Shell Chemicals

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Reconstitutable Liquid Laundry Detergents for Reduced Environmental Impact

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Reconstitutable Liquid Detergent – Contents

- Concept and advantages
- Approach: Dissolution of concentrates
- Formulation routes to faster reconstitutability
- Laboratory test: Relevance to consumer experience
- Conclusions

Reconstitutable Liquid Detergent – Concept and Advantages

- Background: Liquid detergents continue to grow globally and are becoming the form of choice in many markets

- Reconstitutable concept:
  - Consumer adds concentrate to re-usable bottle, dilutes (e.g. at 1:1 or 1:2) with tap water at home
    - Final product dosed at similar volume to regular HDLs
    - Concept similar to fabric softeners

- Advantages:
  - Reduced environmental impact (from packaging and transportation of liquids)
  - Reduced costs associated with the above
  - Accurate dosing by consumer

Reconstitutable Liquid Detergent – Approach and Requirements

- Approach: Use High Active Matter (HAM) anionic / non-ionic mixtures:
  - Alcohol Ethoxylate (AE)
    - replaces water as the solvent
  - Alcohol ethoxysulfates and alkybenzene sulfonates
    - The acid forms neutralised with concentrated NaOH solution or Triethanolamine (TEA) in the presence of AE
  - Water <10%w

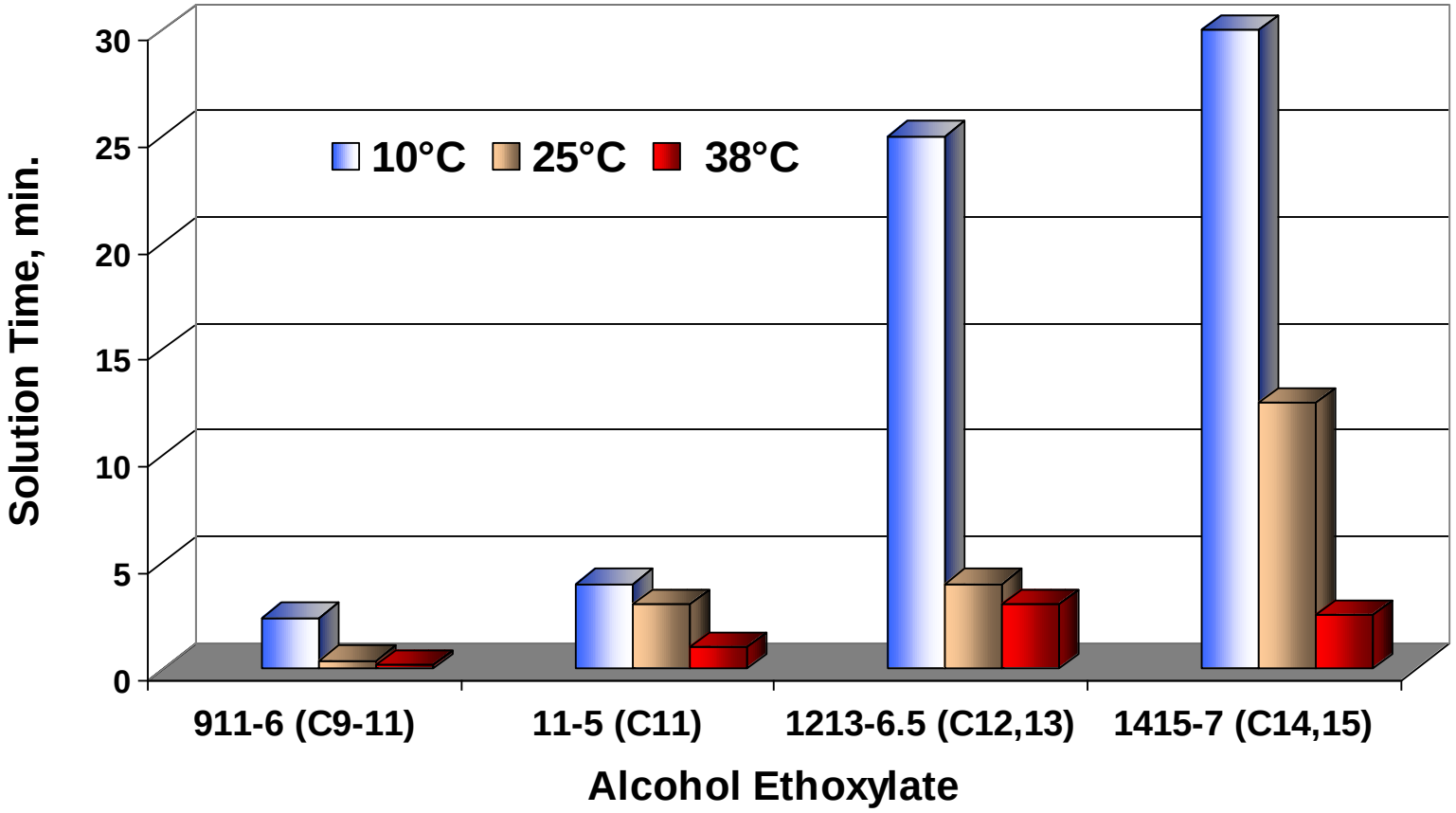
- Requirements:
  - Acceptable cleaning properties
  - Fast dissolution into water down to 10°C and no gel formation in bottle
  - Minimal inert ingredients to minimise packaging and costs

Test Methods

- Dynamic Solution Time Measurement
  - Measures time to fully dissolve surfactant concentrate in water
  - 0.2 ml concentrate added with stirring to 50 ml water at 10, 25 and 38°C
  - Strong formulation dependence
  - Fast dissolution required, target <30 sec at 10°C

- Gel Region Measurement
  - Measures gel / liquid boundary for different water / concentrate ratios and temperatures
  - Temperature is reduced in stages from 70°C

Higher Temperatures and Shorter-Chain Alcohol Ethoxylates Yield Faster Dissolution Times



- ✓ Alcohol Ethoxylates: Modified OXO process alcohols (80% linear, 20% branched on C2 carbon) with different EO contents
- ✓ AE 911-6 gives <20 sec solution time at 25 and 38 °C

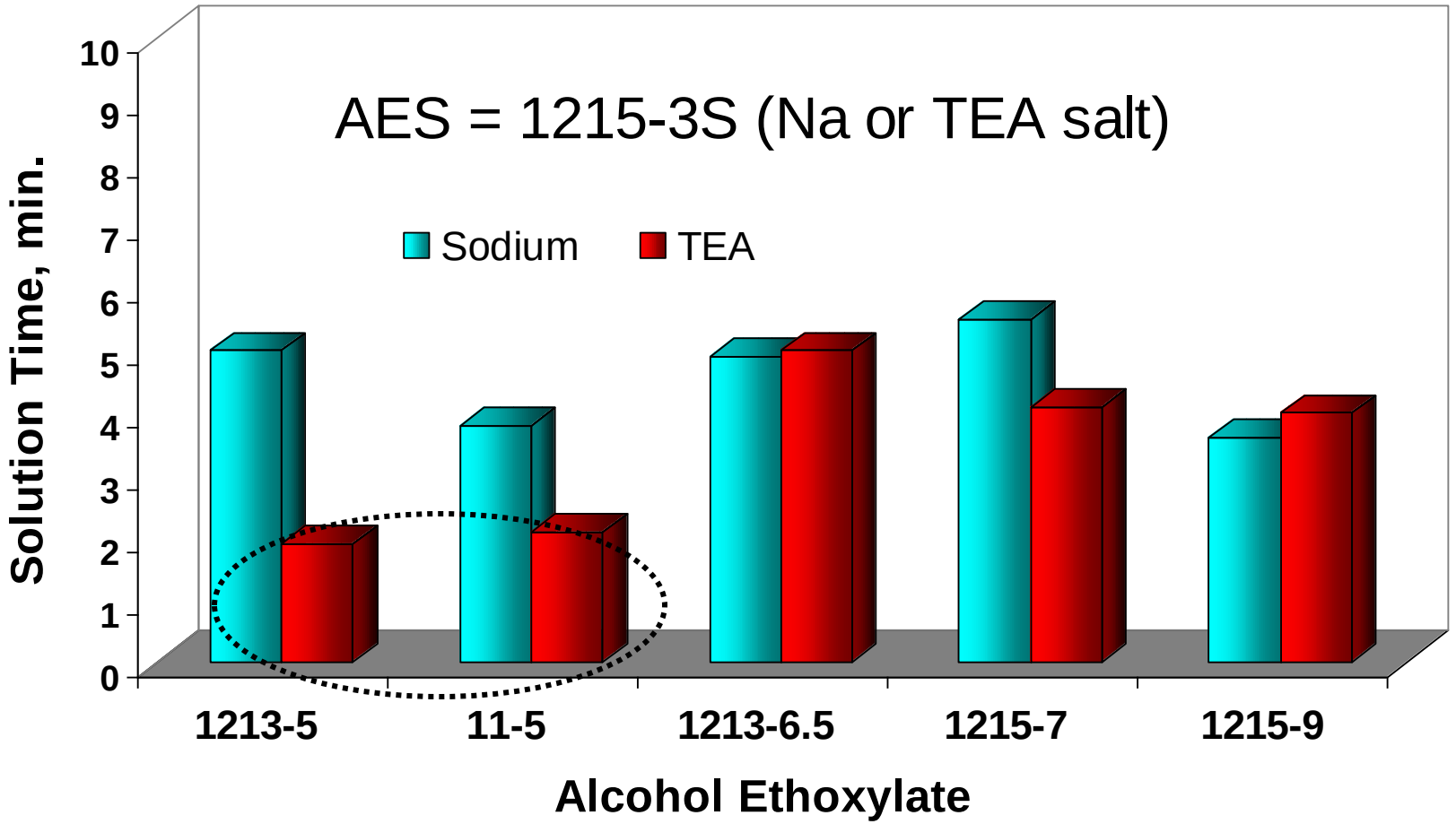
Test Formulation Concentrates

With LAS :	Without LAS:
60% AE	60% AE
17.5% AES (Na or TEA salt) 17.5% C12 LAS (Na or TEA salt)	35% AES (Na or TEA salt)
5% Water	5% Water

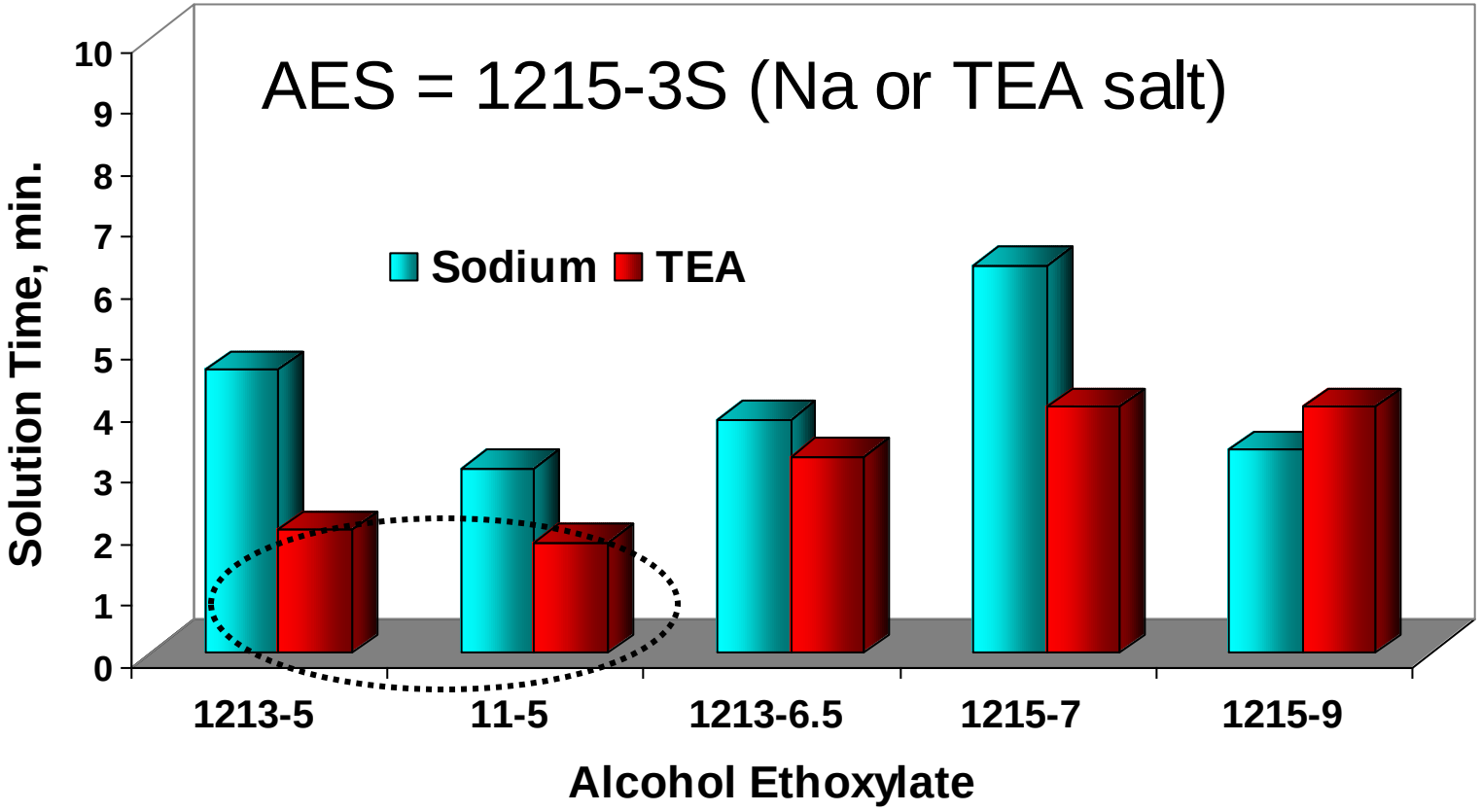
LAS = Linear alkylbenzene sulfonate
AE = alcohol ethoxylate
AES = alcohol ethoxysulfate
TEA = triethanolamine (a cation known to reduce/prevent gels)

NOTE:
1) Both formulations representative of US-type,1/8 cup, unbuilt HDLs
2) Alcohol Ethoxylates: modified OXO process alcohols, with EO content of 5-9

Solubility (at 25°C) of LAS-free Formulations:
Shorter chain, 5-mole Ethoxylates with TEA are best



Solubility (at 25°C) of LAS-containing Formulations:
Shorter chain, 5-mole Ethoxylates with TEA are best



- ✓ NOTE: When AES = 1213-6.5S same trends seen as above, with / without LAS

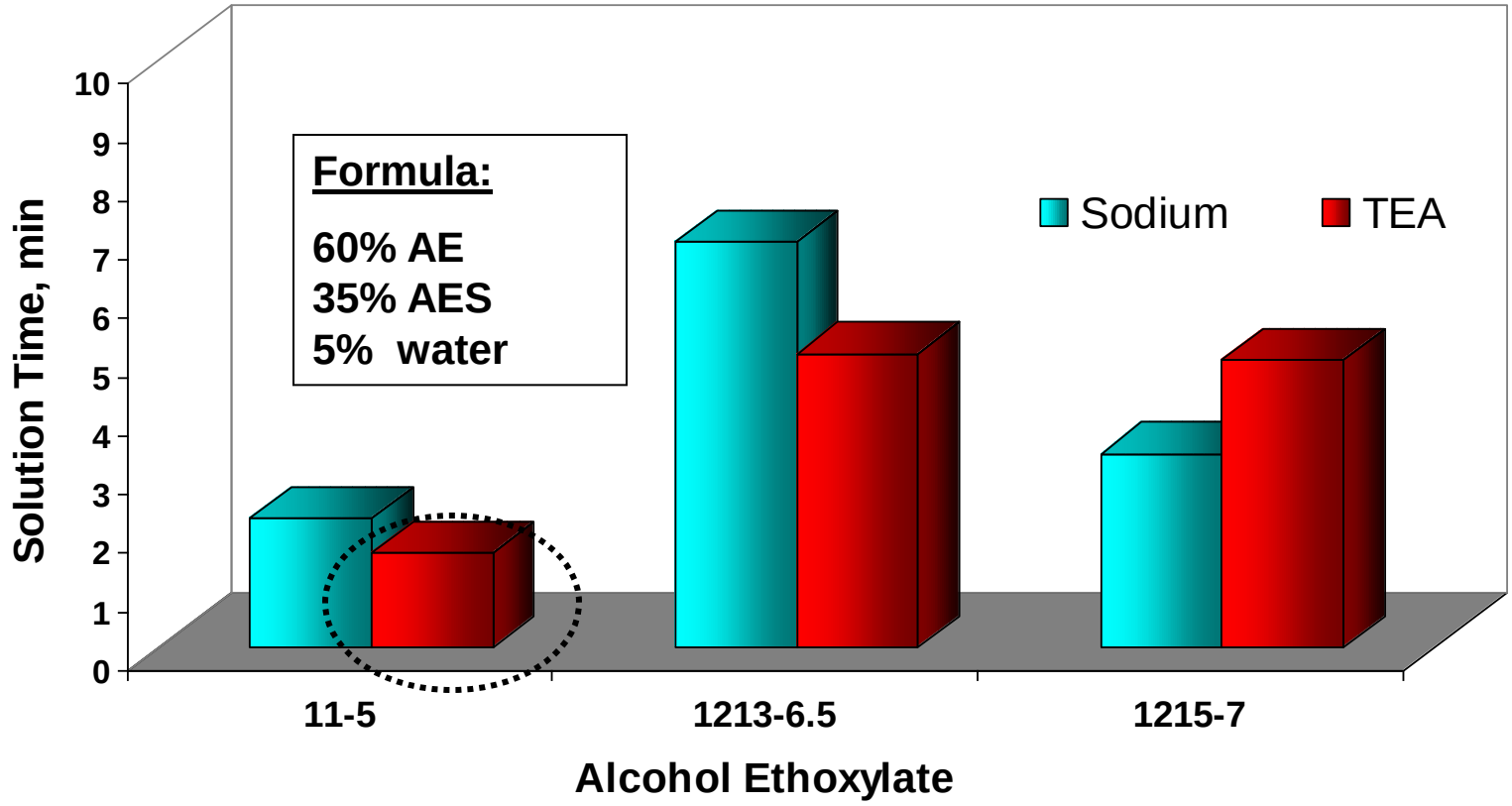
Routes to Faster Reconstitutability

- AE: Alcohol chain length and EO effect
  - Shorter chain alcohols with 5 mole EO best
- AES: Cation effect
  - TEA better than Na

- Now, investigate:
  - Effect of AES and AE components based on the same AE
  - Effect of additives such as ethanol and monoethanolamine (MEA)

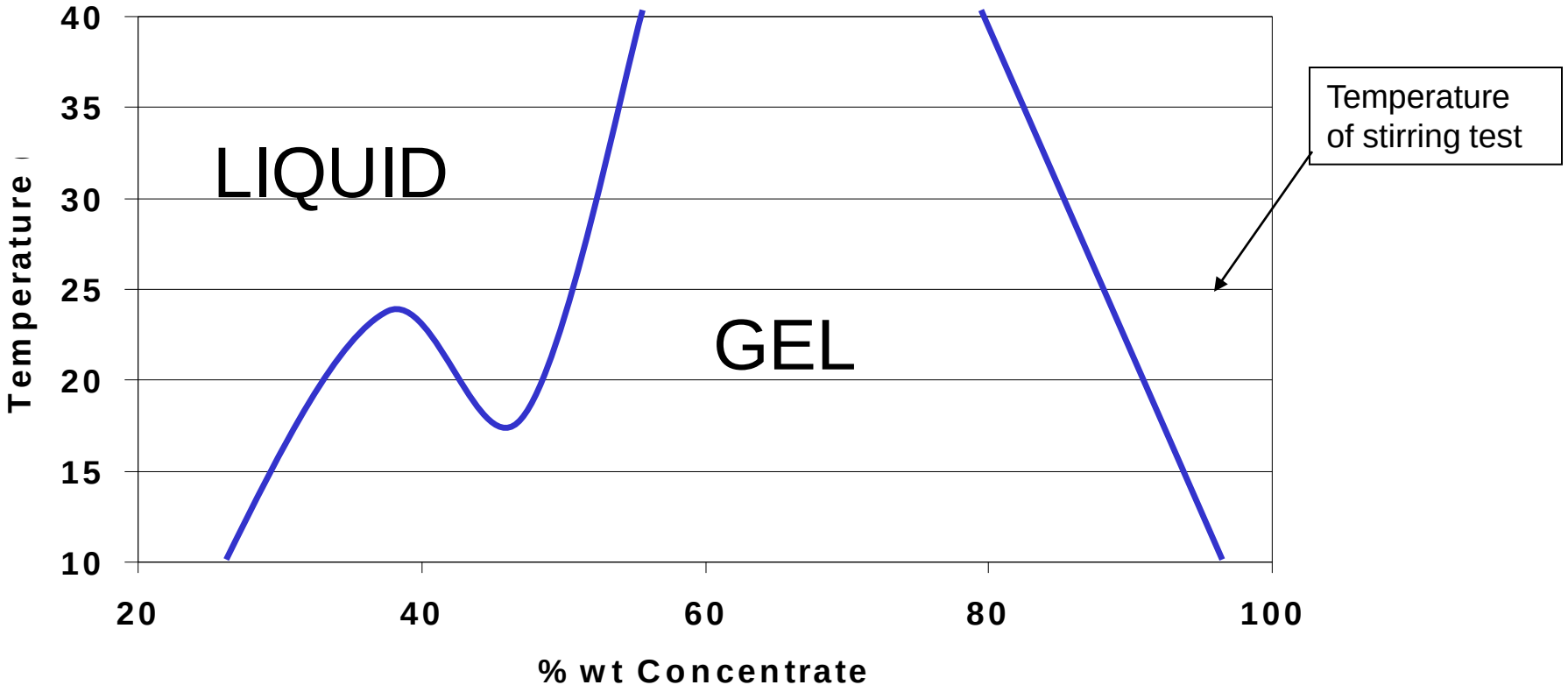
Routes to Faster Reconstitutability:

For AES and AE based on same AE, Short Chain 5-mole Ethoxylates Yield Fastest Dissolution Times at 25°C



- ✓ Similar trend seen with LAS-containing formulations
- ✓ In 5 out of 6 cases, TEA salt gave best solubility
- ✓ 1-2 min. solution times still too long ! Need to explore additives

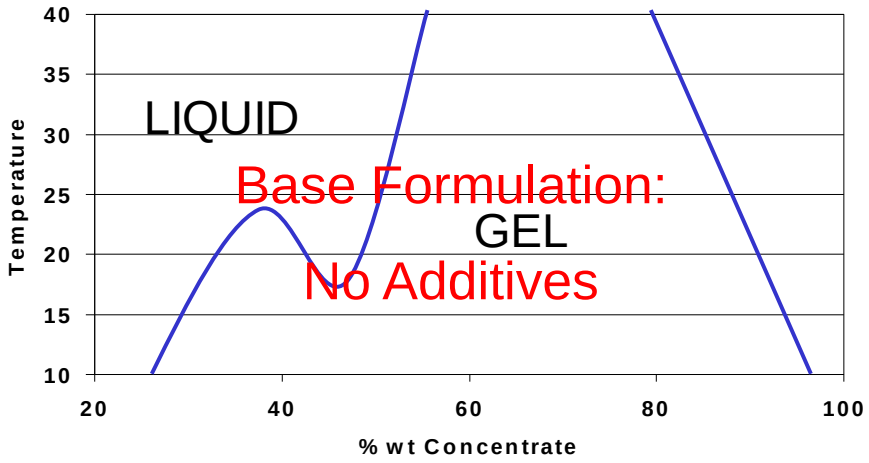
Routes to Faster Reconstitutability:
Base formulation has significant gel region



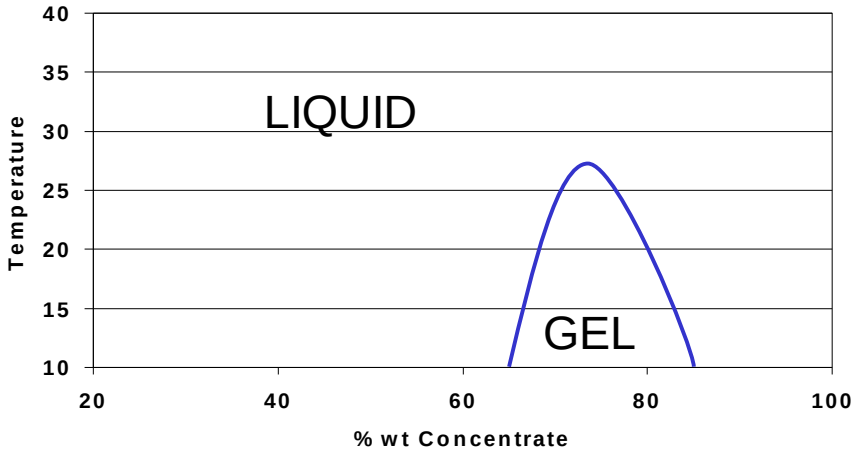
Base formulation (no additives):

60% AE 11-5, 17.5% AES 11-5S(TEA), 17.5% C12LAS (TEA)

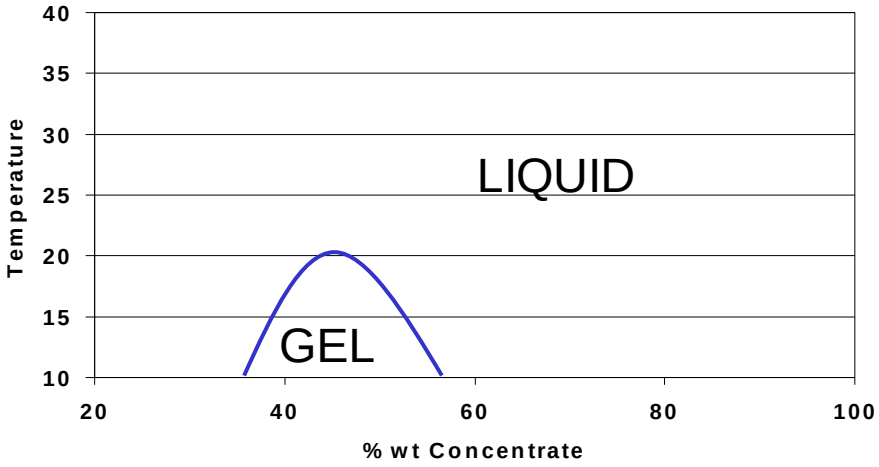
Routes to Faster Reconstitutability:
Use of Additives/Co-solvents Prevents Gels



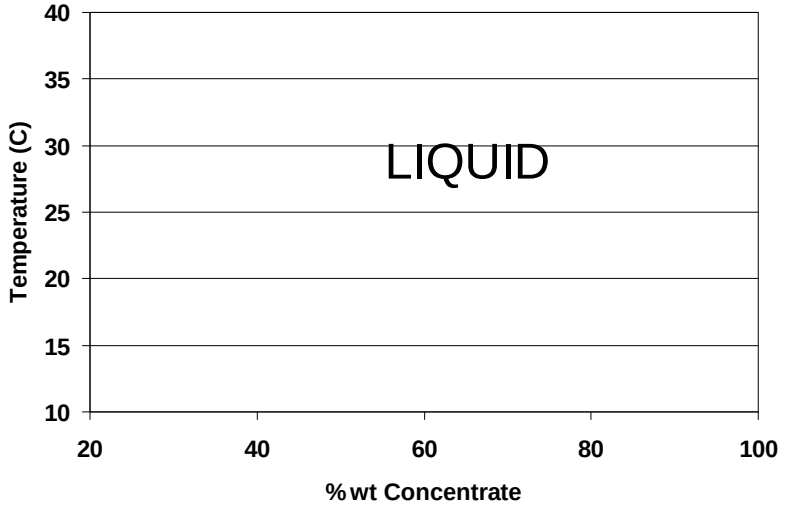
Base formulation



Base: +10% MEA



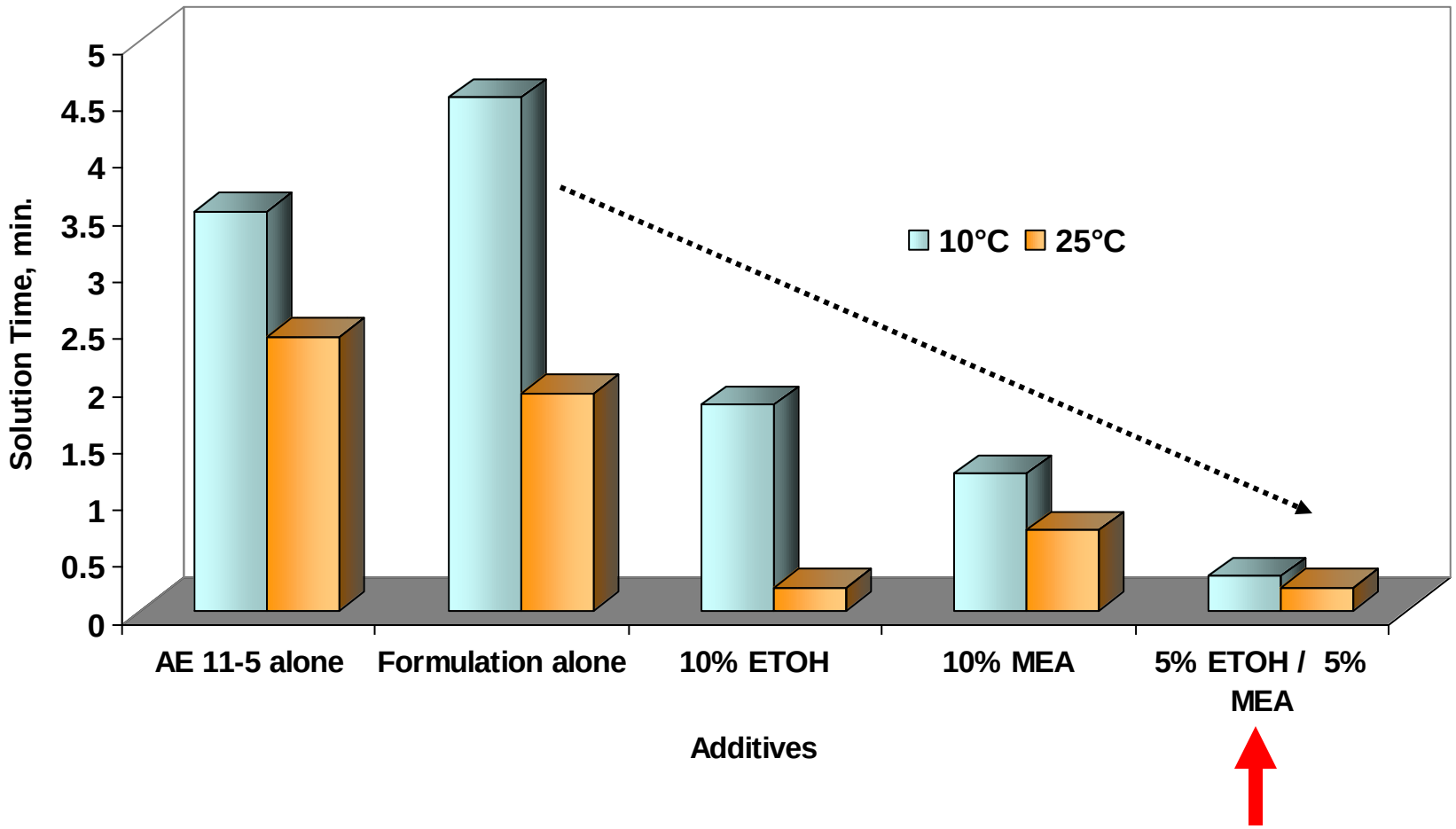
Base: +10% Ethanol



Base: +5% MEA and 5% Ethanol

Best Prototype Formulation with Additives Dissolves Efficiently at Low Temperature

Formulation: 60% AE 11-5; 17.5% AES 11-5S(TEA);17.5% C_{12} LAS(TEA)



20-30 sec achieved

Formulation Limits for Good Reconstitutability*

AES : LAS Ratio	Anionic Surfactant (wt %)	Salt Composition
1 : 1	**40 max**	100% TEA
1.33 max : 1	35	100% TEA
1 : 1	35	**66% TEA, 33% Na max**

*Based on most promising system:
AE 11-5 / AES 11-5S (TEA) and C12 LAS (TEA) with 5% ethanol and 5% MEA.

Formulation flexibility is possible, but slower dissolution when:

- ✓ Total % anionic or % AES increased,
- ✓ Levels of additives reduced, or
- ✓ Na salts substituted for TEA salts

Laboratory vs. Consumer Solution Times

- The lowest water temperature of 10°C was chosen to represent the coolest (and most difficult) tap water temperature

- The laboratory test (spinning stir bar in a volume of liquid) is rather “mild” and solution times will be long compared with consumer experience with reconstitutability

- A consumer shaking a concentrate with water would experience good mixing *within several shakes*, if the formula takes 20-30 seconds to dissolve in the laboratory test

Conclusions: Reconstitutable Liquid Detergents

- A consumer reconstitutable, unbuilt, formulation successfully developed
- Advantages, compared to dilute, direct-use formulations:
  - Reduced environmental impact and costs
  - Maintain ease of use and accurate dosing by the consumer
- Use of MEA + ethanol as additives allows rapid dilution with cold water
  - The practical formulation identified contained a short carbon chain AE 11-5, TEA salt of the AES, and the TEA salt of C12LAS
- Flexibility in composition is possible, within certain constraints
- Enzyme stability is a potential benefit of the reconstitutability approach:
  - Although not studied, undiluted (low water) concentrates expected to give improved long-term stability

Acknowledgements

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Thank you for your attention