Experiment 1 — Structured R&D ELN Record (DoE Batch Study)

Focus: Fragrance Solubilization / Foam-Control Trade-off
Project: CLEARSCALP Sulfate-Free Shampoo Initiative
Experiment ID: CS-221-A07 (DoE Set 1)
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Objective

Evaluate the impact of **PEG-40 Hydrogenated Castor Oil (HCO) concentration and addition strategy** on:

- Fragrance clarity/stability (eucalyptus oil system)
- Processing foam
- Final viscosity and clarity retention

Experimental Design (Parallel DoE)

A single master batch was prepared and split into **three identical 1.0 kg sub-batches** after surfactant hydration. All three were processed in parallel using identical equipment sets.

Prototype	PEG-40 HCO (%)	Fragrance Addition Strategy	Shear Strategy
A (Control)	1.5%	Direct addition into main vessel	Moderate shear only
B (High Solubilizer)	3.0%	Pre-mixed + homogenized premix	High shear incorporation
C (Process Control)	1.5%	Fragrance-in-solubilizer premix	Split shear (low → high)

Formula Summary (Base System — Identical for all prototypes)

Ingredient	Function	Target %
DI Water	Carrier	q.s.

Ingredient	Function	Target %
Sodium Lauroyl Sarcosinate	Primary Surfactant	14.0
Cocamidopropyl Betaine	Secondary Surfactant	7.0
Hydroxyethylcellulose	Rheology Modifier	0.7
Citric Acid (20%)	pH Adjuster	variable
Eucalyptus Oil Blend	Fragrance	0.8

Equipment

Three identical processing lines were used in parallel:

Equipment	IDs
Silverson Homogenizer	MIX-04 / MIX-05 / MIX-06
Overhead Stirrer	STR-11 / STR-12 / STR-13
Brookfield DV2T	VIS-08
pH Meter	PH-03

Procedure Overview (Shared Upstream Processing)

Phase 1 — Hydration (Common Batch, then Split)

08:10–08:45

- DI water charged into 6 L vessel
- Hydroxyethylcellulose dispersed under 250 rpm agitation
- Hydration completed at 68°C

Observation: minor initial lumping resolved within 12–14 min across system

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Phase 2 — Surfactant Build (Common Base)

09:00–09:40

- Sodium Lauroyl Sarcosinate added under controlled agitation (180–220 rpm)

- CAPB added post-clear transition

Result:

- Single homogeneous translucent base achieved
- Base viscosity uniform prior to split (±2% variance)

Phase 3 — Split and Parallel Prototype Processing

Base was split equally into three jacketed 2 L vessels (A, B, C) at 52°C.

Prototype A — Low Solubilizer / Minimal Process Change

09:55–10:35

- PEG-40 HCO (1.5%) added directly into main vessel
- Fragrance added without premix
- Mixed at 2500 rpm (Silverson) for 3 min

Observations:

- Immediate transient haze
- Partial recovery after 20 min mixing
- Foam generation moderate

Prototype B — High Solubilizer / High Shear Design

09:55–10:35 (parallel run)

- PEG-40 HCO (3.0%) + fragrance pre-mixed
- Homogenized at 4000 rpm for 90 sec prior to addition
- Incorporated under 3500 rpm for 4 min

Observations:

- Fully transparent premix prior to addition
- Minor clouding upon addition (resolved within 3–5 min)
- Highest foam generation during processing

Prototype C — Split Strategy (Low → High Shear Transition)

09:55–10:35 (parallel run)

- PEG-40 HCO (1.5%) used in fragrance premix
- Premix added under low shear (1500 rpm)
- Followed by 2 min high shear polish step (3200 rpm)

Observations:

- No immediate haze on addition
- Slight turbidity during low shear phase
- Clarity fully restored only after high shear step
- Lowest foam generation of all prototypes

Phase 4 — Cooldown & pH Adjustment (All Samples)

10:40–11:30

- Cooled to 34°C under slow agitation
- pH adjusted to 5.4–5.6 range using citric acid

Results Summary

Parameter	A (Control)	B (High Solubilizer)	C (Process Control)
Final Appearance	Slight haze	Clear	Clear
Time to Clarity	Partial / delayed	Fast recovery	Dependent on shear step
Foam Generation	Medium	High	Low
Viscosity (cP)	5,600	5,850	5,700
Stability @ 40°C (24h)	Slight opalescence risk	Stable clear	Stable clear
Processing Robustness	Moderate	Sensitive to foam	Most robust

Conclusion

1. Increasing PEG-40 HCO to 3% (Prototype B) provides the most robust **thermodynamic solubilization**, but introduces **high process foam burden**.
2. Prototype C demonstrates that **process design (shear sequencing)** can compensate for lower solubilizer loading while maintaining clarity.
3. Prototype A confirms that 1.5% PEG-40 HCO is insufficient under direct addition conditions.

Decision / Next Study Direction

- Advance **Prototype C-type process architecture** for scale-up screening
- Investigate **foam suppression via surfactant order modulation rather than solubilizer increase**
- Consider hybrid condition: 2.0% PEG-40 HCO + split-shear addition profile