SAND2007-3685C

Foam Processing Studies for Physically Blown Epoxy Foams

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37th PolyMAC Conference, June 19, 2007

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Processing of foam encapsulants used in a system upgrade

- REF308, REF320, and EF-AR20 utilize Fluorinert FC-72 electronic fluid as physical blowing agent
  - FC-72 boiling point = 56°C
  - Must be uniformly dispersed in foam mixture
  - Foam made with 2-part kits
    - Part A = resins
    - Part B = curing agents, **FC-72 blowing agent**, surfactant, Cab-o-sil M5
      - Vigorously shaken to disperse FC-72 in Part B just prior to mixing foam
    - Foam formulations documented in material and processing specifications
  - Part A (resin) preheated at 55°C, and Parts A and B are mixed at the appropriate ratio for 1 minute – hand mixing with tongue depressor and KCP "malt mixer" have been used
  - Mixture is poured/injected into mold or container and cured for 2 hours at 65°C, then 22 hours at 75°C
- This work was initiated due to differences seen in REF308 and 320 foams that were hand mixed vs. malt mixed

KCP "malt mixer"

Hand mixing done with tongue depressor spatula

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Initial observation of foam rise vs. foam mixing

- Larger quantities of foam mixed using KCP malt mixer (10-15K rpm), smaller quantities hand mixed with tongue depressor spatula
- Observed in REF 308 and 320 foams that high speed malt mixing resulted in less expansion (higher density) foams than hand mixing
- Blowing agent loss not suspected since no significant loss measured in hand-mixing and blade-mixing experiments
- Suspect that emulsion of FC-72 in foam mixture may be affected by mixing speed/technique
  - FC-72 droplet size?
  - Air entrainment affecting cell nucleation?
  - Lisa Mondy, et. al. investigating
- Hand mixing performing better, but may be more variable (operator dependencies)
- Initiate foam hand-mixing study to investigate variables that affect foam rise, density, etc.

REF320 foams

KCP "malt" mix **Hand mix**

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Operator (4 variations)

Ed

Jim

Morgan

Sarah

Container size (4 variations)

1000 ml plastic cup

500 ml plastic cup

150 ml plastic beaker

100 ml plastic beaker

Mixing speed (4 variations)

~240 rpm

~200 rpm

60 rpm

12 rpm

Standard Foam Processing:

Operator: Patti

Mixing speed: normal (~150 rpm)

Mixing time: 1 minute

Mixing method: stir and scrape

Mixer: tongue depressor

Container: 250 ml plastic beaker

Hand mixing study using EF-AR08 foam to identify variables that affect foam rise, structure, etc.

- **2 samples made for each variable**
- **Samples analyzed, data averaged**

Mixer (5 variations)

Spoon-like spatula

½ width tongue depressor

Tongue depressor, 2 holes

Tongue depressor, 4 holes

Whisk

Mixing method (4 variations)

Fold only

Stir, scrape bottom only

Stir, scrape sides only

Stir, don't contact cup

Mixing time (5 variations)

15 sec

30 sec

90 sec

2 min

3 min

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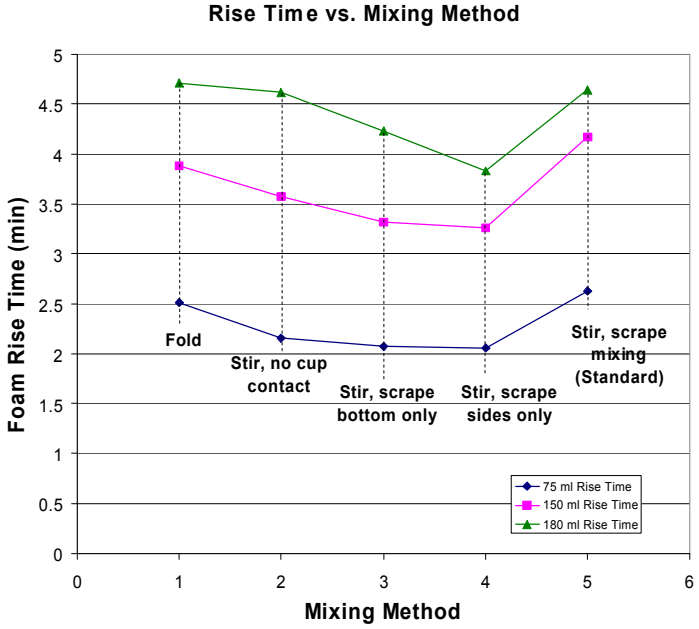
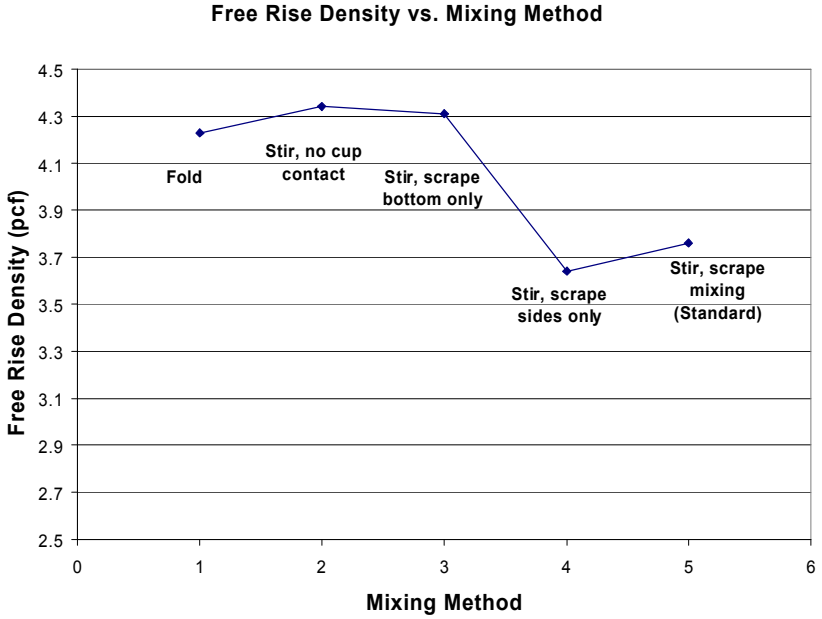
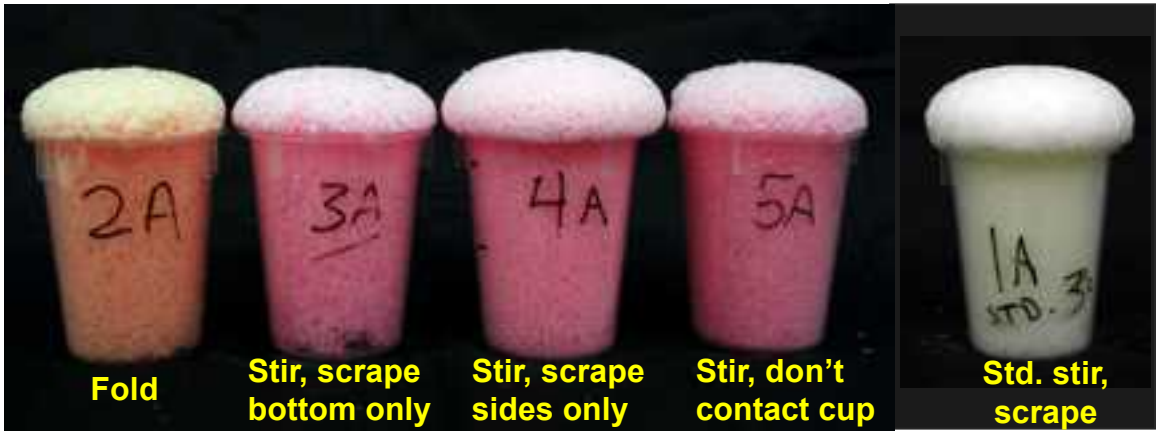
Diagnostics for foam mixing study

- Foam rise time (measured rise time to 75 ml, 150 ml, 180 ml volumes)
- Free rise density measurement
- Microscopy to observe foam cell structure
- Shape of foam cap – no significant changes observed
- Added food coloring dye to observe mixing flow patterns in foams – Part A, Part B, and dye mixed very easily – no flow patterns observed in foams

Vary mixing method – effect on rise time, density







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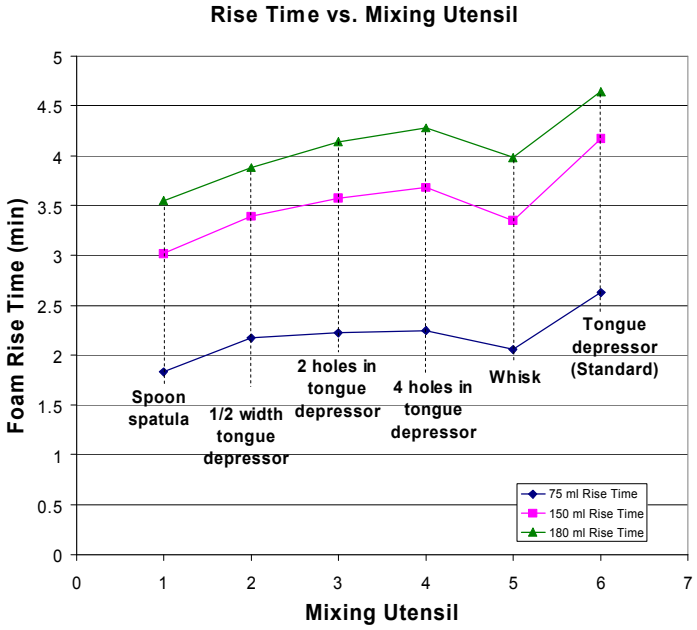
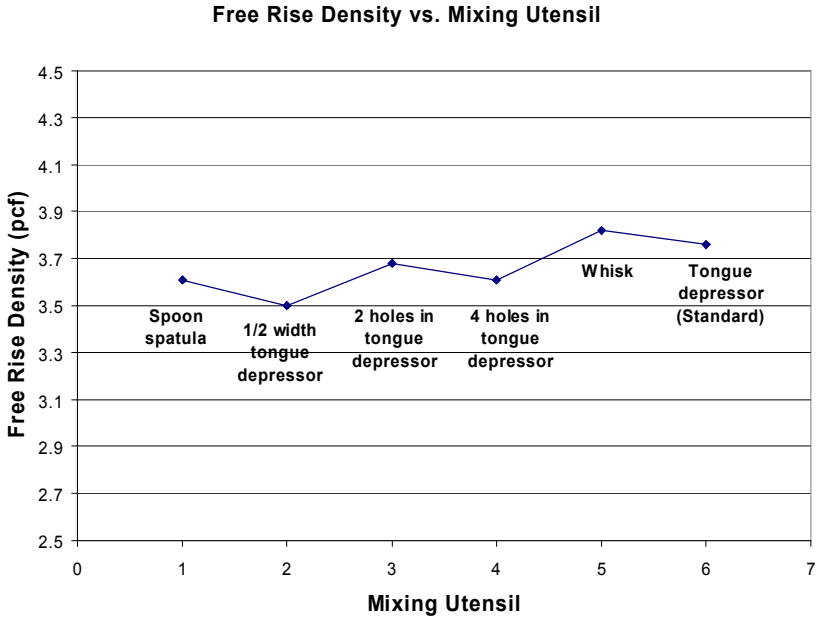
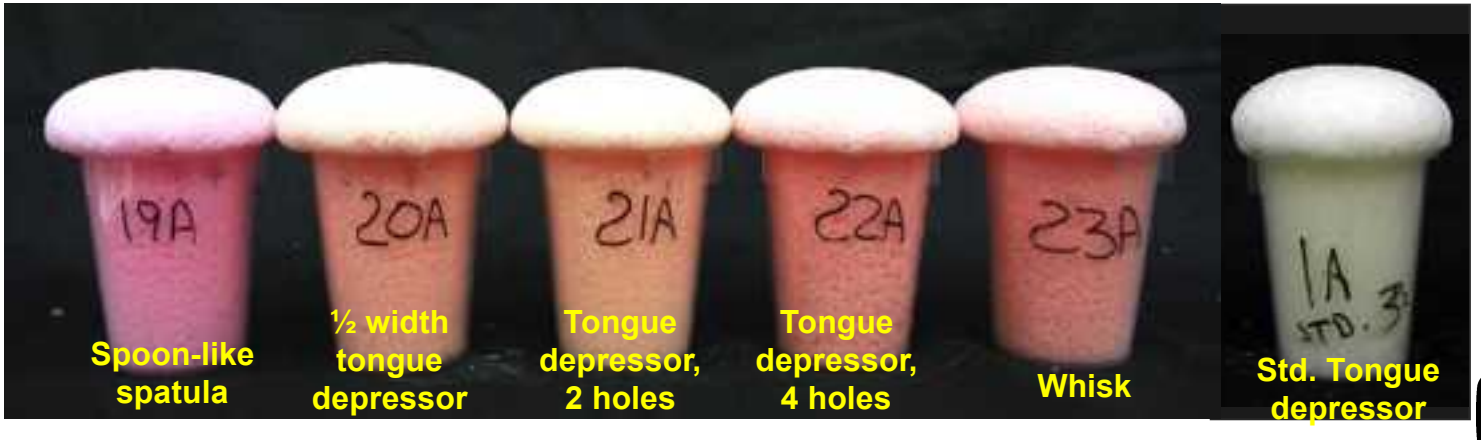
Vary mixing utensil – rise time, density tended to increase with more aggressive mixing utensil







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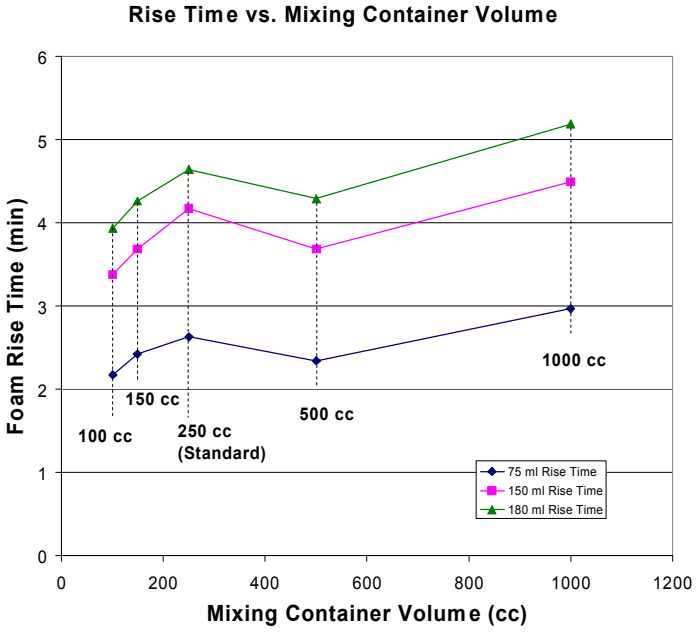
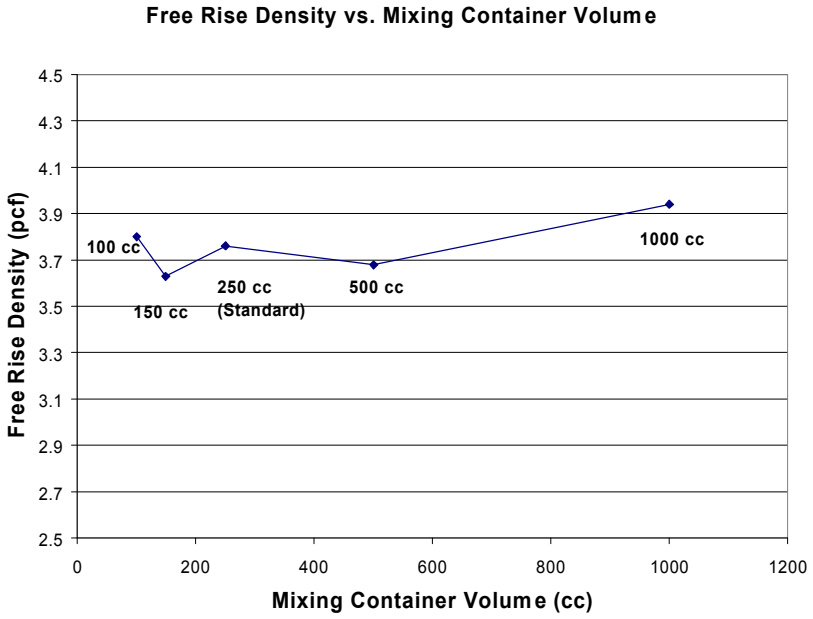
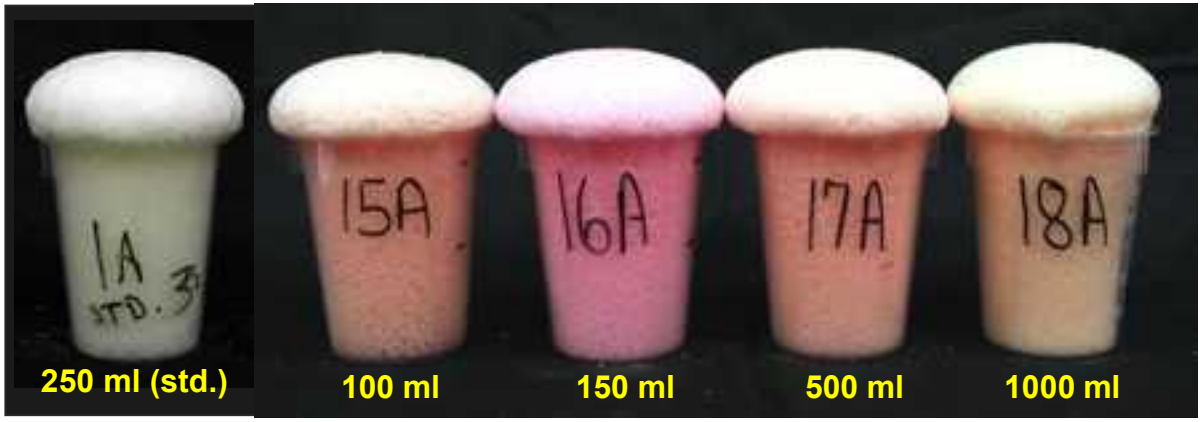
Vary mixing container volume – some effect on rise time, density







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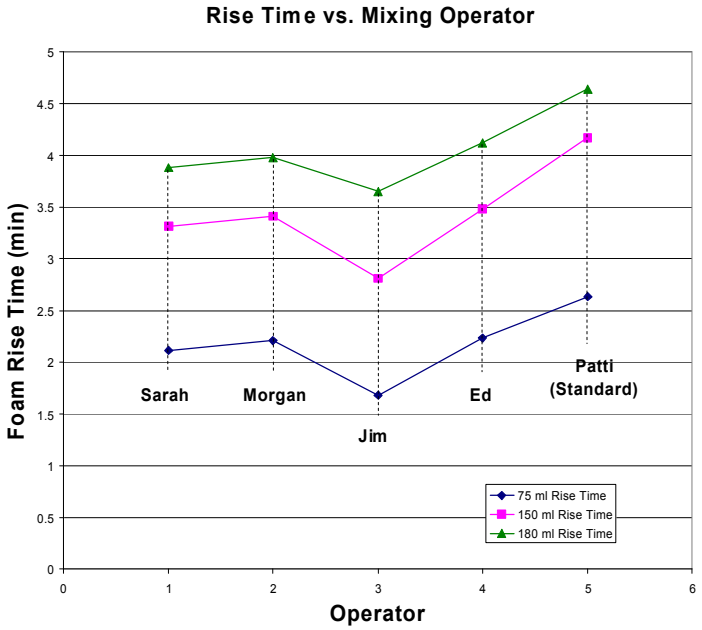
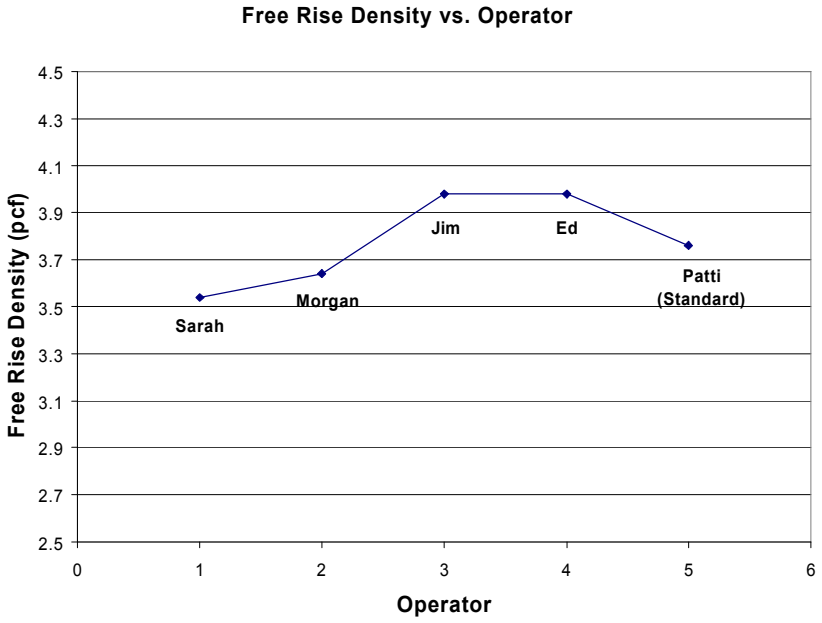
Vary mixing operator – some effect on rise time, density





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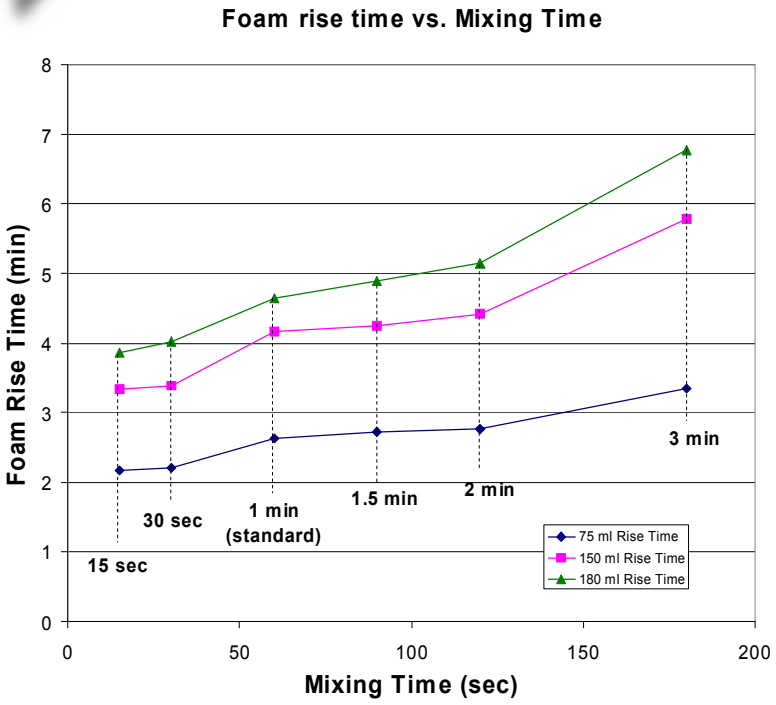
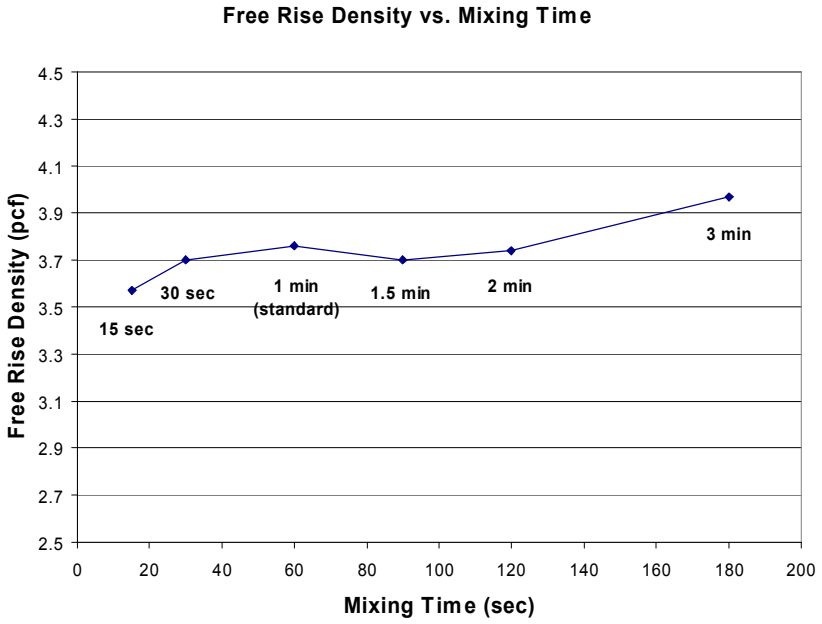
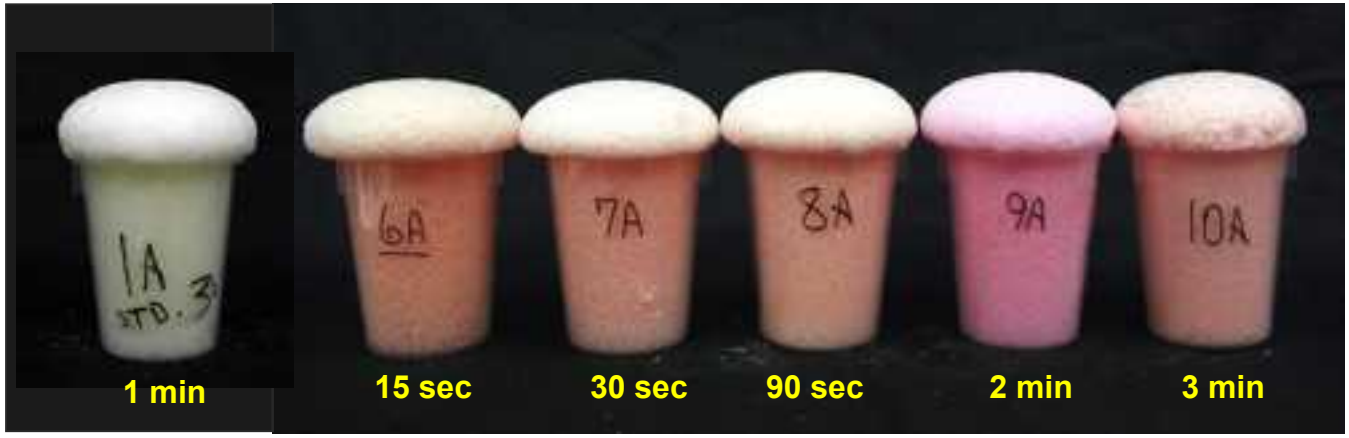
Vary mixing time – rise time, density tended to increase with mixing time







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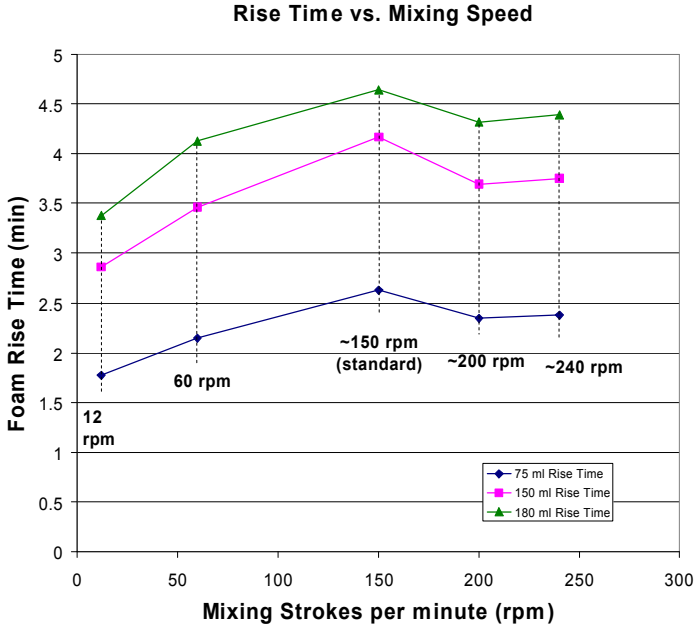
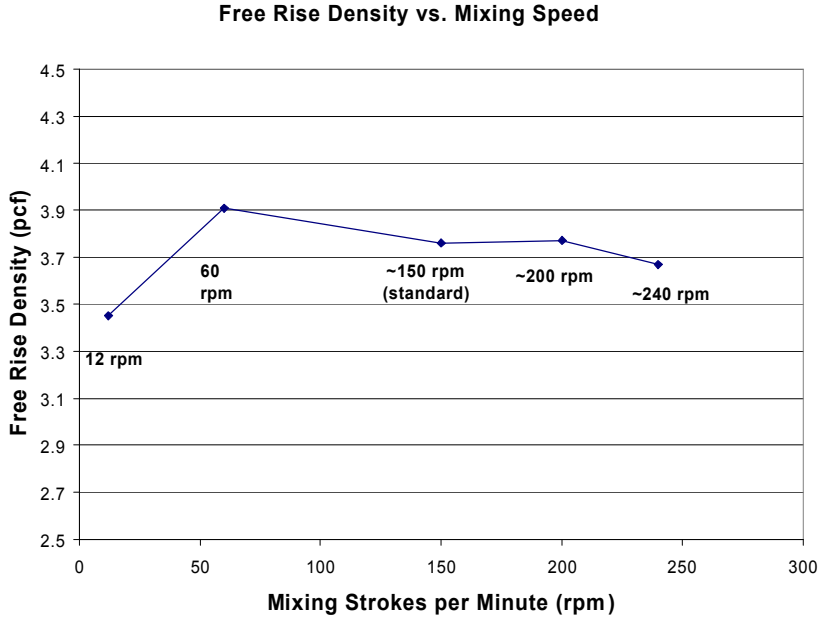
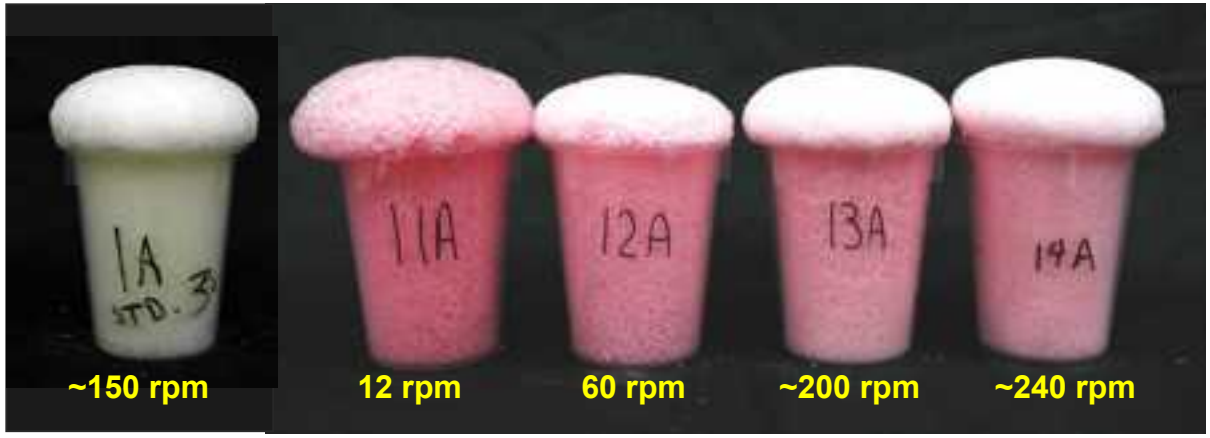
Vary mixing speed – some effect on rise time, density







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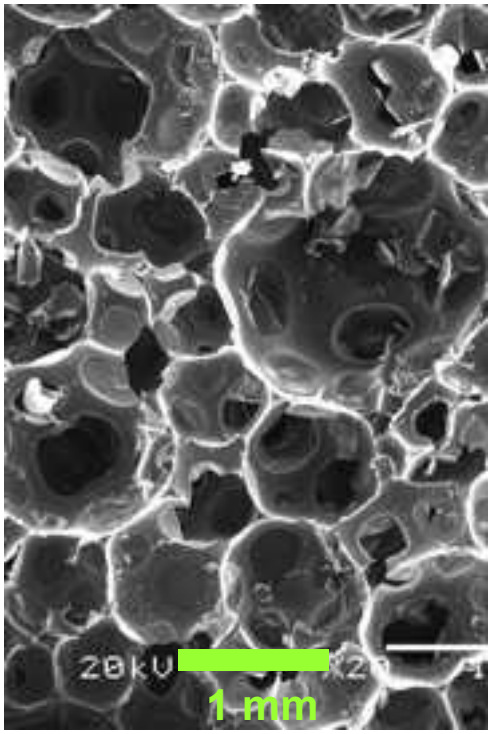
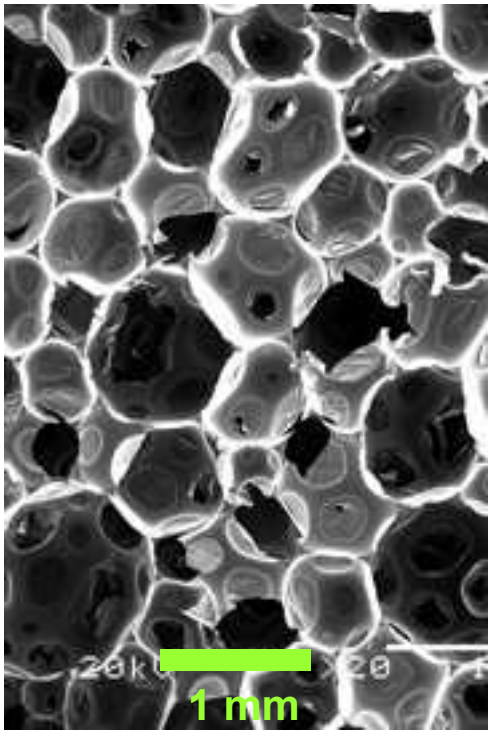
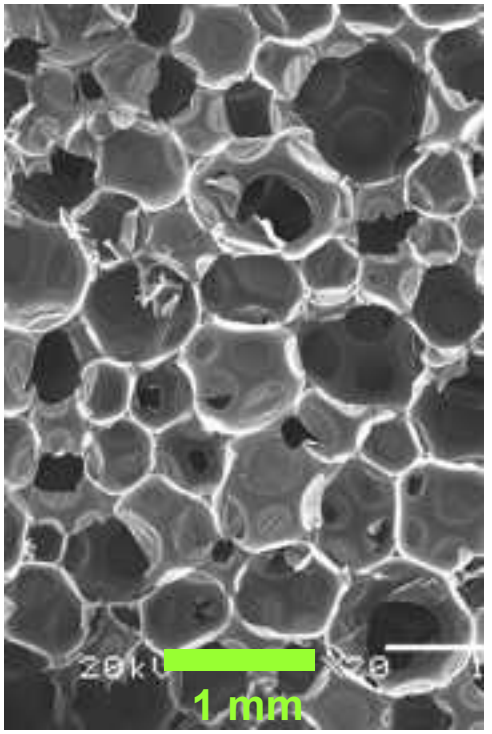
SEM analysis shows that foam cell size tends to be smaller with longer mixing time



15 sec mix time

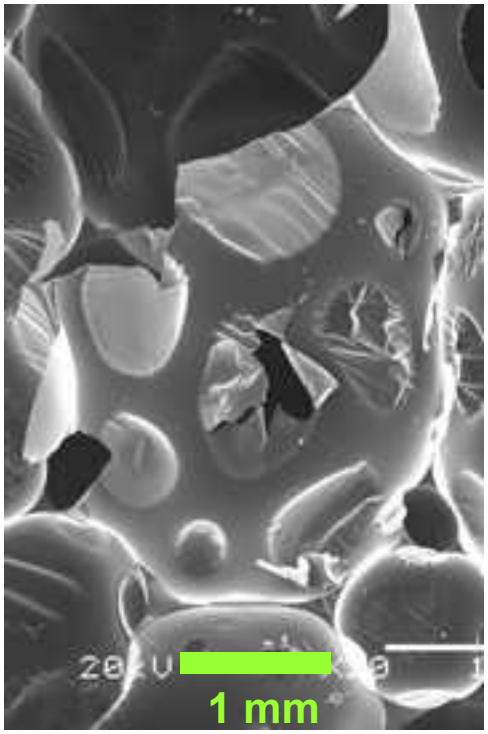


1 min mix (standard)

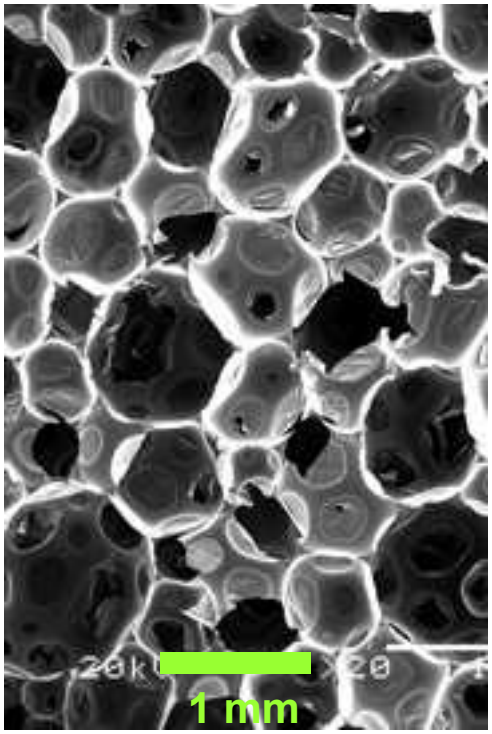


3 min mix time

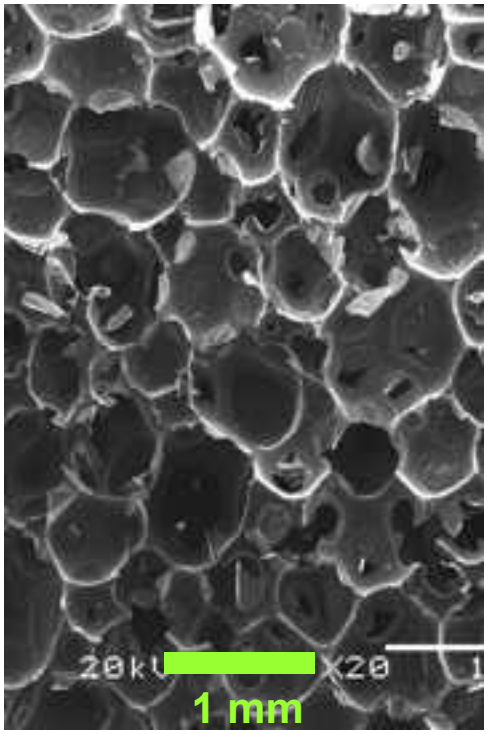
SEM analysis shows that foam cell size tends to be smaller with faster mixing speed



12 rpm mixing speed



~150 rpm (standard) mixing speed



~240 rpm mixing speed

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General trends observed

- Foam rise time tended to increase as:
  - mixing time increased
  - mixing speed increased
  - mixing utensils were used that provided more vigorous mixing
  - larger mixing containers were used that allowed for more thorough mixing
- Foam cell size tended to decrease as:
  - mixing time increased
  - mixing speed increased
  - mixing utensil and mixing container size may affect cell size, but less obvious
- EF-AR08 density change observed with mixing speed not as significant as REF308/320
  - REF308/320 more viscous and appears to be affected more by changes in mixing speed and mixing time
- Some operator dependency in hand mixing process

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Attempt to find mechanical mixer that will give good foam results similar to hand mixing for REF foams, with less operator dependency

REF308 free rise foams

Standard hand mix

300 rpm 4-blade impeller mix

800 rpm 4-blade impeller mix

1300 rpm 4-blade impeller mix

4-blade impeller mixer at appropriate mixing speed (~800-1300 rpm) yields foam rise comparable to hand mixing

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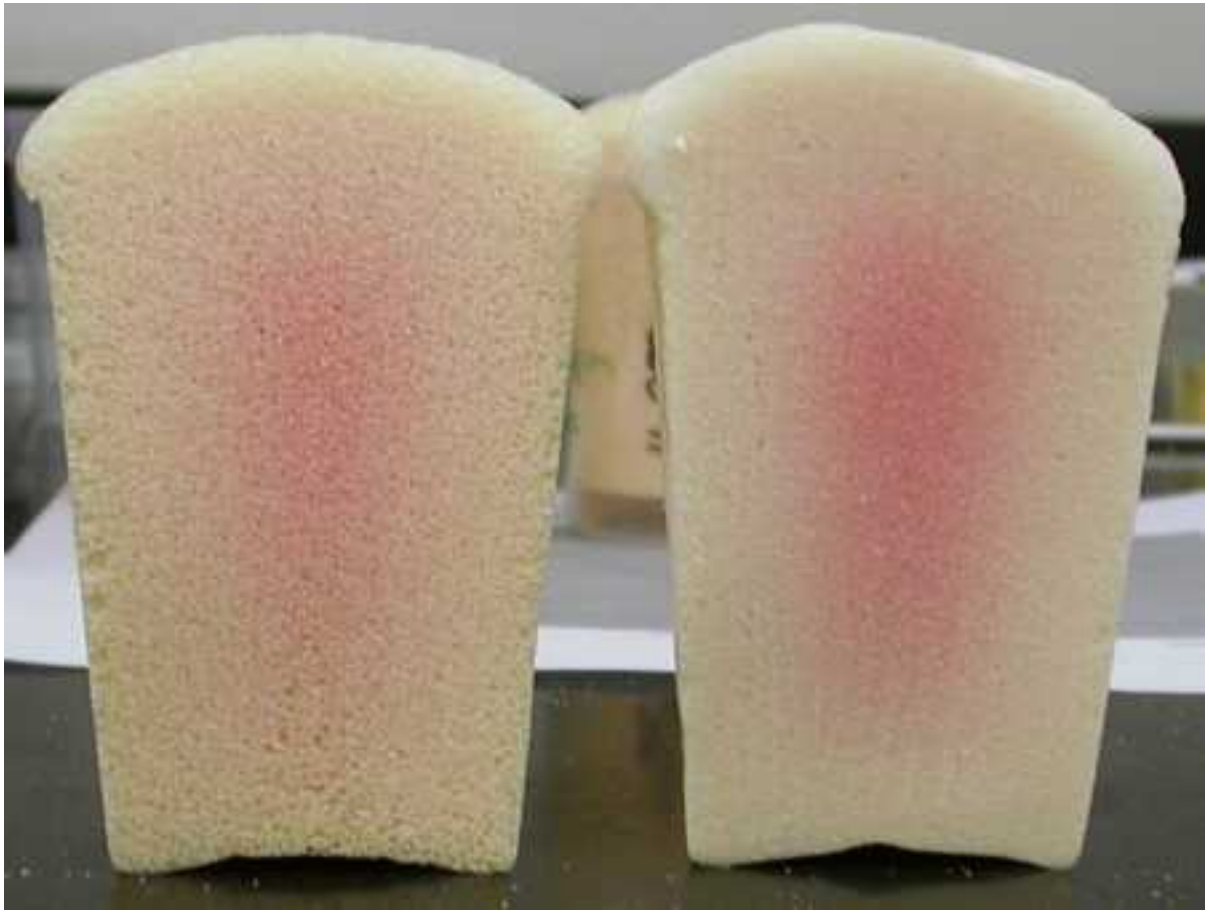
REF308 foam made with 4-blade impeller mixing has somewhat finer cell structure than hand mixing with comparable expansion



Hand mixing

1300 rpm 4-blade impeller mixing

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Summary

- REF308, REF320, EF-AR20 foams being used in a system upgrade
- Differences seen in foam rise, density of REF308/320 with hand mixing vs. KCP malt mixing – more expansion with hand mixing
  - Differences in FC-72 dispersion, nucleation, etc., being investigated by Lisa Mondy, et. al.
- Performed hand mixing study to investigate variables in the process
  - Mixing speed, mixing time, utensil, etc.
- Identified general foam rise and structure trends in hand mixing process
  - Foam rise time (and density, to lesser extent) tended to increase with increasing mixing time and speed
  - Foam cell size tended to decrease with increasing mixing time and speed
  - Some operator dependency in hand mixing process
- Identified 4-blade mechanical mixer that yields foam rise, density comparable to hand mixing at appropriate RPM – less operator dependency in mechanical mixing process

Acknowledgments

Thanks to:

- Dick Grant – SEM analysis
- Morgan Estill – Foam processing support and photography

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