D-{bis [bis (phosphonomethyl) amino] methylene} phosphoramidic acid.

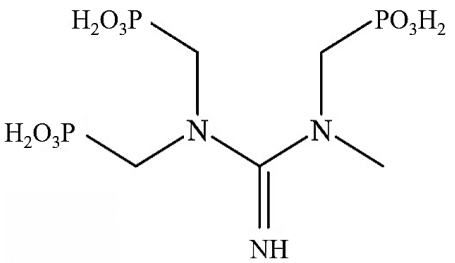

Figure 10. [(N-methyl-N-(phosphonomethyl) carbamimidyl) azanediyl) bis (methylene) bis (phosphonic acid)].

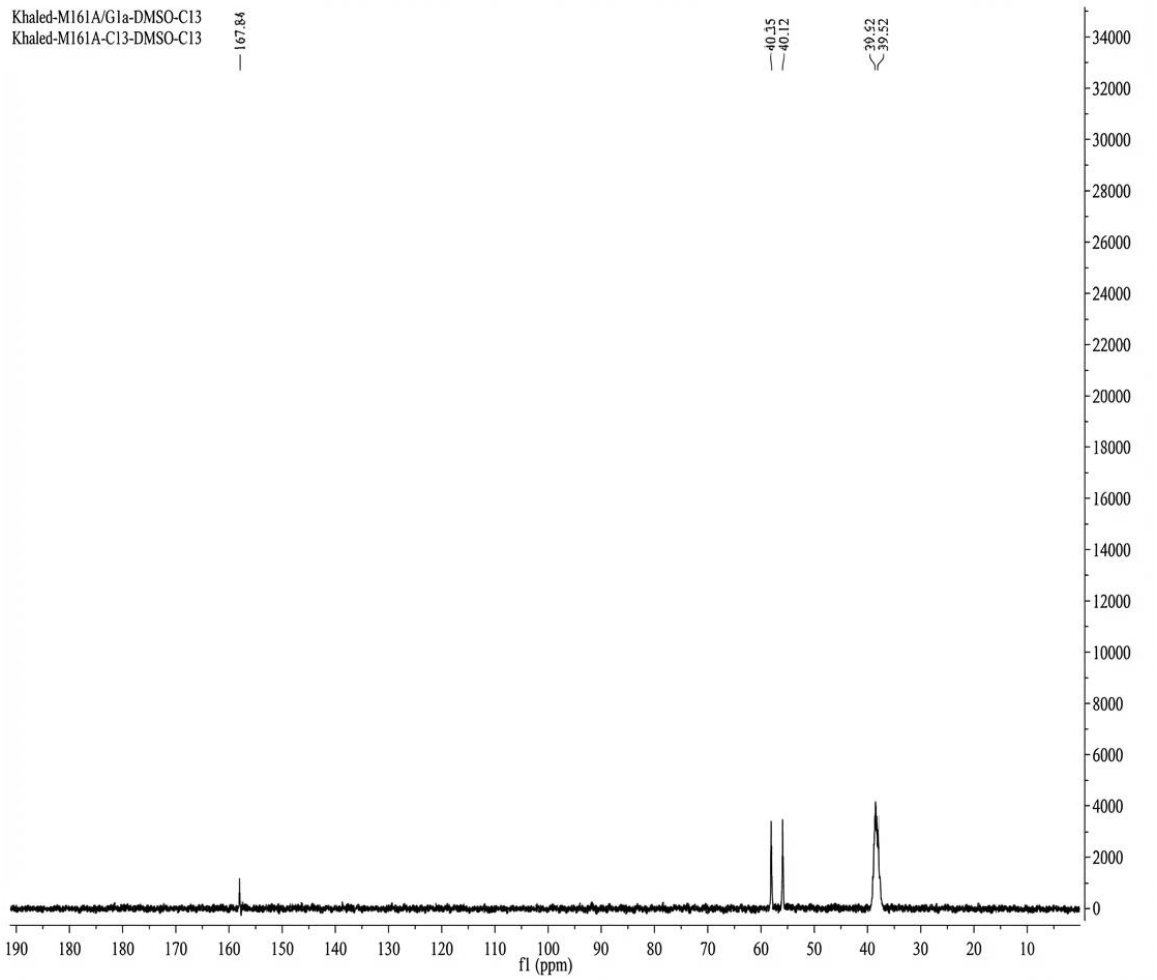

Figure 11. C^{13} NMR spectrum for guanidine.

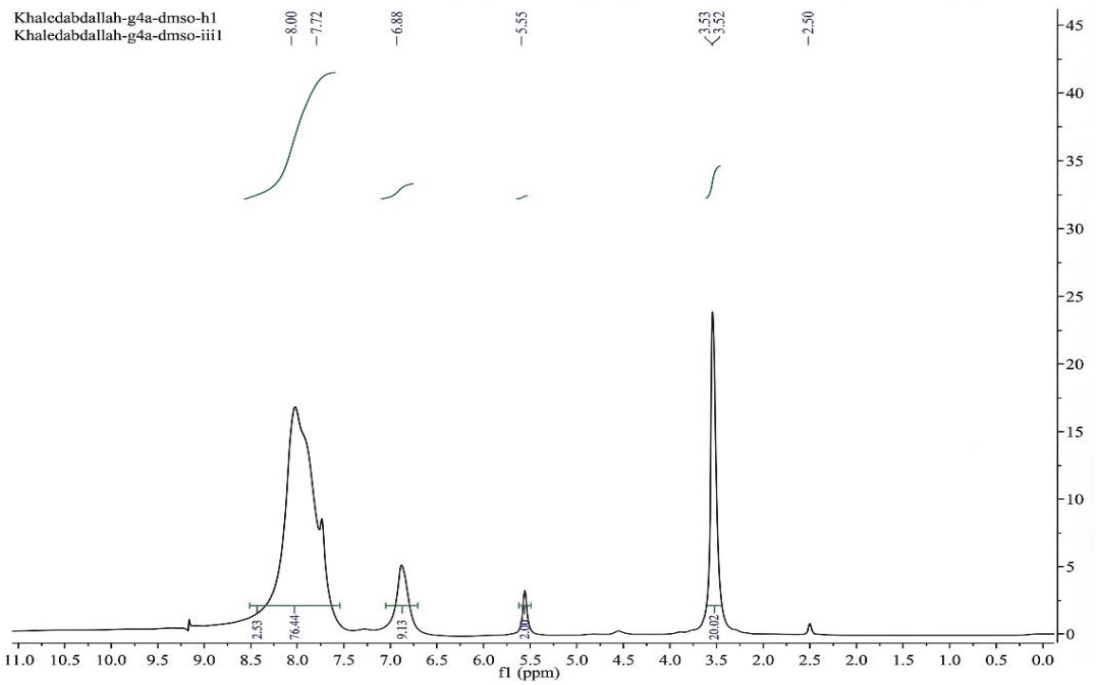

Figure 12. ^{1}H NMR spectroscopy for guanidine.

Table 11. The 13C NMR chemical shift for guanidine

Carbon signal	Chemical shift (δ, ppm)	Carbon
C-	38.19 ppm	CH_3
C-	38.42 ppm	CH_3
C-	55.9 ppm	CH_2
C-	58.01 ppm	CH_2
C-	58.01 ppm	CH_2
C-	157.84 ppm	C

Table 12. The 1H NMR chemical shift for guanidine

Functional group	Chemical shift (δ, ppm)
OH	5.5 ppm
OH	5.5 ppm
OH	5.5 ppm
OH	5.5 ppm
OH	5.5 ppm
OH	5.5 ppm
NH	7.72 ppm
CH_2	3.5 ppm
CH_2	3.5 ppm
CH_2	3.5 ppm

RESULTS OF NMR C^{13} SPECTROSCOPY

C^{13} NMR spectroscopy will be used to analyze the carbon framework of the synthesized molecule in Table 11. This technique differentiates between various carbon types (methyl, carbonyl, etc.) based on their environment, aiding in confirming the success of the functional group substitution, as shown in Figure 11.

RESULTS OF ^{1}H NMR SPECTROSCOPY

^{1}H NMR spectroscopy was employed to analyze the chemical environment of the hydrogen atoms in the synthesized molecule Table 12 This technique confirms the success of functional group substitution by identifying the number and location of hydrogen atoms, as shown in Figure 12.

RESULTS OF MASS SPECTROSCOPY (MS)

The compound underwent fragmentation, producing a molecular ion peak (M+) at M/2 = 372, with a relative intensity of 171.1 on the spectrum (Figure 13). This peak represents the molecular weight of the studied compound.

The spectrum also shows a base peak (Beas peak) at M/2 = 81, corresponding to the molecular weight of PO_3H_2 (phosphoric acid monohydrate) with a significant relative intensity of 289506.

A prominent peak at the top of the spectrum with M/2 = 64 and an exceptionally high relative intensity of 639943.1 could be attributed to the Beas peak of PO_2 (phosphorus dioxide).

CONCLUSION

In this study, we synthesized and evaluated a chelating compound that inhibits scale formation in liquid transportation pipes.

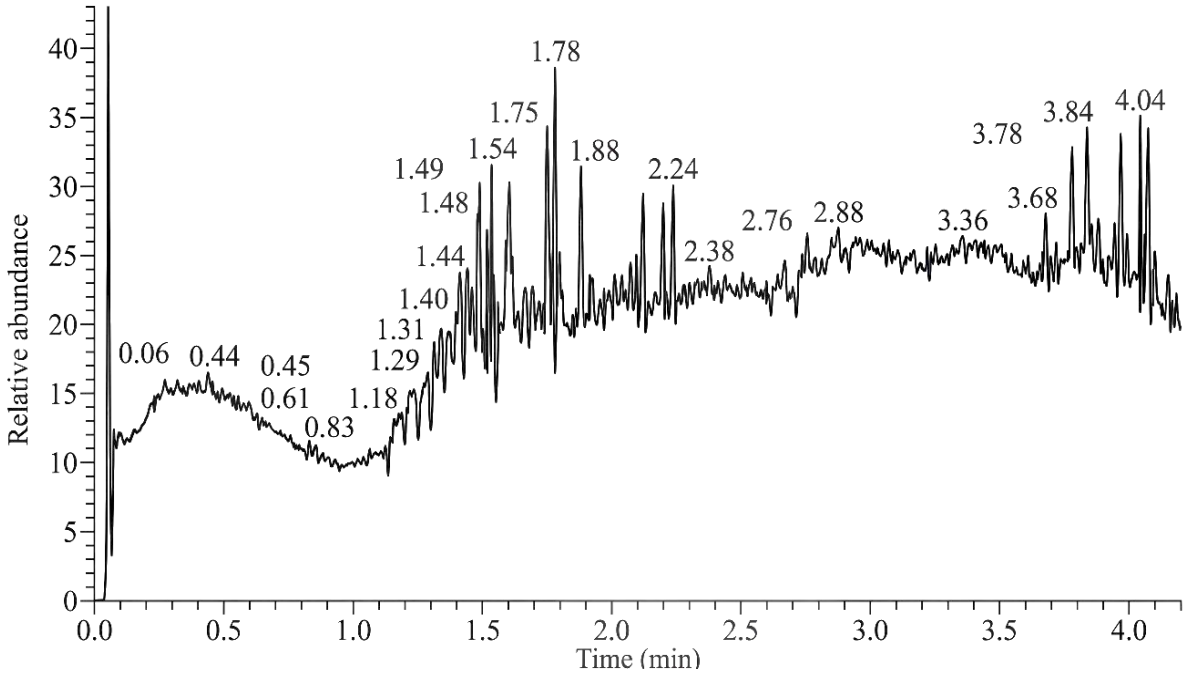

Figure 13. Mass spectroscopy of guanidine.

By substituting hydrogen atoms in guanidine with phosphonate groups, we developed compounds with strong chelation properties to prevent scale build-up. Guanidine was modified with three phosphonate groups owing to steric and electronic factors, achieving a stable inhibition performance. The structure of the substituted guanidine was confirmed using multiple analytical techniques, including NMR, FT-IR, and mass spectroscopy

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