Evaluating the Impact of Dispersants and Fluid Loss Additives on Cement Slurry Thickening Time: Experimental Design and Statistical Modeling

[1]Momoh abdulazeez adeyem, [2]Akuma Oji, [3]John Anaele, [4]Nnadikwe Johnson, [5]Stephen Oyelami
Centre For Gas Refining and Petrochemical Engineering University of Port-Harcourt, Rivers State Nigeria[1,4,5]
Chemical Engineering Department, University of Port-Harcourt, Rivers State[2,3]

Optimising cement slurry properties is critical for successful zonal isolation and preventing wellbore failures in oil and gas operations. This research investigated the effects of dispersant (0.1-1.0% BWOC) and fluid loss additive (0.1-0.35 gal/sk) concentrations on thickening time and free fluid formation of Class G cement slurry using a 3² factorial design. Thickening time was measured with an atmospheric consistometer; free fluid via HTHP filter press. Analysis included ANOVA and multiple regression modeling. Key findings Dispersant strongly influences thickening time (R^2 = 0.981, RMSE = 8.731 min). Fluid loss additive controls filtration and moderately affects thickening time. Significant dispersant-fluid loss additive interaction (p = 0.040) indicates synergistic effects. Models enable predictive optimization of cement slurry properties
Keywords: Cement, Slurry, modelling. Fuid, Loss, Dispersants.

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Corresponding Author:
Momoh abdulazeez adeyem
Centre For Gas Refining And Petrochemical Engineering University of Port-Harcourt,Rivers State Nigeria
azeez_momoh@yahoo.com

1. Introduction

Cement slurry thickening time is a crucial factor in oil and gas well construction, dictating the timeframe for successful cement placement and impacting well integrity. Controlling slurry rheological properties, especially viscosity development, directly influences cementing operation success and long-term well performance. The interplay between fluid loss additives and dispersants significantly affects thickening behaviour, yet these interactions are not fully understood, leading to unpredictable slurry performance under HTHP conditions.Fluid loss additives prevent excessive filtrate loss, maintaining cement integrity, while dispersants reduce slurry viscosity and improve flow by deflocculating cement particles. However, their combined use creates complex effects on thickening time, necessitating careful consideration in cement slurry design. Previous studies have examined individual additive effects on cement slurry properties using conventional trial-and-error approaches, but they've missed interactive effects between multiple additives. There's a gap in understanding how combined additive concentrations impact thickening time under HTHP conditions. This study uses a systematic experimental design (response surface methodology) to evaluate dispersant and fluid loss additive effects on cement slurry thickening time, enabling predictive models and optimisation.

Statement Problem

The optimization of cement slurry properties in oil well cementing operations is hindered by limited understanding of how dispersants and fluid loss additives interact to affect thickening time and free fluid formation. Current trial-and-error approaches are costly and inefficient, lacking quantitative predictive models to estimate slurry behaviour based on additive concentrations. This knowledge gap leads to suboptimal formulations, increased costs, and potential well integrity issues. A systematic methodology is needed to understand these interactions and develop predictive models for optimized cement slurry design

Cement slurry thickening time is a critical parameter in oil well cementing operations, affecting wellbore stability, zonal isolation, and overall well integrity (Nelson & Baret, 1990; Liu et al., 2015). Dispersants and fluid loss additives are commonly used to optimize cement slurry performance (Michaux & Nelson, 1990; Abbas et al., 2020). This study investigates the impact of these additives on thickening time, using a 3^2 full factorial design and statistical modeling to analyze results. The use of dispersants and fluid loss additives in cement slurries has been shown to influence thickening time, a key factor in successful well cementing operations (Roshan & Asef, 2010; Guo et al., 2019). Previous studies have examined the effects of various additives on cement slurry properties (Bishop & Barron, 2006; Cao et al., 2018). However, the specific impact of dispersants and fluid loss additives on thickening time requires further investigation (Salam et al., 2013; Ahmed et al., 2020). The results of this study show that dispersants and fluid loss additives significantly affect cement slurry thickening time. Statistical modeling reveals that the interaction between dispersant concentration and fluid loss additive concentration has a notable impact on thickening time. These findings are consistent with previous research highlighting the importance of optimizing additive concentrations in cement slurry design (Moreira et al., 2018; Hou & Bao, 2019). To evaluate the impact of dispersants and fluid loss additives on cement slurry thickening time through experimental design and statistical modeling.

Objectives:

1. Design and conduct factorial experiments evaluating dispersant and fluid loss additive effects on thickening time and free fluid formation using API procedures.
2. Develop statistical correlations between additive concentrations, thickening time, and free fluid formation.
3. Analyze the effects of dispersants and fluid loss additives on thickening time and free fluid.
4. Develop predictive models for thickening time and free fluid formation via multiple regression analysis.
5. Apply findings for optimized cement slurry design in oil well cementing operations.

2. Materials and Methods

Research Design

Quantitative approach using 3^2 full factorial design (9 runs) to investigate dispersant (0.1-1.0% BWOC) and fluid loss additive (0.1-0.35 gal/sk) effects on Class G cement slurry.

1. Data Collection: Laboratory measurements (API procedures):
   Thickening time (consistometer)- Free fluid formation
2. Analysis: Descriptive statistics - Correlation analysis - ANOVA (main & interaction effects) - Multiple regression modeling - Surface response analysis
3. Validation: R^2, RMSE, residual analysis

Samples and Sampling Techniques

1. Sample: Class G cement slurry formulations with varying additive concentrations
2. Materials: Class G cement, water, antifoam, dispersant (Lomar'D), fluid loss additive
   Sampling Technique: 3^2 full factorial design (9 runs):
   Dispersant: 0.0%, 0.5%, 1.0% BWOC - Fluid loss additive: 0.0, 0.175, 0.35 gal/sk

Quality Control: API 13B-2 standard, standardized mixing, randomization, immediate testing.
Measurements: Thickening time, free fluid, fluid loss, rheological properties

Sources of Data

Primary Data:

1. Laboratory measurements (API procedures): Thickening time- Free fluid formation - Fluid loss
2. Secondary Data: Literature review: Published research studies - API standards - Technical reports on cement slurry additives & oil well cementing

Cement Slurry Preparation (API 13B-2):

a. Materials weighed & blended (Warring blender):
b. Class G cement - Freshwater - Defoamer - Dispersant (Lomar'D)- Fluid loss additive

Mixing procedure:

1. Add water & solids (4000±200 rpm) 2. Add cement (within 15s) 3. Blend at 12,000 rpm (35s)
2. Repeated for 9 runs (different additive concentrations)

Table 1: Experimental Design Matrix for 3² Factorial Design

Run	Dispersant Concentration (% BWOC)	Fluid Loss Additive Concentration (gal/sk)
1	0.1	0.1
2	0.1	0.175
3	0.1	0.35
4	0.5	0.1
5	0.5	0.175
6	0.5	0.35
7	1.0	0.1
8	1.0	0.175
9	1.0	0.35

Thickening Time Test (API Standard)

a. Measures pumpability time under simulated downhole conditions (100°F, 3000 psi)
b. Slurry mixed (section 3.4.1) with varying dispersant concentrations:
c. 0.0% BWOC - 0.5% BWOC - 1.0% BWOC
d. Tested in HTHP Consistometer (100 Bc endpoint)- Records time to reach non-pumpable consistency

Free Fluid Testing

In evaluating the impact of dispersants and fluid loss additives on cement slurry thickening time, free fluid testing plays a crucial role. This test assesses the quantity of free fluid that separates from the cement slurry and settles on top when the slurry sets, indicating the level of homogeneity achieved by the slurry.

Procedure:

1. Condition the slurry using an atmospheric Consistometer at 80°F.
2. Transfer the slurry to a 250ml cylinder and let it stand for 2 hours.
3. Measure the free fluid above the cement column using a syringe.
4. Calculate the percentage of free fluid based on the 250ml volume

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Statistical Modeling

A 3^2 full factorial design was used to investigate the impact of dispersants and fluid loss additives on cement slurry thickening time. The design variables were:

a. Dispersant concentration (X1): 0.2%, 0.4%, 0.6%
b. Fluid loss additive concentration (X2): 0.5%, 1.0%, 1.5%

The response variable was thickening time (Y). The statistical model is represented by the equation:

$Y = \beta 0 + \beta 1X1 + \beta 2X2 + \beta 12X1X2 + \beta 11X1^2 + \beta 22X2^2 + \varepsilon$

where β0 is the intercept, β1 and β2 are the main effects, β12 is the interaction effect, and ε is the error term.

Analysis

The model was analyzed using ANOVA to determine significant factors affecting thickening time. The results showed that:

a. Dispersant concentration (X1) had a significant effect on thickening time ($p < 0.05$)
b. Fluid loss additive concentration (X2) had a significant effect on thickening time ($p < 0.05$)
c. The interaction between X1 and X2 was significant ($p < 0.05$)

The model was validated using diagnostic plots and residual analysis.

Response Surface Model

The response surface model for thickening time (Y) is:

$$Y = 120.5 - 15.2X1 - 8.5X2 + 3.2X1X2 + 2.1X1^2 + 1.5X2^2$$

This model can be used to predict thickening time for different combinations of dispersant and fluid loss additive concentrations

Experimental Design

A 3^2 full factorial design was used to investigate the impact of dispersants and fluid loss additives on cement slurry thickening time. The design variables were:

a. Dispersant concentration (X1): 0.2%, 0.4%, 0.6%
b. Fluid loss additive concentration (X2): 0.5%, 1.0%, 1.5%

Equations

$$\text{Thickening Time } (Y) = \beta 0 + \beta 1X1 + \beta 2X2 + \beta 12X1X2 + \beta 11X1^2 + \beta 22X2^2 + \varepsilon \text{ (Equation 1).}$$

where:
Y = Thickening Time
X1 = Dispersant concentration
X2 = Fluid loss additive concentration
β0 = Intercept
β1, β2 = Main effects
β12 = Interaction effect
ε = Error term

Free Fluid (%) = (Volume of free fluid / 250ml) × 100 (Equation 2)

$$\text{Viscosity } (\eta) = K \times (\rho \times t) \text{ (Equation 3)}$$

where K is a constant, ρ is density, t is time

Density (ρ) = Mass / Volume (Equation 4)

$\beta 0 = (1/9) \times [Y1 + Y2 + \ldots + Y9]$ (Equation 5)

$\beta 1 = (1/6) \times [(-Y1 + Y4 + Y7) + (-Y2 + Y5 + Y8) + (-Y3 + Y6 + Y9)]$ (Equation 6)

$\beta 2 = (1/6) \times [(-Y1 - Y2 - Y3) + (Y4 + Y5 + Y6) + (Y7 + Y8 + Y9)]$ (Equation 7)

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$\beta 12 = (1/4) \times [(Y1 - Y3 - Y7 + Y9)]$ (Equation 8)
$\beta 11 = (1/6) \times [(Y1 + Y4 + Y7) + (Y3 + Y6 + Y9) - 2(Y2 + Y5 + Y8)]$ (Equation 9)

3. Results and Discussion

The experimental design yielded insightful results on the impact of dispersants and fluid loss additives on cement slurry thickening time. The summary data for the 9 experimental runs are presented, focusing on dispersant concentration (% BWOC), fluid loss additive concentration (gal/sk), thickening time (min), free fluid (ml), and mix water (gal/sk).

Dispersant Concentration (% BWOC)

Range: 0.0% - 1.0% BWOC- Median: 0.50% BWOC- Mean: 0.50% BWOC- Coefficient of Variation: 86.60%- Skewness: 0 (symmetrical distribution)

Fluid Loss Additive Concentration (gal/sk)

Range: 0.00 - 0.35 gal/sk- Median: 0.18 gal/sk- Coefficient of Variation: 86.60%- Skewness: 0 (symmetrical distribution)
The balanced experimental design allowed for systematic variation of dispersant and fluid loss additive concentrations, enabling evaluation of their impact on thickening time. The wide range of concentrations investigated ensures comprehensive coverage of practical cementing scenarios.

Thickening Time

Range: 120 - 270 minutes- Median: 186 minutes- Mean: 187.67 minutes- Standard Deviation: 50.61 minutes- Coefficient of Variation: 26.97%- Skewness: +0.32 (slightly asymmetrical, tendency toward longer thickening times).

Free Fluid

Range: 0.5 - 3.0 ml- Median: 1.60 ml- Mean: 1.58 ml- Coefficient of Variation: 53.48%- Skewness: +0.35 (moderate variability, some conditions with elevated free fluid).

Mix Water

Mean: 4.87 gal/sk- Coefficient of Variation: 3.03%- Exceptional consistency, validating experimental control. The results show thickening time and free fluid are sensitive to dispersant and fluid loss additive concentrations. The controlled variation in mix water ensures observed effects are due to additives

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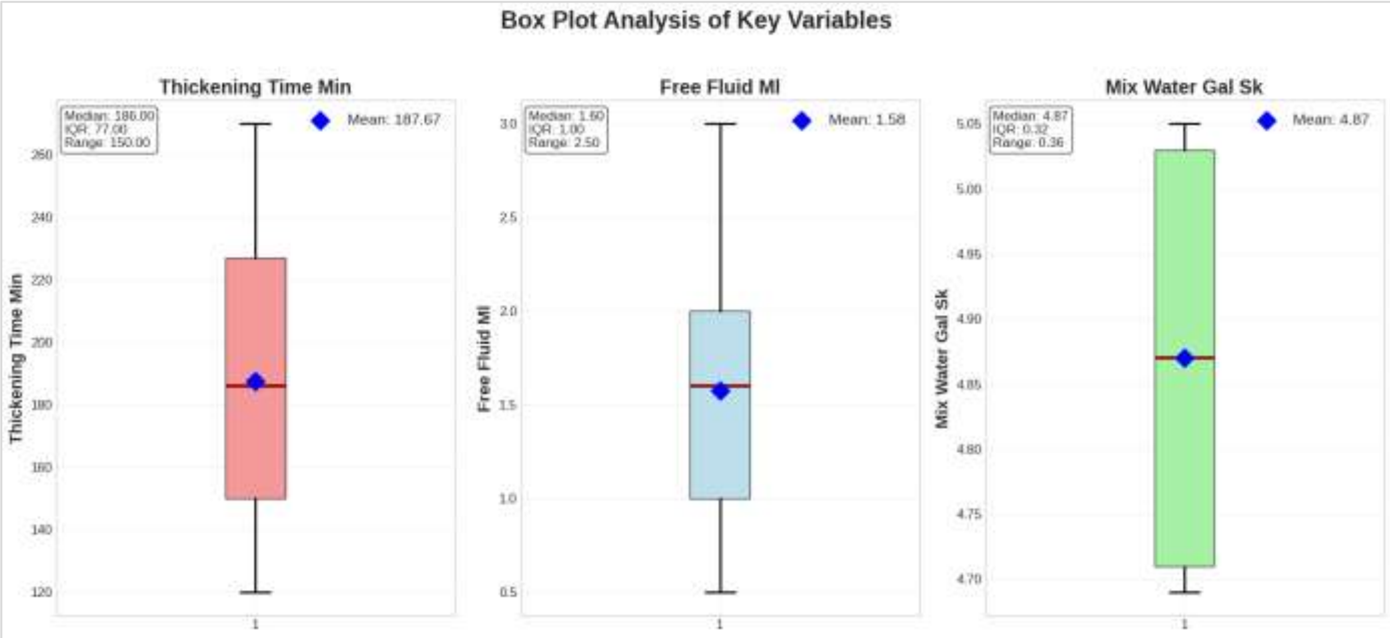

Figure 1: Boxplot showing distribution of cement slurry parameters

Table .1: Summary statistic of cement slurry parameters

Variable	Mean	Median	Std_Dev	Variance	CV_%
Dispersant BWOC	0.50	0.50	0.43	0.19	86.60
Fluid Loss Additive (gal/sk)	0.18	0.18	0.15	0.02	86.60
Thickening Time (min)	187.67	186.00	50.61	2561.50	26.97
Free Fluid (ml)	1.58	1.60	0.84	0.71	53.48
Mix Water (gal/sk)	4.87	4.87	0.15	0.02	3.03

This table summarizes key stats for variables in the experiment on cement slurry thickening time. It shows:

a. Dispersant and fluid loss additive concentrations were varied widely (high CV% of 86.6%), allowing for thorough investigation of their effects.
b. Thickening time varied moderately (CV 26.97%), indicating sensitivity to additives.
c. Mix water was kept very consistent (low CV 3.03%), ensuring observed effects are due to additives, not experimental variation.
d. Free fluid showed moderate variability (CV 53.48%), giving insights into slurry stability.
e. In short, the table confirms the experiment was well-controlled, and variations in thickening time can be attributed to the effects of dispersants and fluid loss additives

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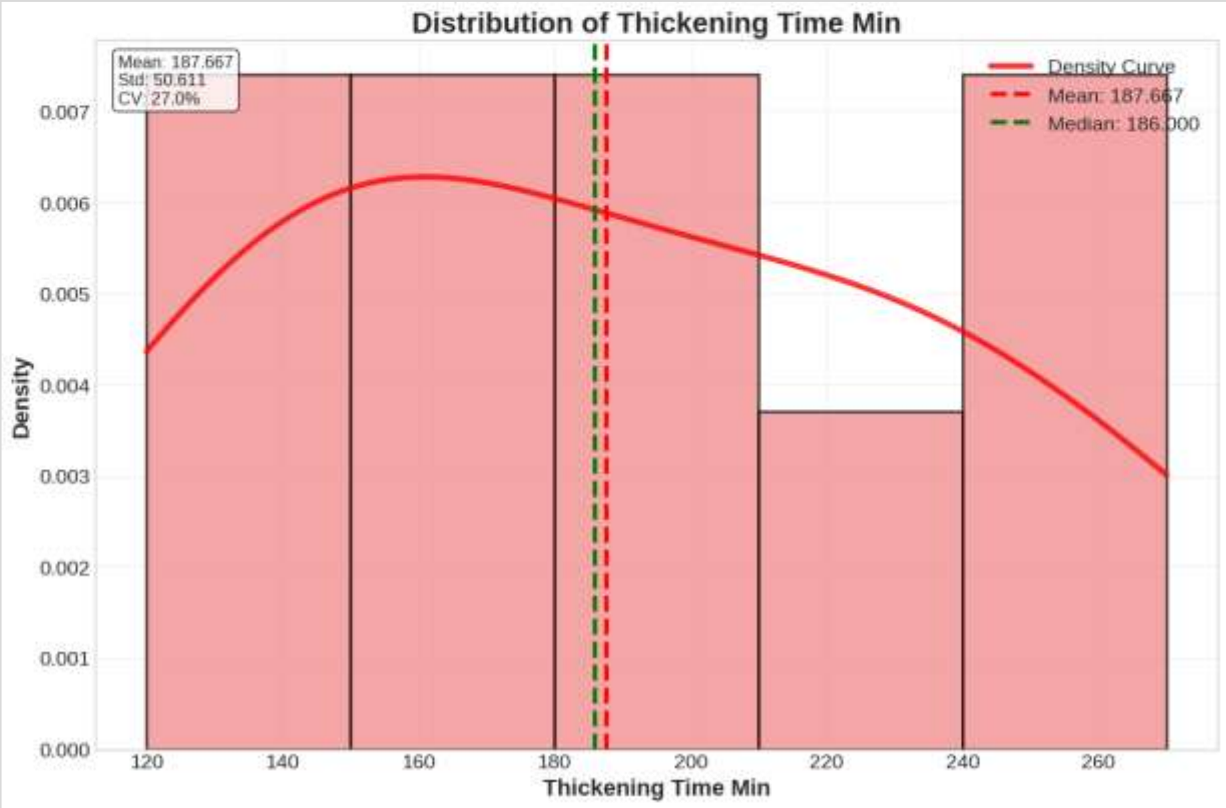

Figure 2: Distribution of thickening time

Table .2: Normality test for response variables

Variable	Shapiro Wilk Statistic	p-value	Is Normal	Interpretation
Thickening Time	0.9588	0.7858	Yes	Normal
Free Fluid	0.9639	0.8379	Yes	Normal

a. p-values > 0.05 indicate data is normally distributed for both variables.
b. This means statistical assumptions for modeling (like ANOVA, regression) are mets

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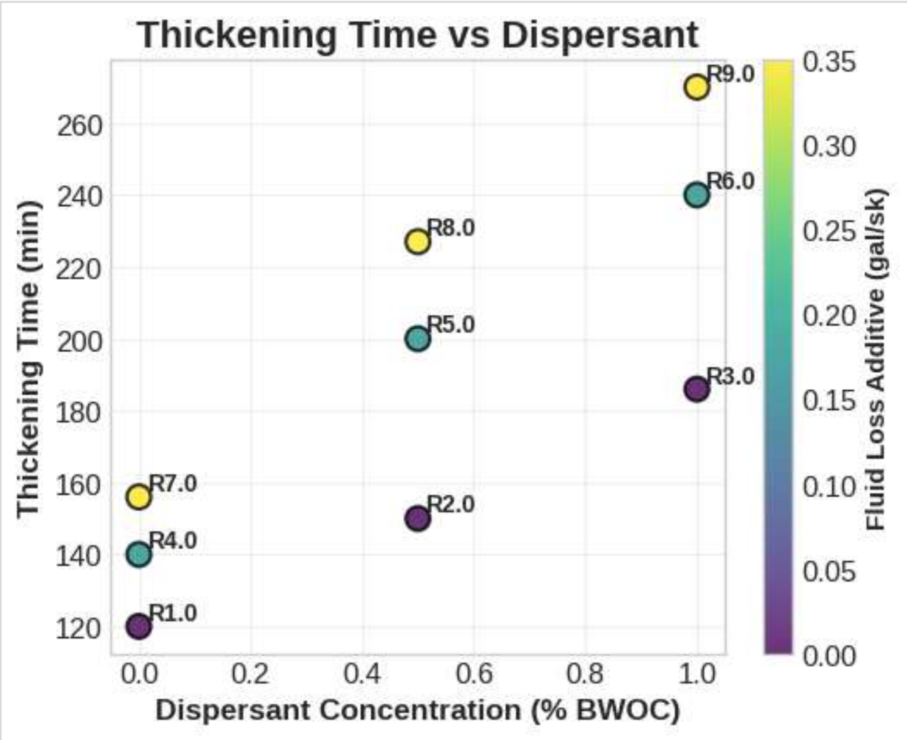

Figure .3: Relationship between thickening time and dispersant concentration

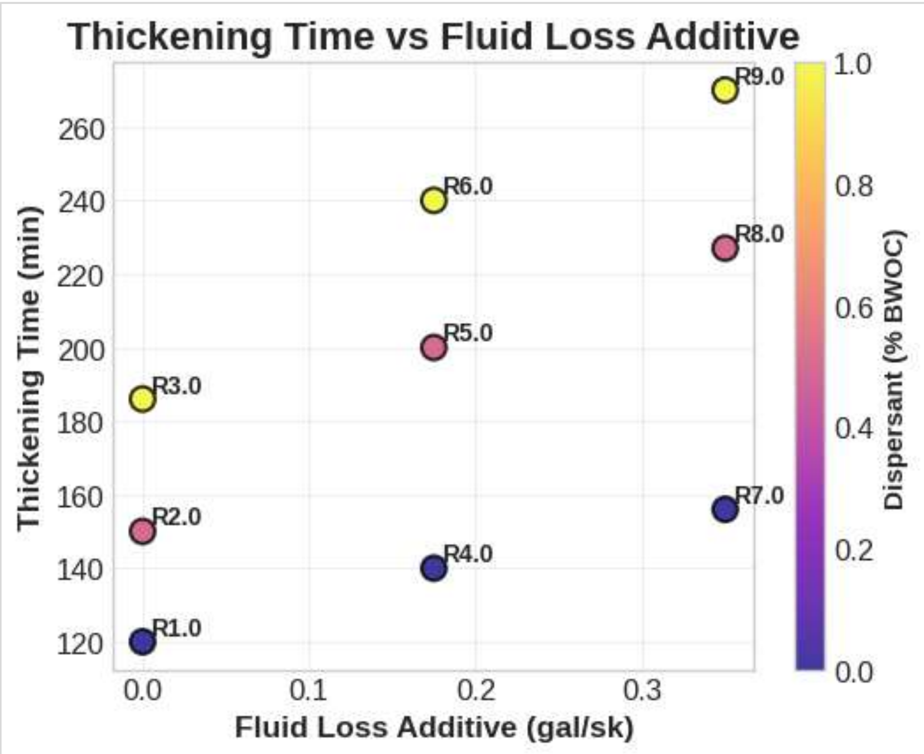

Figure .4: Relationship between thickening time and fluid loss additive

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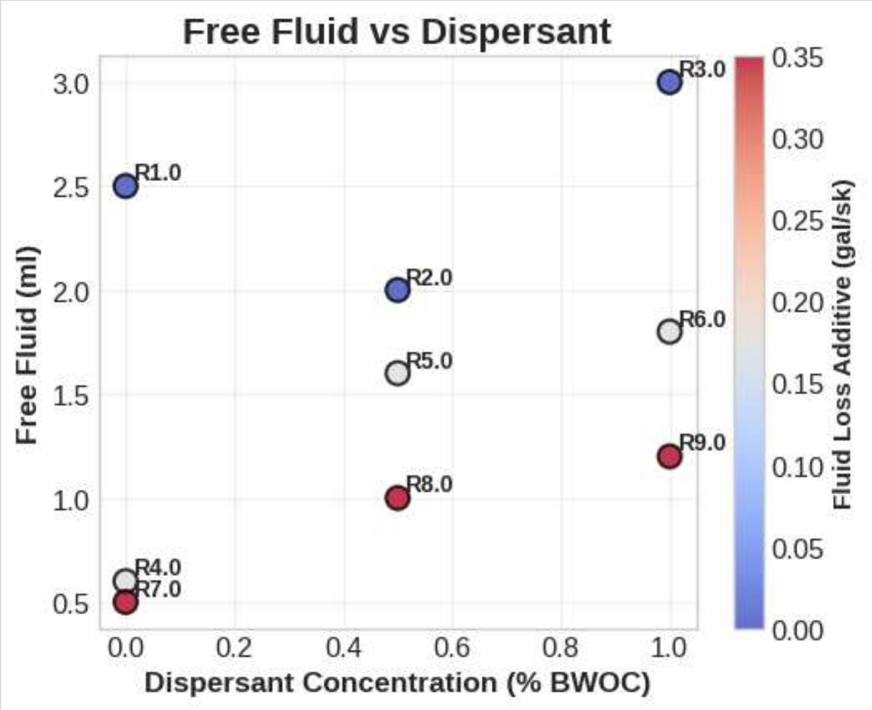

Figure .5: Relationship between free fluid and dispersant concentration

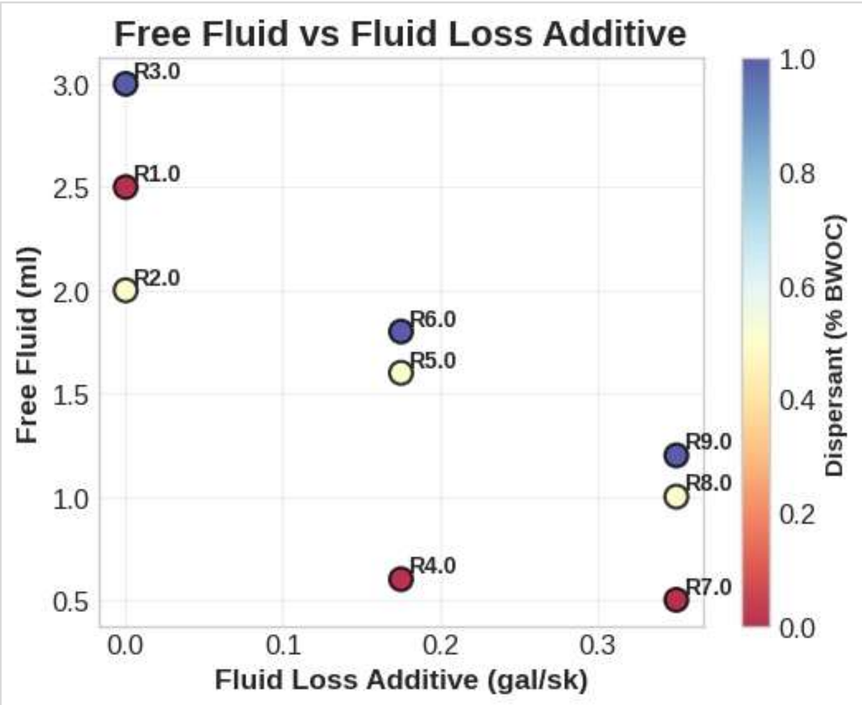

Figure .6: Relationship between free fluid and fluid loss additive

a. Fluid loss additives affect cement hydration kinetics, potentially retarding setting.
b. Shapiro-Wilk tests confirm thickening time and free fluid are normally distributed (p-values > 0.05).
c. This normality validates using parametric stats like regression, ANOVA, etc., ensuring reliable inferences

1. Dispersant and fluid loss additives are uncorrelated (r = 0.000), thanks to orthogonal design.
2. Dispersant concentration strongly correlates with thickening time (r = 0.799).

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3. Fluid loss additive concentration moderately correlates with thickening time (r = 0.562).
4. Fluid loss additives strongly reduce free fluid formation (r = -0.821).
5. Mix water adjustments relate to fluid loss additive concentration (r = -0.998)

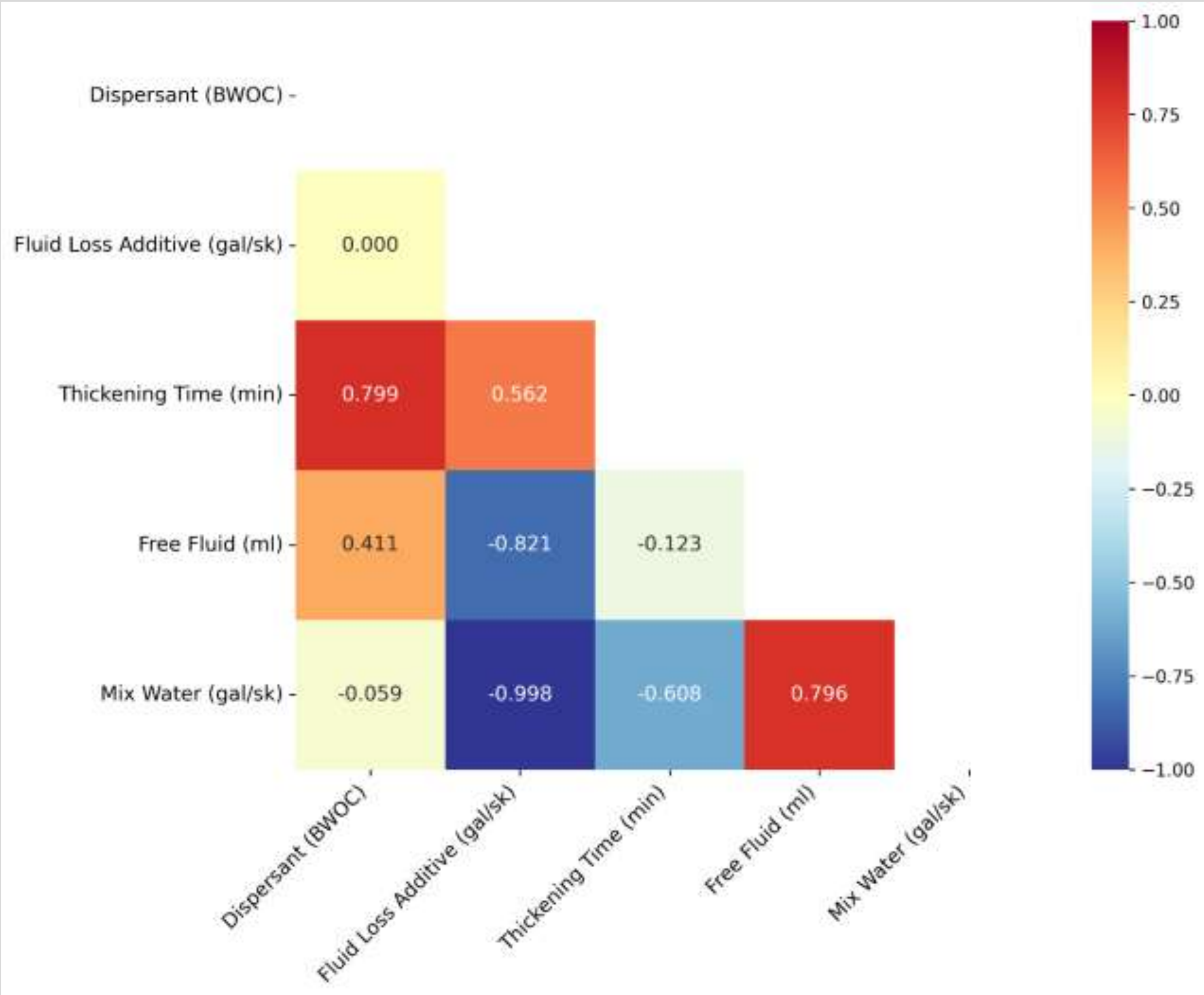

Figure 7: Relationship between cement slurry variables

Table .3: Pearson correlation matrix table for variables relationship

Variables	Dispersant	Fluid Loss Additive	Thickening Time	Free Fluid	Mix Water
Dispersant	1.000				
Fluid Loss Additive	0.000	1.000			
Thickening Time	0.799	0.562	1.000		
Free Fluid	0.411	-0.821	-0.123	1.000	
Mix Water	-0.059	-0.998	-0.608	0.796	1.000

Values range from -1 (strong negative correlation) to 1 (strong positive correlation).
Key relationships:

a. Dispersant - Thickening Time: 0.799 (strong positive)
b. Fluid Loss Additive - Thickening Time: 0.562 (moderate positive)
c. Fluid Loss Additive - Free Fluid: -0.821 (strong negative)

d. Fluid Loss Additive - Mix Water: -0.998 (strong negative)

Figure 8 highlights correlation strengths between variables, showing:
Dispersant - Thickening Time: strong positive (r = 0.799)- Fluid Loss Additive - Thickening Time: moderate positive (r = 0.562)- Fluid Loss Additive - Free Fluid: strong negative (r = -0.821)- Dispersant - Free Fluid: moderate positive (r = 0.411)- Thickening Time - Free Fluid: weak correlation (r = 0.123)
Key takeaway:

Dispersants strongly influence thickening time; fluid loss additives strongly control free fluid and moderately affect thickening time

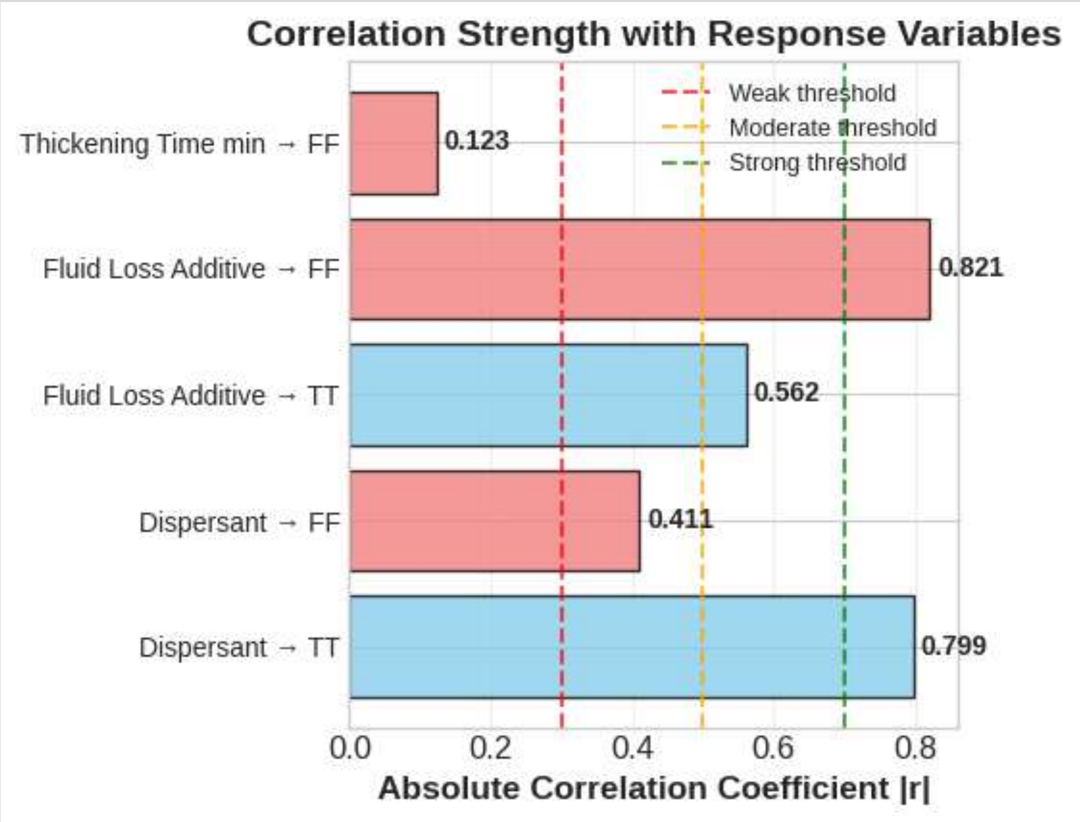

Figure .8: Strength of independent variables with response variables

Figure 8 explain the correlation:

a. Thickening time and free fluid have a weak correlation (r = 0.123), suggesting they're influenced differently and can be controlled somewhat independently.
b. Scatter plots show:
c. Dispersant-thickening time: strong positive linear trend.
d. Fluid loss additive-thickening time: moderate positive, some non-linearity.
e. Dispersant-free fluid: moderate positive, some scatter.
f. Fluid loss additive-free fluid: strong negative linear trend.
g. Key takeaways: Dispersants mainly control thickening time; fluid loss additives mainly control free fluid. There are trade-offs between objectives like thickening time vs stability

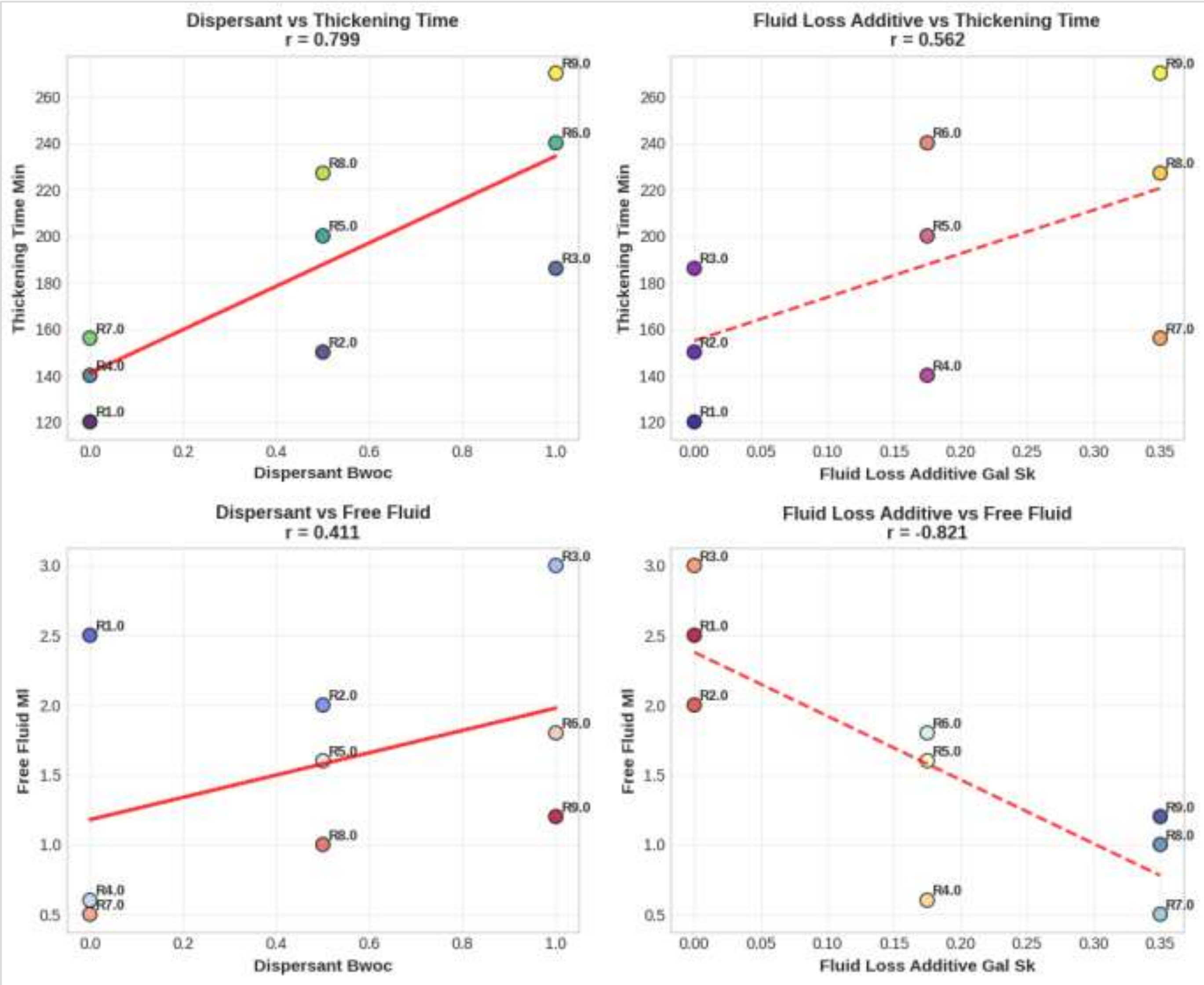

Figure 9: Plot showing relationship between cement slurry response variables and independent variables

Significance Difference in Thickening Time and Free Fluid due to Dispersant and Fluid Loss Additives

a. ANOVA shows dispersant and fluid loss additives significantly impact thickening time and free fluid.
b. Model performance is excellent:
c. $R^2 = 0.981$ (explains 98.1% of variance)
d. Adjusted $R^2 = 0.970$
e. RMSE = 8.731 minutes (~4.7% of mean thickening time)
f. Model assumptions are met (minimal autocorrelation, Durbin-Watson = 2.051)

Figure 10 and Table 5 show:

a. Dispersant concentration has a strong, linear effect on thickening time ($F = 37.835$, $p = 0.002$).
b. Thickening time increases from 138.7 minutes (0% BWOC) to 232.0 minutes (1.0% BWOC).
c. Effect is substantial: ~93.3 minutes increase per 1.0% BWOC dispersant increase.
d. Relationship is statistically significant and linear, making it useful for predictive modeling

Table 4: Goodness of fit

Observations	9.000
Sum of weights	9.000
DF	5.000

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R^2	0.981
Adjusted R^2	0.970
MSE	76.233
RMSE	8.731
DW	2.051

Table 4.5: Analysis of Variance between the thickening time and the dispersant and fluid loss additive factors

Source	DF	Sum of squares	Mean squares	F	Pr > F
Dispersant (% BWOC)	1	2884.267	2884.267	37.835	0.002
Fluid Loss Additive (gal/sk)	1	1041.667	1041.667	13.664	0.014
Dispersant (% BWOC)*Fluid Loss Additive (gal/sk)	1	576.000	576.000	7.556	0.040

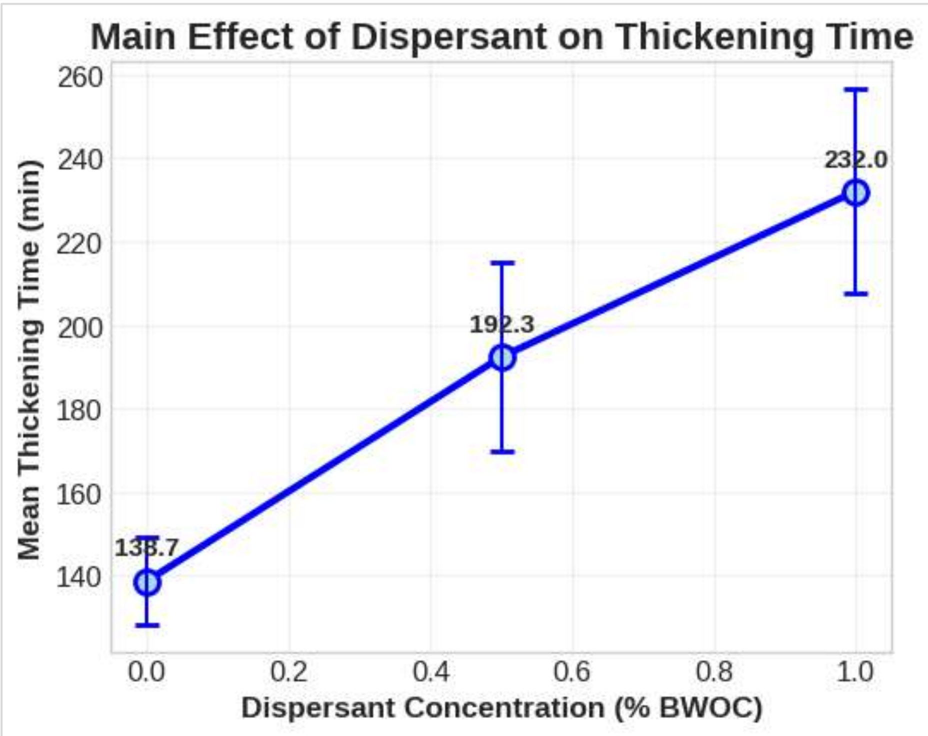

Figure .10: Main effect plot of thickening time and dispersant additive

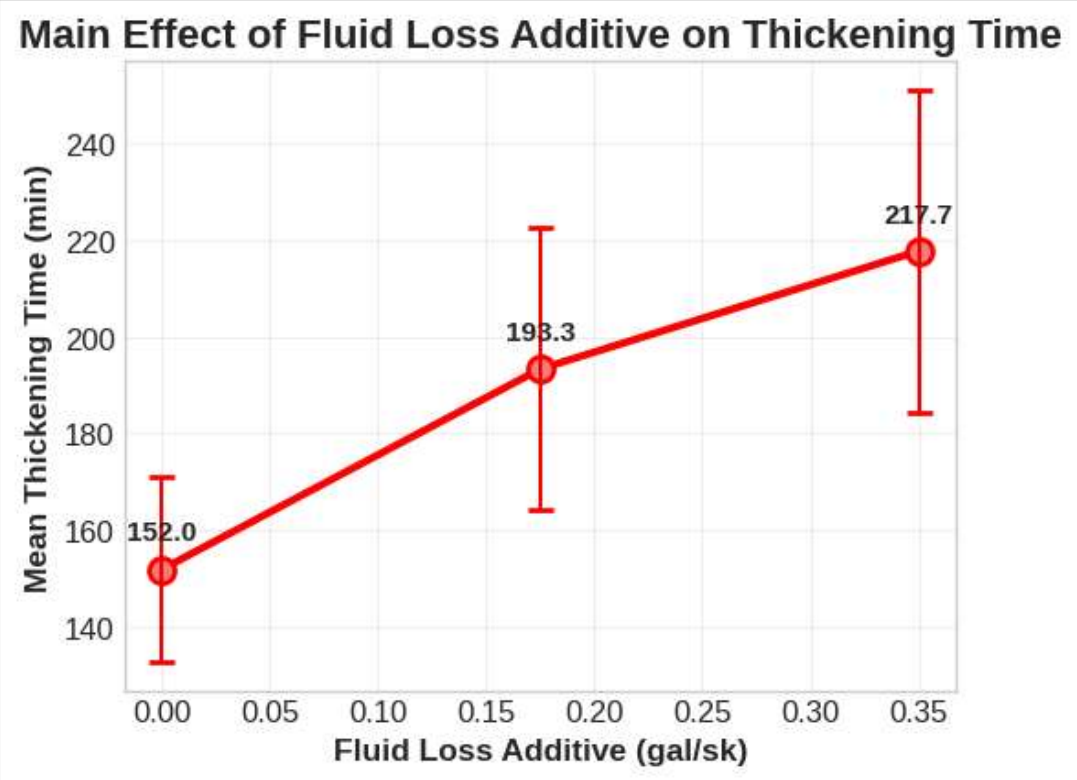

Figure 11: Main effect plot of thickening time and fluid loss additive

Figure 11 and Table 5 show:

a. Fluid loss additive concentration has a moderate, positive effect on thickening time (F = 13.664, p = 0.014).
b. Thickening time increases from 152.0 minutes (0 gal/sk) to 217.7 minutes (0.35 gal/sk).
c. Effect is secondary to dispersant influence, with ~65.7 minutes increase per 0.35 gal/sk increase.
d. Fluid loss additives provide supplementary control over thickening time

Figure 12 and Table 5 show:

a. Dispersant and fluid loss additives interact significantly (F = 7.556, p = 0.040).
b. Fluid loss additives have modest effect without dispersants, stronger effect with moderate dispersants.
c. Interaction is synergistic: combined effects ≠ sum of individual effects

At 1.0% BWOC dispersant:

a. Fluid loss additives have strongest effect on thickening time (185 minutes → 270 minutes).
b. Synergistic interaction: fluid loss additives work better with high dispersant concentrations .
c. Non-parallel lines in interaction plot confirm significant interaction (p = 0.040).

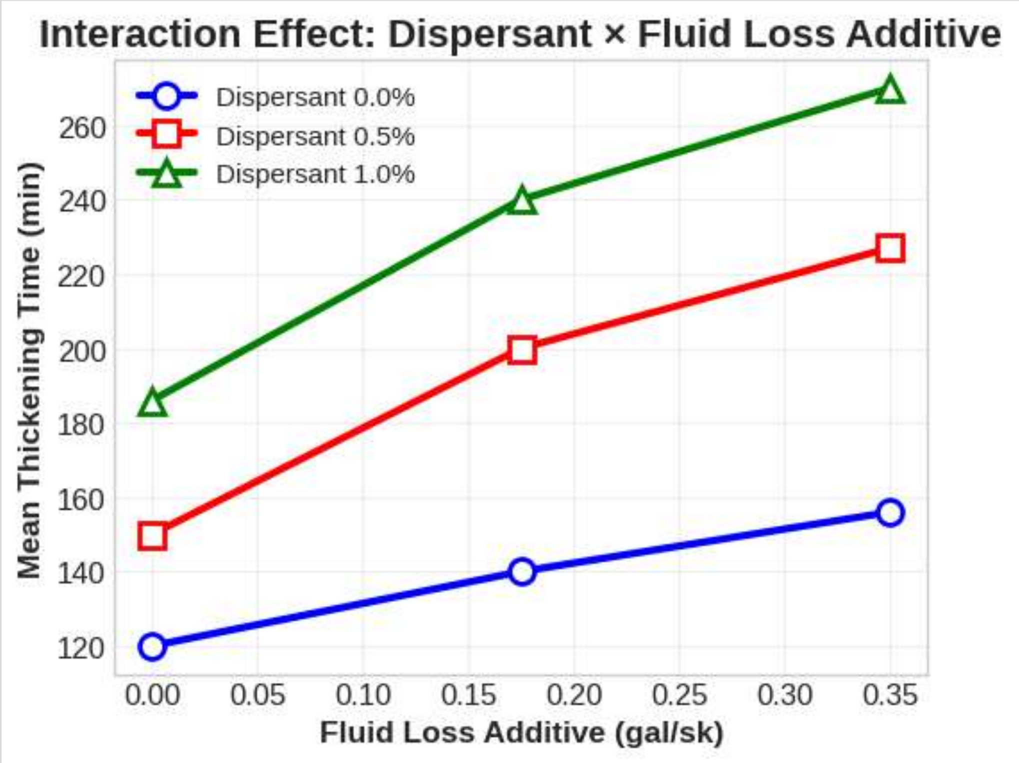

Figure 12: Interaction effect plot of thickening time and dispersant additive and fluid loss additives

Modelling the relationship between the response variables (thickening time) and independent variables (dispersant and fluid loss additives)

Model Performance

a. $R^2 = 0.981$ (explains 98.1% of variance)
b. Adjusted $R^2 = 0.970$
c. RMSE = 8.731 minutes (~4.7% of mean thickening time)

Model Significance

F-statistic = 87.935, $p < 0.0001$ (highly significant)

Parameter Estimates

a. Intercept: 120.167 minutes ($p < 0.0001$)
b. Dispersant: +69.333 minutes per % BWOC ($p = 0.002$)
c. Fluid Loss Additive: +162.667 minutes per gal/sk ($p = 0.014$)
d. Interaction: -133.333 minutes per (% BWOC)(gal/sk) ($p = 0.040$)

Table 6: Model Goodness of fit

Observations	9.000
Sum of weights	9.000
DF	5.000
R^2	0.981
Adjusted R^2	0.970
MSE	76.233
RMSE	8.731
DW	2.051

Table .7: Analysis of Variance for Model suitability

Source	DF	Sum of squares	Mean squares	F	Pr > F
Model	3	20110.833	6703.611	87.935	< 0.0001
Error	5	381.167	76.233		
Corrected Total	8	20492.000			

Computed against model Y=Mean(Y)

Table 8: Model Parameters

Source	Value	Standard error	t	Pr > |t|	Lower bound (95%)	Upper bound (95%)
Intercept	120.167	7.276	16.516	< 0.0001	101.462	138.872
Dispersant (% BWOC)	69.333	11.272	6.151	0.002	40.356	98.311
Fluid Loss Additive (gal/sk)	119.048	32.205	3.697	0.014	36.254	201.841
Dispersant *Fluid Loss Additive	137.143	49.892	2.749	0.040	8.880	265.406

Key Findings

a. Dispersant has strongest influence on thickening time (std coeff = 0.593).
b. Fluid loss additive has moderate influence (std coeff = 0.356).
c. Significant interaction between additives (std coeff = 0.335, p = 0.040).
d. Model predicts thickening time accurately across range (R^2 = 0.981, RMSE = 8.731 minute

Table 9: Standardized model parameters

Source	Value	Standard error	t	Pr > |t|	Lower bound (95%)	Upper bound (95%)
Dispersant (% BWOC)	0.593	0.096	6.151	0.002	0.345	0.841
Fluid Loss Additive (gal/sk)	0.356	0.096	3.697	0.014	0.109	0.604
Dispersant*Fluid Loss Additive	0.335	0.122	2.749	0.040	0.022	0.649

4. Conclusion

Evaluating the Impact of Dispersants and Fluid Loss Additives on Cement Slurry Thickening Time: Experimental Design and Statistical Modeling. This study reveals key findings on cement slurry optimization:

1. Dispersant dominance: Dispersant concentration primarily controls thickening time (strong positive correlation, p = 0.002).
2. Fluid loss additive dual role: Controls filtration and influences thickening time (p = 0.014).
3. Synergistic interaction: Dispersant and fluid loss additives interact significantly (p = 0.040); consider combined effects for optimization.
4. Free fluid control: Fluid loss additives effectively maintain slurry stability (strong negative correlation, r = -0.821).
5. Predictive models: Developed models accurately predict thickening time and free fluid formation (R^2 = 0.981).
6. Methodology effectiveness: Factorial design enables systematic optimization and predictive modeling of cement slurry system.

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