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EVALUATION OF POLYACRYLIC-POLYASPARTIC ACID BLENDS AS ECO-FRIENDLY SCALE INHIBITORS IN OILFIELD PRODUCED WATER

E. O. OHIMOR; & E. A. AGHOGHOPHIA

Department of Chemical Engineering, Federal University of Petroleum Resources, Effurun, Delta State, Nigeria.

Corresponding Author: aghoghophia.agem@gmail.com

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ABSTRACT

This study evaluates the performance of blends of commercial polyacrylic acid (PAA) and polyaspartic acid (PASA) as alternative scale inhibitors to phosphorus-base (toxic scale inhibitors), in oilfield produced water system. Produced water from an oilfield in Niger Delta, Nigeria, was tested using an Optimal (Combined) Design Methodology and static jar tests to measure scale inhibition. The effects of PAA and PASA dosage, temperature, and pH on scale

INTRODUCTION

The oil and gas industry faces persistent challenges related to fluid flow during production, notably gas hydrates, emulsions, corrosion, wax deposition, and scaling are collectively known as flow assurance issues. Among these, gas hydrates, emulsions, corrosion, and scaling are strongly associated with water presence in hydrocarbon systems (Kelland, 2014; Sangwai and Dandekar, 2022).

Produced water, a major byproduct of oil and gas extraction, contains various dissolved salts, heavy metals, and organic compounds. One critical issue arising from its handling is the precipitation of inorganic scales, particularly calcium, barium, and strontium carbonates and sulfates, which can deposit throughout upstream, midstream, and downstream infrastructure. Scale accumulation can severely impede production by blocking pipelines, pumps, tubing, valves, and reservoir pore spaces—leading to equipment damage, reduced flow efficiency, unplanned shutdowns, and increased operational costs (Kumar, 2023; Hendraningrat *et al.*, 2024).

Flow stations, where multiphase fluids are received, separated, and conditioned, are especially susceptible to

inhibition efficiency were examined. Statistical analysis identified a quadratic model as the best fit for the data, with an R^2 of 0.9375 and an adjusted R^2 of 0.7969. Results showed significant interaction among the studied parameters affecting scale inhibition. Within the experimental range, the maximum inhibition efficiency of 97.42% was achieved at 50°C, pH 8, with 0.055 ppm each of PAA and PASA. Using the Desirability Methodology, the model's predictions were validated and found reliable, particularly at temperatures below 50°C and pH slightly above 7. For instance, at 38.45°C, pH 7.05, and dosages of 0.028 ppm PAA and 0.082 ppm PASA, the model predicted 99.79% inhibition, with an actual efficiency of 100%. At higher temperatures and pH (69.11°C, pH 9.93), the model overestimated inhibition efficiency, predicting 101.21% while the actual was 74.06%. Another test at 80.44°C and pH 7.34 showed close agreement between predicted (99.92%) and actual (97.85%) efficiencies. These results indicate that blending PAA and PASA offers a synergistic effect, effectively controlling scale formation in oilfield equipment under the tested conditions. As recommendation, further analysis of scale crystal morphology using scanning electron microscopy to better understand the effect of the blends on scale structure should be done. Overall, this research supports the development of environment friendly, phosphorus-free scale inhibitors for oilfield applications.

Keywords: Flow assurance issues, Scaling, Polyacrylic acid, Polyaspartic acid, Scale inhibitor, Biodegradable inhibitors, Produced water, Oilfield applications, Statistical modelling.

scale-related issues due to their central role in processing produced water (Amiri *et al.*, 2020).

Background and Motivation

Chemical scale inhibitors are widely used to mitigate inorganic scaling. Phosphorus-based inhibitors remain common due to their effectiveness but pose environmental concerns related to toxicity and biodegradability. As regulations tighten, attention has shifted toward environmentally friendly, biodegradable alternatives.

Polyacrylic acid (PAA) and polyaspartic acid (PASA) are emerging candidates. PAA functions effectively as a dispersant and performs well in high-calcium environments, while PASA is both biodegradable and a competent chelating agent (Yan *et al.*, 2023; Zhigen *et al.*, 2023). A blend of PAA and PASA may offer synergistic benefits, combining performance efficiency with environmental compatibility. However, systematic data on their blended use in oilfield produced water applications remains

scarce. This study investigates the scale inhibition performance of PAA-PASA blends under simulated oilfield conditions.

Statement of the Problem

Polyacrylic acid (PAA) has long been applied as a reliable synthetic polymer for controlling scale in oilfield systems, but its limited biodegradability raises concern about environmental persistence (Mazumder, 2020; Kan, Dai, and Tomson, 2020). Polyaspartic acid (PASA), in contrast, is increasingly regarded as a greener alternative owing to its biodegradability, and recent studies have investigated its derivatives and modifications to improve thermal stability and inhibitory efficiency (Cheng *et al.,* 2021; Yan, Tan, and Qi, 2023; Guo *et al.,* 2020). Despite these advances, systematic evaluations of blended PAA–PASA formulations under oilfield-relevant conditions remain largely absent from the peer-reviewed literature. Published work typically examines single-component systems or chemically modified variants in simplified laboratory tests, without considering the potential synergistic effects of combining PAA and PASA (Mady, Rehman, and Kelland, 2021; Cai *et al.,* 2022; Jarrahian, Sorbie, and Boak, 2022). Notably, patent filings document composite formulations containing PAA and PASA, indicating industrial interest in such blends (CN111233174A, 2019; CN111908626A, 2020). However, the lack of rigorous academic studies evaluating the performance these blends as scale inhibitors in realistic oilfield scenarios highlights a significant knowledge gap. The present work addresses this gap by systematically assessing blended PAA–PASA inhibitors under conditions that approximate oilfield applications, thereby contributing new evidence toward environmentally sustainable and effective scale control strategies.

Aim of the Study

To evaluate the effectiveness of blended commercial polyacrylic acid (PAA) and polyaspartic acid (PASA) as scale inhibitors for oilfield produced water.

Objectives of the Study

- Collect and preserve fresh produced water samples from the South-South region of Nigeria.
- Characterize the chemical and physical properties of the samples.
- Design experiments using Optimal Combined Design via Design Expert software (v13.0.5.0, 64-bit).
- Formulate different blend ratios of PAA and PASA.
- Conduct laboratory tests to measure scale inhibition efficiency of the blends.
- Analyze results to determine key parameter interactions, develop predictive models, and perform model optimization using desirability functions.

- Experimentally validate model predictions to identify optimal blend conditions.

Relevance of the Study

Scaling in flow stations compromises fluid handling efficiency, causes equipment damage, and leads to unplanned downtime. This study evaluates PAA-PASA blends as potential green scale inhibitors tailored for oilfield environments. The research offers insights into:

- Optimal blend ratios of PAA and PASA for scale control.
- Performance under varying pH and temperature conditions representative of flow station operations.
- A biodegradable and cost-effective alternative to conventional inhibitors.

The outcomes aim to support sustainable operations in oil and gas fields, align with environmental regulations, and reduce maintenance costs.

Scope of the Study

The study focuses on laboratory evaluation of PAA-PASA blend performance in inhibiting the formation of mineral scale deposits such as $CaCO_3$, $CaSO_4$, and $BaSO_4$, scales in oilfield produced water.

Specific elements include:

- Formulation of blend ratios for inhibitor testing.
- Standard static bottle tests for assessing inhibition efficiency.
- Testing across pH (6.00–8.00), temperature (50–70°C), and inhibitor concentration (0.01–0.1 ppm) ranges.
- Statistical analysis and optimization using Design Expert software.

Limitations of the Study

This work is constrained by several factors:

1. Experiments are confined to lab-scale simulations and may not fully represent field conditions.
2. Produced water samples, while real, may not reflect the full variability of field compositions.
3. Operational parameters are limited to defined pH, temperature, and concentration ranges.
4. Long-term degradation, stability, and biofouling resistance were not assessed.
5. No cost-benefit or full environmental impact analysis is included.
6. Field-scale validation was beyond the scope of this study.
7. Potential measurement errors from titration and gravimetric methods.
8. Crystal morphology analysis (e.g., FTIR, SEM-EDS, XRD) was not conducted.

9. Variations in polymer molecular weight and chemical characterization were not considered.
10. Scaling tendency and indices of the water samples were not analyzed.

LITERATURE REVIEW

Flow stations are critical infrastructures in oil and gas production, yet they are notably prone to scaling due to alterations in the physicochemical properties of processed fluids. These changes often result from operations like waterflooding and degassing. Amiri *et al*. (2020) identify flow stations as key scaling hotspots, attributing this to sharp pressure drops and the interaction of varying aqueous chemistries from multiple wells. Increased fluid residence time, especially in separators, and poor scale control strategies further exacerbate scaling challenges.

The non-renewable nature of petroleum continues to drive exploration into unconventional reservoirs, including deepwater, shale plays, heavy oil sands, and gas hydrates (Halliburton and Oyekunle, 2013; Makwashi *et al.,* 2018; Sun *et al.,* 2018; Zarana and Ashish, 2022; Kumar, 2023). However, these sources intensify flow assurance problems, which include—but are not limited to—scaling, corrosion, gas hydrate formation, erosion, wax and asphaltene deposition, slugging, and emulsions (Makwashi *et al.,* 2018; Muhammad, 2018; Haijing, 2022; Zarana and Ashish, 2022). Notably, scaling, corrosion, gas hydrates, and emulsions are consistently linked to aqueous phases during production (Kelland, 2014; Sangwai and Dandekar, 2022).

Scale formation occurs when sparingly soluble salts precipitate from the aqueous phase and adhere to internal surfaces such as pipelines, valves, pumps, wellbores, and separators. This deposition is often driven by shifts in temperature, pressure, and ion concentration, and is classified as a type of water-related fouling (Kan and Tomson, 2012; Kan *et al.,* 2019; Ghalib *et al.,* 2023). Common scale types include calcium sulfates (e.g., anhydrite, gypsum), calcium carbonates (e.g., calcite, aragonite), and sulfides (e.g., pyrite, sphalerite) (Al-Rawahi *et al.,* 2017; Kamala *et al.,* 2018).

The scaling process follows five stages: initiation, transport, deposition, removal, and aging (Al-Rawahi *et al.,* 2017; Mohammad and Jafar, 2020; Yan *et al.,* 2023). Two dominant mechanisms drive scale formation in oilfield systems: self-scaling and incompatible water scaling (Al-Rawahi *et al.,* 2017; Al-Samhan *et al.,* 2020). Self-scaling occurs during fluid extraction when reservoir equilibrium is disrupted, promoting precipitation of naturally present ions under Le Chatelier's principle (Wayne *et al.,* 2008; Angelo and Ferrari, 2024). In contrast, incompatible water scaling arises when injection water (typically seawater) mixes with formation water rich in divalent cations like Ba^{2+}, Sr^{2+}, and Ca^{2+}, promoting scale precipitation during waterflooding (Sassan, 2016; Al-Rawahi *et al.,* 2017).

Scale management strategies fall into three main categories (Al-Rawahi *et al.,* 2017):

1. Mechanical and chemical descaling

2. Anion sequestration from injection water
3. Scale inhibition techniques

Mechanical methods (e.g., jetting, drilling, milling) are suited for hardened deposits, while chemical descaling uses acid-based solvents to dissolve scale. Despite being cost-effective, acids may lead to corrosion and toxic by-products (Nasr-El-Din *et al.*, 2000; Zhigen *et al.*, 2023).

Anion sequestration involves removing sulfate and other scale-forming ions from injection water, thereby mitigating incompatible water scaling (Mehadi *et al.*, 2020; Tzioumis *et al.*, 2023).

Among the three, chemical scale inhibition is the most widely applied due to its efficiency and cost-effectiveness. These inhibitors, often phosphonates or polymer-based, interfere with nucleation and crystal growth, thus preventing scale deposition (Li *et al.*, 2024). Physical field-based methods—such as using ultrasonic waves, electric or magnetic fields—are also employed, though their effectiveness is limited in low-hardness and low-temperature environments (Xu *et al.*, 2021; Basheer *et al.*, 2021; Yan *et al.*, 2023; Khamis *et al.*, 2024). Chemical inhibitors offer greater versatility and operational compatibility.

A summary of recent studies on chemical scale inhibitors is presented in Table 1, reflecting their compositions, and performance metrics (Mohammad and Jafar, 2020; Zhigen *et al.*, 2023).

Table 1: Summary of reported scale inhibitors, and their inhibition performance.

SCALE INHIBITORS	Dosages (mg L^{-1})	Percentage Performance	SCALANT	Reference
NATURAL ORGANIC MOLECULE SCALE INHIBITORS				
Biopheols (Olive leaf extract)	50	83	$CaCO_3$	Li *et al.*, 2016
Punicalin (Punica granutum hull extract)	100	88		Abdel-Gaber *et al.*, 2012
Sunflower oil (Helianthus annus seed extract)	50	100	$CaSO_4$	Abd-El-Khalek *et al.*, 2019
		84	$BaSO_4$	
PHOSPHORIC SCALE INHIBITORS				
2-PhosphonoButane-1,2,4-Tricarboxylic Acid (PBTC)	12	91	$CaCO_3$	Liu *et al.*, 2013.
1-HydroxylEthylidene-1,1-Diphosphonic Acid (HEDP)	4	90	$CaSO_4$	
2-PhosphonoButane-1,2,4-Tricarboxylic Acid (PBTC)	12	43	$Ca_3(PO_4)_2$	Fu *et al.*, 2011.
GREEN SCALE INHIBITORS				
PolyEpoxySuccinic Acid	12	90	$CaCO_3$	Liu *et al.*, 2012
PolyEthylene Glycol Double-ester of Maleic Anhydride/Acrylic Acid	12	89		Liu *et al.*, 2013

SCALE INHIBITORS	Dosages (mg L^{-1})	Percentage Performance	SCALANT	Reference
HydrolyzedPolyMaleic Anhydride	2	79	$CaSO_4$	Fu *et al.*, 2011
Acrylic Acid - AllyPolyEpoxyCarbonate copolymer	2	84		Xue *et al.*, 2012
PolyAspartic Acid (PASP)	10	98		Zhao *et al.*, 2016
PolyAcrylic Acid (PAA)	12	90	$Ca_3(PO_4)_2$	Fu *et al.*, 2011
PolyEpoxySuccinic Acid	12	34		
HydrolyzedPolyMaleic Anhydride	12	43		

Table 1 highlights that most research on scale inhibitors focuses on calcium-based scales, due to their prevalence in oil and gas operations (Kan and Tomson, 2012; Kelland, 2014; Hanji *et al.,* 2022). While specific inhibitors effectively target individual scale types, oilfield systems often experience mixed scale deposits, which form complex structures resistant to simple remediation methods (Kelland, 2014). Treating each scale type individually is neither economically viable nor environmentally sustainable. Phosphorus-based inhibitors pose ecological risks, while chelating agents like EDTA are costly due to their stoichiometric nature.

As a result, there is growing interest in developing cost-effective, environment friendly inhibitors that can handle mixed scale formation. Polymeric inhibitors are particularly attractive due to their structural versatility, thermal stability, broad pH tolerance, and compatibility with high calcium concentrations (Kelland, 2014). An ideal inhibitor should also be non-toxic, biodegradable, long-lasting, and chemically compatible with other additives such as corrosion inhibitors. However, no single compound meets all these criteria, prompting the use of inhibitor blends (Al-Rawahi *et al.,* 2017; Kamala *et al.,* 2018). Such blends offer advantages in terms of cost, synthesis complexity, and performance.

Recent research has increasingly emphasized the development of eco-friendly scale inhibitors as substitutes for conventional phosphonate-based formulations. Natural and polymeric inhibitors, such as polyaspartic acid and its derivatives, have been widely investigated for their biodegradability and reduced environmental footprint (Mazumder, 2020; Cai *et al.,* 2022). Plant-derived extracts and amino acid–based inhibitors have also shown promise in laboratory-scale evaluations, though their performance under harsh oilfield conditions remains inconsistent (Ghriga *et al.,* 2019; Guo *et al.,* 2020). Nanomaterial-based inhibitors, including modified silica and hybrid organic–inorganic composites, have demonstrated enhanced adsorption and extended retention, but their high production cost and uncertain long-term environmental impact limit immediate field application (Cheng *et al.,* 2021; Al-Shaketheep *et al.,* 2023). Compared with these approaches, polymer blends represent

a more practical compromise, combining proven efficacy with opportunities for greener design.

To provide a balanced perspective, it is important to critically compare eco-friendly scale inhibitor classes beyond traditional phosphonate systems. Recent efforts have focused on biodegradable polymers, plant-derived inhibitors, and nanomaterial-based formulations, each offering distinct benefits but also facing practical limitations in terms of cost, thermal stability, or field scalability. Table 2 summarizes the key strengths and shortcomings of these alternatives, highlighting that while several promising options exist, systematic evaluations of blended polymer systems such as polyacrylic acid–polyaspartic acid (PAA-PASA) remain scarce in peer-reviewed literature.

Table 2: Comparison of Eco-Friendly Scale Inhibitors

Type	Examples	Strengths	Limitations	Key References
Polymeric inhibitors (biodegradable)	Polyaspartic acid (PASP), PASP derivatives, modified PAA	Proven efficiency, biodegradable, environmentally acceptable, can be synthesized at scale	Moderate thermal stability, limited systematic field data	Mazumder (2020); Cai *et al.*, (2022); Guo et al. (2020)
Natural inhibitors	Plant extracts (e.g., henna, eucalyptus), amino acid–based compounds	Renewable, non-toxic, biodegradable	Variable performance in harsh brines, limited reproducibility, scale-up challenges	Ghriga *et al.*, (2019)
Nanomaterial-based inhibitors	Silica nanoparticles modified with PASP or phosphonate-free groups; hybrid organic–inorganic composites	High adsorption capacity, prolonged retention, potential synergistic effects	High production costs, uncertain long-term environmental risks, limited field trials	Cheng *et al.*, (2021); Al-Shaketheep *et al.*, (2023)
Blended polymer systems (emerging)	PAA–PASP blends	Potential synergy of high efficiency (PAA) + biodegradability (PASP), industrial interest (patents), scalable	Very limited peer-reviewed systematic studies under oilfield conditions	CN111233174A (2019); CN111908626A (2020)

As shown in Table 2, although eco-friendly inhibitors have advanced considerably, most approaches remain constrained by either cost, operational reliability, or limited validation under realistic oilfield conditions. In this context, blended polymer systems such as polyacrylic acid–polyaspartic acid (PAA-PASA) offer a promising balance between performance and sustainability, yet systematic studies evaluating their efficiency, retention, and applicability in oilfield environments are still lacking. This gap forms the basis of the present investigation.

Polyacrylic acid (PAA) and polyaspartic acid (PASA) are promising candidates in the context of eco-friendly scale inhibitor. PAA, an anionic polyelectrolyte at neutral pH, has demonstrated effectiveness as a scale inhibitor, crystal dispersant, and water softener due to its hydrophilicity and acid-base properties (Zhigen *et al.,* 2023). PASA, known for its biodegradability and high calcium affinity, has been used against carbonate, sulfate, and phosphate scales, and also offers anticorrosive properties (Adelnia *et al.,* 2019; Yan *et al.,* 2023; Zhigen *et al.,* 2023).

This study investigates the synergistic use of PAA and PASA. While PASA suppresses scale nucleation and growth, PAA aids in dispersing any precipitates that form, potentially providing comprehensive protection against scale deposition under complex oilfield conditions.

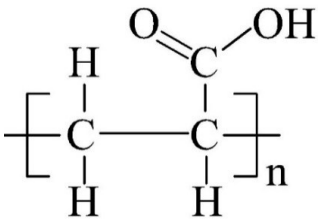

Figure 1: Repeating unit of PAA

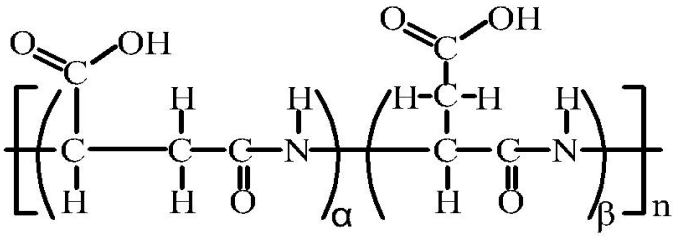

Figure 2: Repeating unit of PASA

As shown in Figures 1 and 2, polyacrylic acid (PAA) contains carboxylic acid (-COOH) groups, while polyaspartic acid (PASA) has both carboxylic acid and amide (-CONH-) functional groups per repeat unit. Upon deprotonation, these groups act as active binding sites for scaling cations. This study evaluates the efficiency of blended commercial PAA and PASA formulations in inhibiting scale formation in oilfield produced water.

METHODOLOGY

Materials

The primary materials used include a produced water sample, scale inhibitors (PAA and PASA), and analytical-grade reagents. Five litres of untreated oilfield produced water were sourced from a production facility in Delta State, Nigeria. Sample handling, preservation, and treatment followed ISO 5667-3 protocols (Syed, 2023). Collection

was done using sterilized glass bottles, stored in sealed, light-proof coolers with ice chips, and transported to the laboratory (Ujile and Dagde, 2014). Samples were refrigerated until analysis. Temperature and pH were measured on-site due to their sensitivity, while other physicochemical properties were determined in the lab (Emam *et al.*, 2014).

Instruments

Key instruments included a pen-type pH meter (PH-009[I]), a constant temperature water bath (G-Bosh DK-420), rotary stirrer, electronic analytical balance (Toledo PL303), mercury-in-glass thermometer, orbital shaker, and pipettes.

Reagents

All reagents were analytical grade, procured from KincelExcel International (181 Warri-Sapele Road). These included PAA (Mw ~2000 g/mol), PASA (Mw ~3000 g/mol), disodium dihydrate salt of EDTA, Eriochrome Black T (EBT) indicator, calcium chloride, sodium hydroxide, hydrochloric acid, ammonia-ammonium chloride buffer, and deionized water.

Experimental Design

The experimental setup was developed using Design Expert software (v13.0.5.0, 64-bit) based on the Optimal (Combined) Design methodology. Input parameters are detailed in Table 3.

Table 3: Input data for Optimal (Combined) Design

PARAMETERS SETTING	NAME	UNIT	CHANGE	TYPE	LEVEL (L)		
					L1	L2	L3
NUMBER OF MIXTURE COMPONENTS: 2	PAA DOSAGE	ppm	HARD	nil	0.01 (low)	nil	0.1 (high)
	PASA DOSAGE	ppm	HARD	nil	0.01 (low)	nil	0.1 (high)
NUMBER OF NUMERIC FACTORS: 2	Temperature (TEMP.)	degree Celsius (^oC)	EASY	DISCRETE	50	60	70
	pH	nil	HARD	DISCRETE	6	7	8
NUMBER OF RESPONSE: 1	Inhibition Efficiency	(%)	nil	nil	nil	nil	nil

The ranges of inhibitor concentration, pH, and temperature used in this study as shown in Table 3 were chosen to align closely with both field practices and reported produced-water geochemistry. Typical polymeric inhibitor dosages lie in the ppm scale for scale control and squeeze treatment programs, with lab studies frequently

exploring up to several hundred ppm (Kan, Dai, and Tomson, 2020; Jarrahian, Sorbie, and Boak, 2022; Mady, Rehman, and Kelland, 2021). The 0.01ppm to 0.1ppm dosage range chosen in this investigation was prompted by the value of the Total Hardness of the produced water sample used for the experimental work, the results of the physico-chemical analysis of the produced water sample is presented in Table 4. With a Total Hardness of 2.4 (mg/L), 0.01ppm to 0.1ppm inhibitor concentration prevent over dosage of the PAA-PASA blends. The pH interval of 6.0–8.0 was adopted to match the span commonly observed in produced waters, which often vary in this window depending on reservoir and treatment conditions (Ullattumpoyil, 2023; Nath, Chowdhury, and Rhaman, 2023). For example, one geochemistry survey in the Ghawar field reported produced water pH values spanning 6.0 to 7.4. (Ullattumpoyil, 2023) Likewise, another review of produced-water characteristics shows that pH values in shale plays can extend from ≈ 3.9 to ≈ 9.3 depending on locale (Nath *et al.,* 2023). The selected temperature points from 50 °C to 70 °C to encompass surface, handling, and moderate-to-high reservoir conditions, ensuring that the thermal stability of PAA-PASA blends and performance trends under field-relevant thermal exposures can be assessed. These parameter windows ensure that the experimental design remains realistic, mechanistically insightful, and comparable with prior laboratory-to-field inhibitor studies.

Results from Experimental Design and Preparation of Inhibitor Solutions

The outcomes of the Optimal (Combined) Design methodology are summarized in Table 5.

Preparation of Inhibitor Solutions

Stock solutions of polyacrylic acid (PAA) and polyaspartic acid (PASA) were prepared by dissolving 0.5 g of each polymer in 1000 mL of deionized water to obtain 500 ppm solutions. Standard working solutions were then prepared using the dilution formula:

$$C_1V_1=C_2V_2$$

where:

C_1 is the concentration of the original/stock solution, V_1 is the volume of the original/stock solution used, C_2 is the concentration in the dilute solution obtained, and V_2 is the volume of dilute solution used.

1 ppm standard solution: 0.2 mL of 500 ppm stock diluted in 100 mL deionized water.

5.5 ppm standard solution: 1.1 mL of 500 ppm stock diluted in 100 mL deionized water.

10 ppm standard solution: 2.0 mL of 500 ppm stock diluted in 100 mL deionized water.

From these standard solutions, inhibitor dosages as given in the experimental design (Table 5), were obtained in 50 mL of produced water:

0.01 ppm inhibitor dosage: 0.5 mL of 1 ppm solution

0.055 ppm inhibitor dosage: 0.5 mL of 5.5 ppm solution

0.1 ppm inhibitor dosage: 0.5 mL of 10 ppm solution

Experimental Procedure

Scale inhibition efficiency was evaluated via static jar tests based on GB/T 16632-2008 (Xinyu *et al.,* 2020; Thin and Defang, 2022), using experimental parameters (temperature, pH, PAA, and PASA concentrations) outlined in Table 5.

For each run, 50 mL of produced water was treated with the appropriate inhibitor concentration. pH was adjusted using 50% HCl or 50% NaOH solutions. A buffer (5 mL of a mixture containing 8.5 mol/dm³ aqueous ammonia and ammonium chloride) was added to maintain pH stability during the reaction.

Treated and control (untreated) samples were incubated in a constant temperature water bath for 24 hours under continuous agitation, corresponding to the designated temperature for each run. After reaction, samples were cooled to room temperature and filtered using double filter paper. Filtrates were titrated with 0.5 mg/L EDTA solution to quantify residual scaling cations.

The output responses (scale inhibition efficiency η) were calculated as:

$$\eta = \frac{C_X - C_Y}{C_O - C_Y} \times 100\%$$

where:

C_X is the concentration of scaling cation left in the produced water sample with added scale inhibitor at the end of the reaction.

C_Y is the concentration of scaling cation left in the produced water sample without scale inhibitor at the end of the reaction.

C_O is the concentration of scaling cation present in the original oilfield produced water sample before the reaction.

RESULTS AND FINDINGS

The physico-chemical analysis of the Produced Water Sample, as presented in Table 4, indicates the presence of various scaling species. The Total Hardness of the sample was measured at 2.40 mg/L, despite the Calcium ion (Ca^{2+}) concentration being recorded at less than 0.001 mg/L. This suggests that calcium is not the dominant contributor to the observed hardness.

Additionally, the total iron content—comprising both ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions—was found to be 0.548 mg/L. Given these values, the hardness must be primarily due to the presence of other multivalent cations such as magnesium (Mg^{2+}), barium (Ba^{2+}), and possibly scandium (Sc^{3+}).

The presence of these alternative scaling ions confirms the complexity of the scaling potential in the produced water sample and underscores its suitability for evaluating the performance of blended scale inhibitors.

Table 4: Physico-chemical Properties of the Oilfield Produced Water Sample.

S/NO.	PARAMETERS	RESULTS	ANALYSIS METHOD
1	pH	9.2 at 34.3°C	SM-4500-B
2	Turbidity (NTU)	100	SM-2130B
3	Conductivity (µs/cm)	1659	SM-2520B
4	Total Dissolved Solids (mg/L)	832	SM-2540C
5	Total Suspended Solids (mg/L)	2460	SM-2540D
6	Total Alkalinity (mg/L)	1212.8	SM-2320B
7	Total Hardness (mg/L)	2.40	SM-2340C
8	Chloride (mg/L)	750	SM-4500C
9	Sulphate (mg/L)	1.00	ASTM D 515-16
10	Carbonate as CO_3^{2-} (mg/L)	0.01	SM-2320B
11	Bicarbonate as HCO_3^- (mg/L)	<0.01	SM-2320B
12	Calcium as Ca^{2+} (mg/L)	<0.001	SM-3500-Ca-B
13	Sodium as Na^+ (mg/L)	0.016	SM-3500-Na-B
14	Potassium as K^+ (mg/L)	11.8	SM-3500-K-B
15	Total iron (mg/L)	0.548	SM-3500-Fe-B
16	Density (kg/m^3)	0.982	ASTM D1429-A

Where: ASTM is American Society for Testing and Materials.
SM is Standard Methods for the Examination of Water and Waste Water.

The influence of interacting variables—PAA-PASA blend dosage, temperature, and pH—on scale inhibition efficiency was evaluated using Optimal (Combined) Design. A maximum inhibition efficiency of 97.42% was achieved. The experimental design matrix and corresponding responses are summarized in Table 5.

Table 5: Optimal (Combined/Mixture) Design Matrices for the scale inhibition efficiency (output response) in terms of actual mixture components and experimental factors values.

Concentration of Scalants Cation Before Experiment (Co): 2.40ppm

EXP. RUNS	INHIBITOR DOSAGE (ppm)		EXPERIMENTAL FACTORS		CONCENTRATION OF SCALING CATION AFTER EXPERIMENT (ppm)		INHIBITION EFFICIENCY (OUTPUT RESPONSE) (%)
	PAA	PASA	TEMP. (°C)	pH	C_X	C_Y	
01	0.01	0.1	50	6	2.81	2.79	-5.13
02	0.01	0.1	60	6	3.24	3.10	-20.00
03	0.01	0.1	70	6	5.10	3.86	-84.93
04	0.1	0.01	70	8	0.55	0.14	18.14
05	0.1	0.01	60	8	2.15	0.79	84.47
06	0.1	0.01	50	8	1.69	0.81	55.35
07	0.055	0.055	50	8	2.36	0.85	97.42
08	0.055	0.055	70	8	1.43	0.16	56.70
09	0.055	0.055	60	8	1.89	0.74	69.28
10	0.055	0.055	60	6	3.61	3.26	-40.70

EXP. RUNS	INHIBITOR DOSAGE (ppm)		EXPERIMENTAL FACTORS		CONCENTRATION OF SCALING CATION AFTER EXPERIMENT (ppm)		INHIBITION EFFICIENCY (OUTPUT RESPONSE) (%)
	PAA	PASA	TEMP. (°C)	pH	C_X	C_Y	
11	0.055	0.055	70	6	3.97	3.41	-55.45
12	0.055	0.055	50	6	2.98	2.93	-9.43
13	0.01	0.1	70	7	1.15	1.12	2.34
14	0.01	0.1	60	7	1.39	1.33	5.56
15	0.01	0.1	60	7	1.38	1.32	5.61
16	0.1	0.01	50	7	1.36	1.21	12.61
17	0.1	0.01	60	7	1.34	1.22	10.3
18	0.1	0.01	60	7	1.35	1.23	10.17
19	0.055	0.055	60	7	1.46	1.41	5.05
20	0.055	0.055	70	7	1.72	1.16	45.16
21	0.055	0.055	60	7	1.45	1.40	5.00
22	0.1	0.01	60	6	3.32	3.14	-24.32
23	0.1	0.01	70	6	4.58	3.64	-75.81
24	0.1	0.01	50	6	2.94	2.89	-8.16
25	0.01	0.1	60	8	2.20	0.83	87.26
26	0.01	0.1	70	8	0.54	0.17	16.59
27	0.01	0.1	50	8	1.57	0.88	45.39

Effect of pH, Temperature, and Inhibitor Dosage on Scale Inhibition Efficiency

As shown in Table 5, scale inhibition efficiency generally increased with rising pH and decreased with temperature, though these effects were also influenced by the specific dosages of polyacrylic acid (PAA) and polyaspartic acid (PASA) in the inhibitor blend. This trend can be attributed to the chemical structures of PAA and PASA (Figures 1 and 2), which contain carboxylic acid groups; PASA also includes an amide group. In alkaline conditions, these functional groups undergo significant deprotonation, increasing the number of reactive sites available to bind scaling cations, thereby enhancing inhibition efficiency. Similar observations were made by Ferguson (2014).

Lower temperatures further improved inhibition efficiency, likely due to reduced kinetic energy of the scaling ions, which allows more effective interaction with the inhibitors. This suggests that scale formation may be more endothermic than the inhibition process.

The optimal performance was recorded in Experimental RUN 07, where a 0.055 ppm blend of PAA and PASA at 50 °C and pH 8 achieved a maximum inhibition efficiency of 97.42%. Comparatively, RUN 06 and RUN 27, which had the same temperature and pH conditions, demonstrated significantly lower efficiencies (55.35% and 45.39%, respectively). The disparity is attributed to variations in the PAA-PASA ratio, indicating that a balanced dosage is crucial and that PAA exhibits notable microscale dispersant properties.

Negative inhibition values, as seen in RUN 03 (-84.93% at 70 °C, pH 6), occurred primarily under acidic conditions. This suggests that the original produced water may have contained fine scale crystals that became more soluble at low pH and high temperature. In such environments, insufficient deprotonation of the inhibitor molecules limits their reactivity with scaling ions. Hence, the inhibitors are more effective under alkaline conditions where functional groups are more ionized and reactive.

DISCUSSION

Statistical Modeling and Analysis of Variance (ANOVA)

The experimental data were best described by a quadratic model, with a predicted R^2 of 0.9375. The small gap between predicted and adjusted R^2 (0.7969) confirms the model's robustness.

ANOVA results (Table 6) were used to evaluate the significance of each model term. Terms with higher F-values and lower p-values were considered more significant. A greater disparity between F- and p-values indicates stronger contribution to the model, aligning with findings by Ajaz *et al*. (2020).

Table 6: ANOVA for the Quadratic model.

Source	F-value	p-value	Comment
Whole-plot	17.20	< 0.0001	significant
Linear Mixture	0.2547	0.6248	
ab	2.409E-06	0.9988	
ad	33.84	0.0002	significant
bd	32.10	0.0002	significant
abd	1.40	0.2649	
ad^2	1.27	0.2864	
abd^2	1.59	0.2359	
Subplot	3.31	0.0379	significant
aC	9.35	0.0121	significant
bC	6.10	0.0331	significant
Cd	1.86	0.2022	
abC	0.5199	0.4874	
aCd	0.1514	0.7054	
bCd			
aC^2	4.92	0.0508	
bC^2	2.64	0.1354	
abCd	0.5990	0.4569	
abC^2	9.09	0.0130	significant

In this study, model terms with p-values below 0.0500 were deemed statistically significant. As shown in Table 6, four interaction terms (aC, ad, bC, and bd) and one

quadratic term (abC^2) met this criterion. Here, 'a', 'b', 'c', and 'd' represent PAA dosage, PASA dosage, temperature, and pH, respectively.

Model Equation

Using an Optimal (Combined/Mixture) Design and actual experimental values for scale inhibition efficiency, a second-order polynomial model was developed. The resulting equation, expressed in terms of L_pseudo components and coded factors, is presented in Equation (1).

Inhibition Efficiency =12.006a + 19.6075b - 0.087ab - 27.1005ac + 44.375ad - 21.065bc + 43.2167bd + 12.75cd + 31.111abc + 44.1367abd - 5.14acd + 0bcd - 30.0385 ac^2 + 16.298ad^2 - 20.5425bc^2 - 35.42abcd + 197.802abc^2 - 81.616abd^2…… Equation (1)

Where:

'a', and 'b' are L_pseudo components, 'c' and 'd' are coded factors.

$$L\_pseudoComponenentValue(a,b) = \frac{100 \times ActualComponentValue}{9} - \frac{1}{9}$$

… Equation (2)

$$CodedTemperatureValue(c) = \frac{ActualTemperatureValue}{10} - 6$$ …… Equation (3)

$$Coded\_pH\_Value(d) = (Actual\_pH\_value) - 7$$ …… Equation (4)

Equation (2), Equation (3), and Equation (4) are used for conversion of actual experimental parameters values to the coded values used in the model Equation (1).

The model demonstrated a strong fit to the experimental data, explaining 79.69% of the variability based on the adjusted R^2 value (0.7969) as shown in Equation (1). Model adequacy was further supported by diagnostic plots (Figures 3 and 4).

Diagnostic Analysis

The normal probability plot of residuals (Figure 3) shows no significant deviation from normality, with data points closely following the reference line—indicating a satisfactory agreement between the model predictions and observed responses.

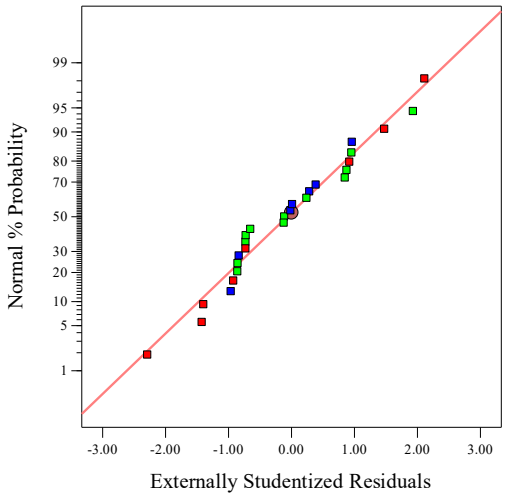

Figure 3: Normal plot of residuals

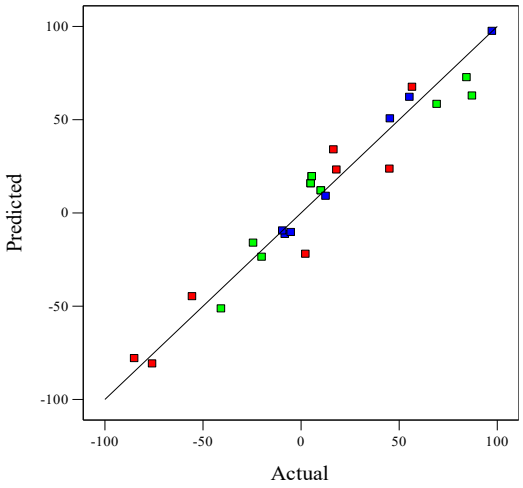

Figure 4: Predicted vs. Actual

Figure 4 illustrates the comparison between predicted and actual responses across the experimental runs. The data points closely follow a straight line, suggesting normal distribution and a strong model fit.

Model Performance Summary

Table 7 provides a summary of the model's predictive performance. The output responses were estimated using Equation (1).

Table 7: Model report for optimal (combined/mixture) design for scale inhibition efficiency in terms of L_pseudo mixture components and experimental coded factors values.

	Mixture Components		**Experimental Factors**		**Response**		**Residual**
EXP. RUNS	a:PAA	b:PASA	c:Temp.	d:pH	ACTUAL Inhibition Efficiency (%)	PREDICTED Inhibition Efficiency (%)	
1	0.00	1.00	-1.00	-1.00	-5.13	-10.3367	5.2067
2	0.00	1.00	0.00	-1.00	-20	-23.6092	3.6092
3	0.00	1.00	1.00	-1.00	-84.93	-77.9667	-6.9633
4	1.00	0.00	1.00	1.00	18.14	23.15	-5.01
5	1.00	0.00	0.00	1.00	84.47	72.679	11.791
6	1.00	0.00	-1.00	1.00	55.35	62.131	-6.781
7	0.50	0.50	-1.00	1.00	97.42	97.500025	-0.08
8	0.50	0.50	1.00	1.00	56.7	67.540025	-10.84
9	0.50	0.50	0.00	1.00	69.28	58.360025	10.92
10	0.50	0.50	0.00	-1.00	-40.7	-51.300025	10.6

11	0.50	0.50	1.00	-1.00	-55.45	-44.770025	-10.68
12	0.50	0.50	-1.00	-1.00	-9.43	-9.510025	0.08003
13	0.00	1.00	1.00	0.00	2.34	-22	24.34
14	0.00	1.00	0.00	0.00	5.56	19.6075	-14.048
15	0.00	1.00	0.00	0.00	5.61	19.6075	-13.998
16	1.00	0.00	-1.00	0.00	12.61	9.068	3.542
17	1.00	0.00	0.00	0.00	10.3	12.006	-1.706
18	1.00	0.00	0.00	0.00	10.17	12.006	-1.836
19	0.50	0.50	0.00	0.00	5.05	15.785	-10.735
20	0.50	0.50	1.00	0.00	45.16	23.64	21.52
21	0.50	0.50	0.00	0.00	5	15.785	-10.785
22	1.00	0.00	0.00	-1.00	-24.32	-16.071	-8.249
23	1.00	0.00	1.00	-1.00	-75.81	-80.82	5.01
24	1.00	0.00	-1.00	-1.00	-8.16	-11.399	3.239
25	0.00	1.00	0.00	1.00	87.26	62.8242	24.4358
26	0.00	1.00	1.00	1.00	16.59	33.9667	-17.377
27	0.00	1.00	-1.00	1.00	45.39	50.5967	-5.2067

Residual Analysis and Model Validation

Residuals (Table 7) were calculated as the differences between observed and predicted values, indicating the portion of experimental variation not explained by the model.

Model Optimization and Verification

The scale inhibition efficiency of PAA–PASA blends was optimized using the desirability function approach. Optimal conditions are summarized in Table 8. Verification was conducted using selected conditions (temperature and pH) that extended beyond the original experimental design space.

Table 8: Optimized solutions for scale inhibition efficiency of 100%, of PAA-PASA blends, (Desirability of 1.00)

S/N	a:PAA (ppm)	b:PASA (ppm)	c:Temp (°C)	d:pH	Actual In.Eff. (%)	Model Predicted In.Eff. (%)	Residuals
1	0.028	0.082	38.45	7.05	100.00	99.79	0.21
2	0.011	0.099	55.08	9.05	81.37	99.58	-18.21
3	0.023	0.087	69.11	9.93	74.06	101.21	-27.15
4	0.057	0.053	80.44	7.34	97.85	99.92	-2.07
5	0.025	0.085	45.93	9.30	84.22	99.67	-15.45

Table 8 presents selected optimized solutions confirming the influence of temperature and pH on the scale inhibition performance of PAA-PASA blends. At

lower temperatures (<40°C) and near-neutral pH (~7), complete inhibition (100%) was achieved (Optimized Solution S/N: 01). However, efficiency declined to 97.85% at 80°C and pH 7.34 (S/N: 04), suggesting enhanced deprotonation of PAA and PASA at this pH, promoting binding with scaling cations. A further decrease in inhibition efficiency was observed at higher alkalinity and temperature, with the lowest efficiency (74.06%) recorded at pH 9.93 and 69.11°C (S/N: 03). The further drop in scale inhibition efficiency as the condition tends higher towards pH 10 is an indication of higher rate of scaling reaction which effectively compete with the antiscaling activity of the blends. These findings indicate that the predictive model is most reliable under milder conditions (temperature <40°C, pH slightly above 7), as seen in the minimal residuals associated with S/N: 01. The validated model provides a useful framework for understanding the behavior of PAA-PASA blends within the tested design space.

While the mechanistic insights into functional group deprotonation and the dispersant activity of PAA help explain the observed inhibition performance, it is equally important to consider the operational implications of adopting PAA-PASA blends in oilfield practice. From a compatibility perspective, polymeric inhibitors generally exhibit fewer antagonistic interactions with commonly deployed additives such as corrosion inhibitors, demulsifier and biocides compared with phosphonate-based systems, which are prone to forming insoluble complexes with divalent cations. Nevertheless, systematic compatibility testing remains essential before field implementation. Cost considerations also influence practical deployment: PAA is inexpensive and widely available but has environmental drawbacks, whereas PASA is costlier yet biodegradable. By blending these polymers, it may be possible to optimize cost-effectiveness while satisfying increasingly strict environmental standards. Furthermore, if PAA-PASA formulations demonstrate improved adsorption and retention in reservoir rock, they could extend squeeze treatment lifetimes, reducing the frequency of costly intervention operations. These factors collectively suggest that polymer blends have potential not only as mechanistically sound inhibitors but also as viable, sustainable alternatives in oilfield scale management, provided that field-oriented evaluations confirm laboratory trends.

CONCLUSION

This study successfully evaluated the efficacy of PAA-PASA blends as scale inhibitors for oilfield produced water. Using an Optimal (Combined) Design methodology over a 24-hour reaction time, the interactive effects of dosage, pH, and temperature on inhibition efficiency were investigated through static jar tests.

A quadratic model was developed and validated using statistical diagnostics, with model adequacy confirmed by R^2 = 0.9375 and Adjusted R^2 = 0.7969. The model demonstrated that inhibition performance improved under conditions of reduced temperature (<50°C) and slightly alkaline pH (>7). Within experimental design range,

the highest inhibition efficiency (97.42%) was obtained at 50°C, pH 8, with 0.055 ppm each of PAA and PASA.

The findings highlight the potential of PAA-PASA blends to serve as effective, economical, and environment friendly scale inhibitors for oilfield applications under the studied conditions.

Recommendations and Future Work

Future studies should consider:

- Kinetics: Investigate scale formation rates under varying conditions (temperature, pH, and cation type).
- Equilibrium analysis: Determine the maximum inhibition efficiency under different operational scenarios.
- Molecular weight effects: Assess how varying molecular weights of PAA and PASA affect blend performance.
- Scale morphology: Use SEM to analyze how the blends alter the crystal structure of scale deposits.
- Pilot testing: Evaluate blend performance at pilot scale for field applicability.
- Recovery and reuse: Explore recovery strategies for PAA and PASA to improve cost-efficiency and sustainability.

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