Quality Assurance of RAM Mixed Compositions

Mr Vincent I. Coombs

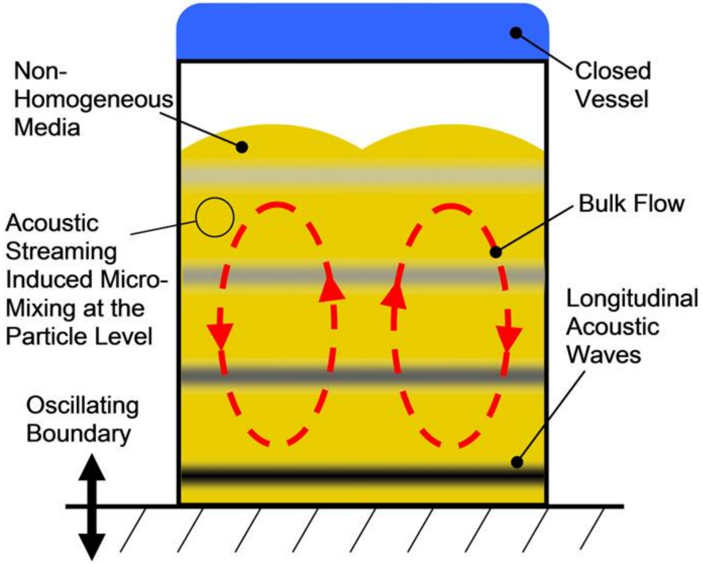
Fulmination 18 April 2023

www.cranfield.ac.uk

Content

Introduction

- RAM: Novel mixing technique based on rapid acceleration of mixing vessel.
- Energy imparted into the vessel contents generating low shear mix zones throughout the entire vessel.
- Contrasted to more conventional mixing techniques (Planetary mixing etc.); high shear in specific mix zones
- Advantages:
  - More Efficient mixing (Shorter mix times)
  - No mechanical components in mix vessel
  - Scalable quantities
- Disadvantages:
  - Little prior experience in the explosives sector.
  - Currently no industrialised example to learn from
  - Burden of evidence on manufacturers.



RAM Mixing Mechanisms
Osorio and Muzzio (2015)

Advantages

- Primarily RAM offers advantages for bulk mixing.
- Not dependant bulk vessels.
- Potential for in case mixing
- Possible issues with certification of batches
    - 1 case = 1 batch/lot
- Solution may lie in justification of reduced inspection using Process Capability.

resodynmixers.com (2023)

Aims

- To investigate the relationship between Mix time and mix homogeneity in RAM mixed compositions
  - Mix times between 10s and 30 mins
  - Inert mixes

- Evaluate the correlation between Mix intensity and homogeneity
  - Mix intensities between 0 and 80g

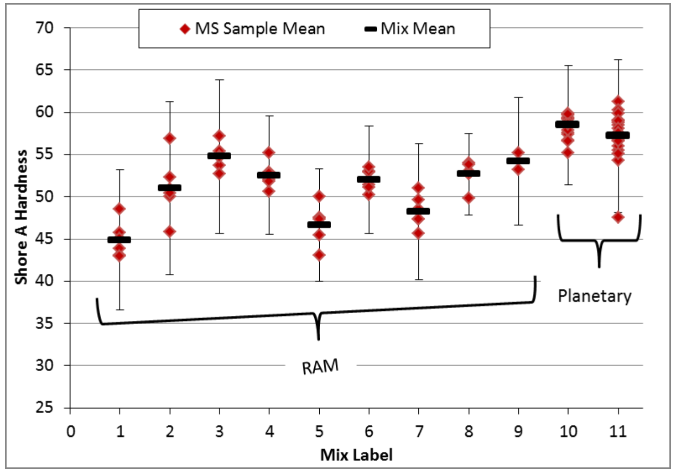
- To evaluate the Process Capability of RAM to generate a homogenous mixture based on control of mix intensity.
  - Measured by Process Capability Ratio (Cp)

Previous Studies

- Mostly from the pharmaceutical field
  - Used to mix API into carriers
- Largely concerned with efficiency of mixing
- Little consideration of repeatability or reliability of the process
- Good Methods for determining Mix Efficacy
- Salient Literature from my review
  - Davey, Wilgeroth and Burn (2019)
  - Orsorio and Muzzio (2015)

Davey, Wilgeroth and Burn

- Investigated differences between Planetary and RAM mixing.
- Considered the effect of Mix intensity on mechanical properties.
- Some work into the effect of mix time on material properties.
  - Relatively high tolerance for mix time given sufficient intensity.
- Showed that RAM comps comparable to Planetary.
- Data that variability of Shore A hardness reduced with greater mix intensity.
  - Homogeneity



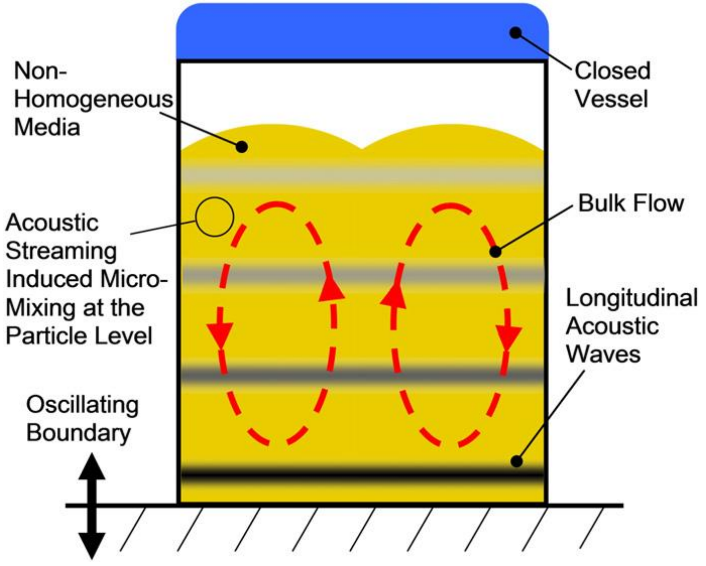
RAM (60 G) vs. planetary comparison (Shore A)
Davey, Wilgeroth and Burn (2015)

Osorio and Muzzio

- Considered the effect of many factors on mix homogeneity:
  - Vessel fill level.
  - Mix time.
  - Acceleration (Intensity).

- Used difference from theoretical concentration as a measure of homogeneity.

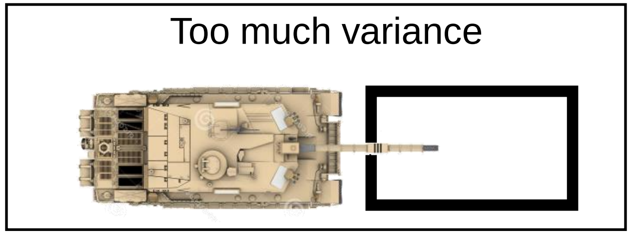
- Found that:
  - Fill level had little effect on homogeneity of mix.
  - Mix time beyond a certain threshold had reduced effect.
  - Acceleration greatly determined the mix homogeneity.

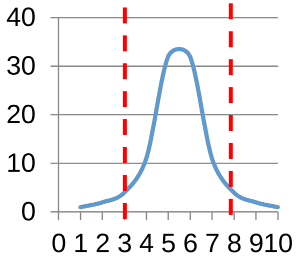


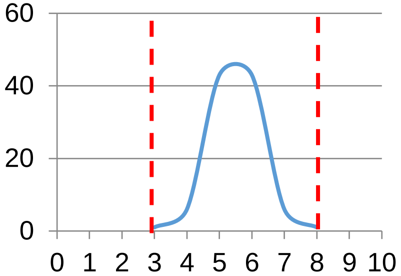
RAM Mixing Mechanisms
Osorio and Muzzio (2015)

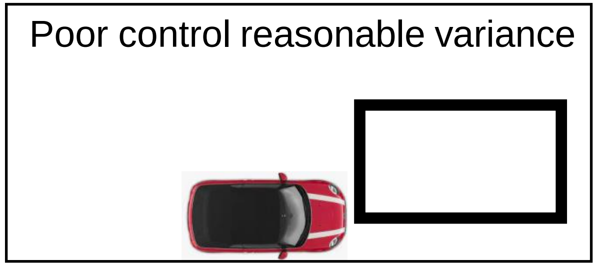
Process Capability Index

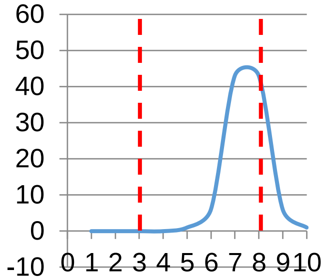
- Measured by Process Capability Ratio(Cp) / Index (Cpk).
- Cpk indicates how centred the process is and its size compared to the spec.
- Cp indicates proximity to limits.
- Influenced by:
    - Natural/induced Process Variability (σ) (Process Limits).
    - Process Mean (x’).
    - Process adherence.
    - Specification Limits (L) (acceptable results).
- Appropriate for processes that are statistically in control
    - Long history of success.
    - Mathematically proven to be statistically reliable.

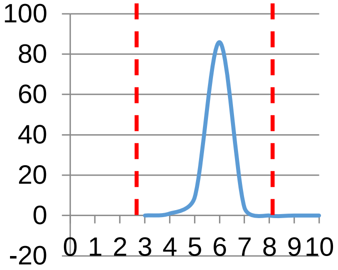


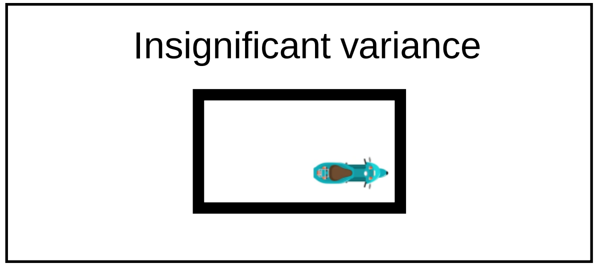












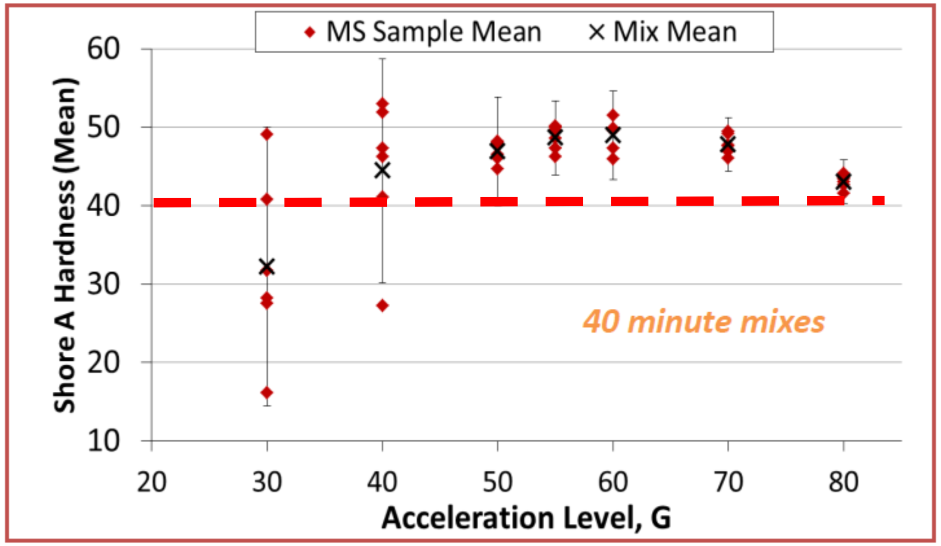
Process Capability Ratio Cp
(Davey, Wilgeroth and Burn (2019))

- Based on
  - proximity of process mean (x’) to limits (L)
  - Variance in process (standard deviation σ)

$$Cp = \frac{(X' - L)}{3 \times \sigma}$$

- Only taking Values from mixes over 50G acceleration (estimated for illustration only)
  - X’ = 46.6
  - L (arbitrary for demo) = 40
  - σ = range/d2 = 0.86

- Cp for Davey Wilgeroth and Burne Data (estimated)
  - 2.55
  - Cp values of between 1.33 and 2 considered Capable

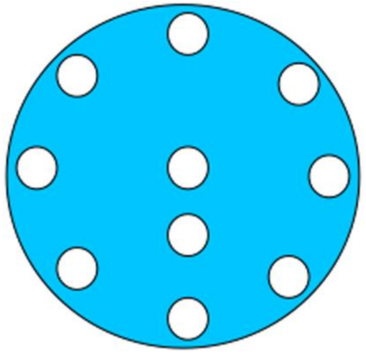


Davey, Wilgeroth and Burn (2019)

Capable?	Cp
Not	0.33
Capable	0.67
Capable	1
with	1.1
tight	1.2
control	1.3
	1.33
	1.4
	1.5
Capable.	1.6
	1.67
	1.8
	2

Methodology (Homogeneity)

- How to measure.
  - Simple 2 part mixture
  - Soluble base and insoluble measured component
- “Formulation:” known ratio of components.
- Post mix sample component ratio compared to bulk ratio
- Homogeneity measured as sample deviation from bulk ratio
- Based on Osorio and Muzzio (2015)



Sampling Protocol
Osorio and Muzzio (2015)

Methodology (Duration and Intensity)

Mix Duration

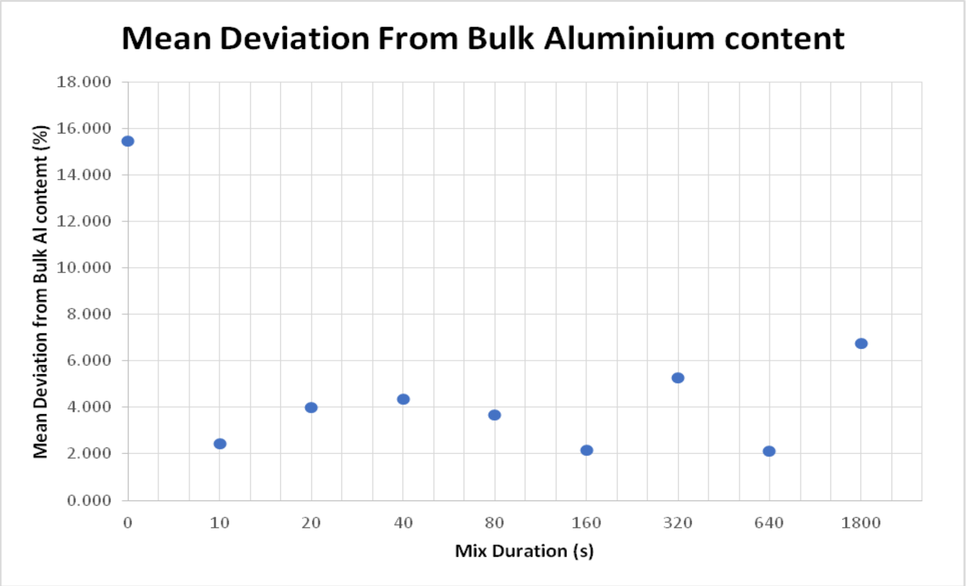
- Several samples mixed for varying lengths of time.
  - 0s (control) to 1800s (30min)
  - Peak Intensity 50g
- 3 runs per mix length
- Plot mean deviations from theoretical bulk mixture
- Determine optimum mix time.

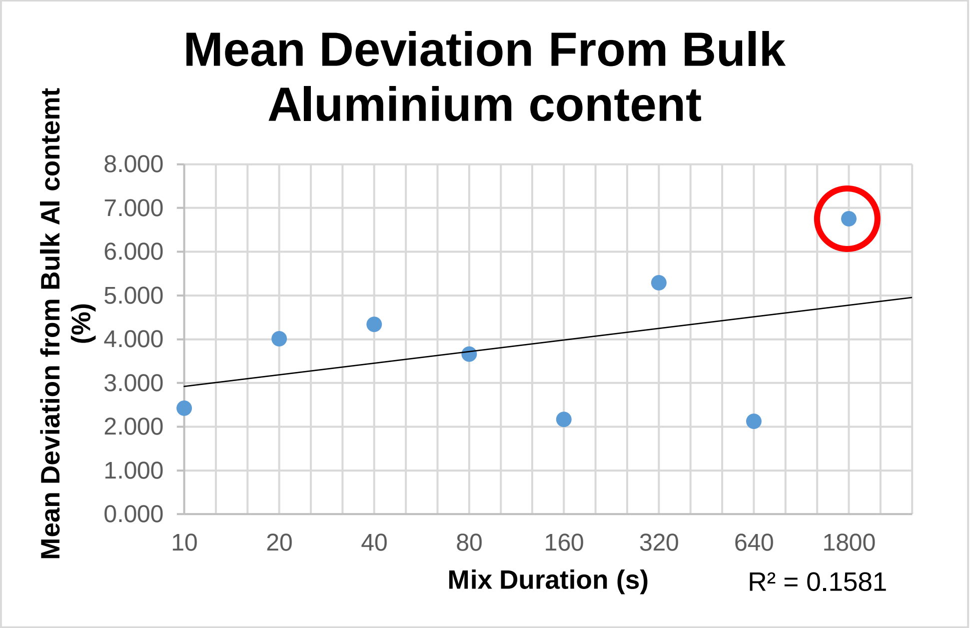
Mix Intensity

- Several samples mixed for varying peak accelerations
  - 0 (control) – 80g
- 3 Runs per level
- Mix time determined by earlier work (640s)
- Plot mean deviations from theoretical bulk mixture
- Plot best fit and determine coefficient of correlation

Results (Mix Duration)

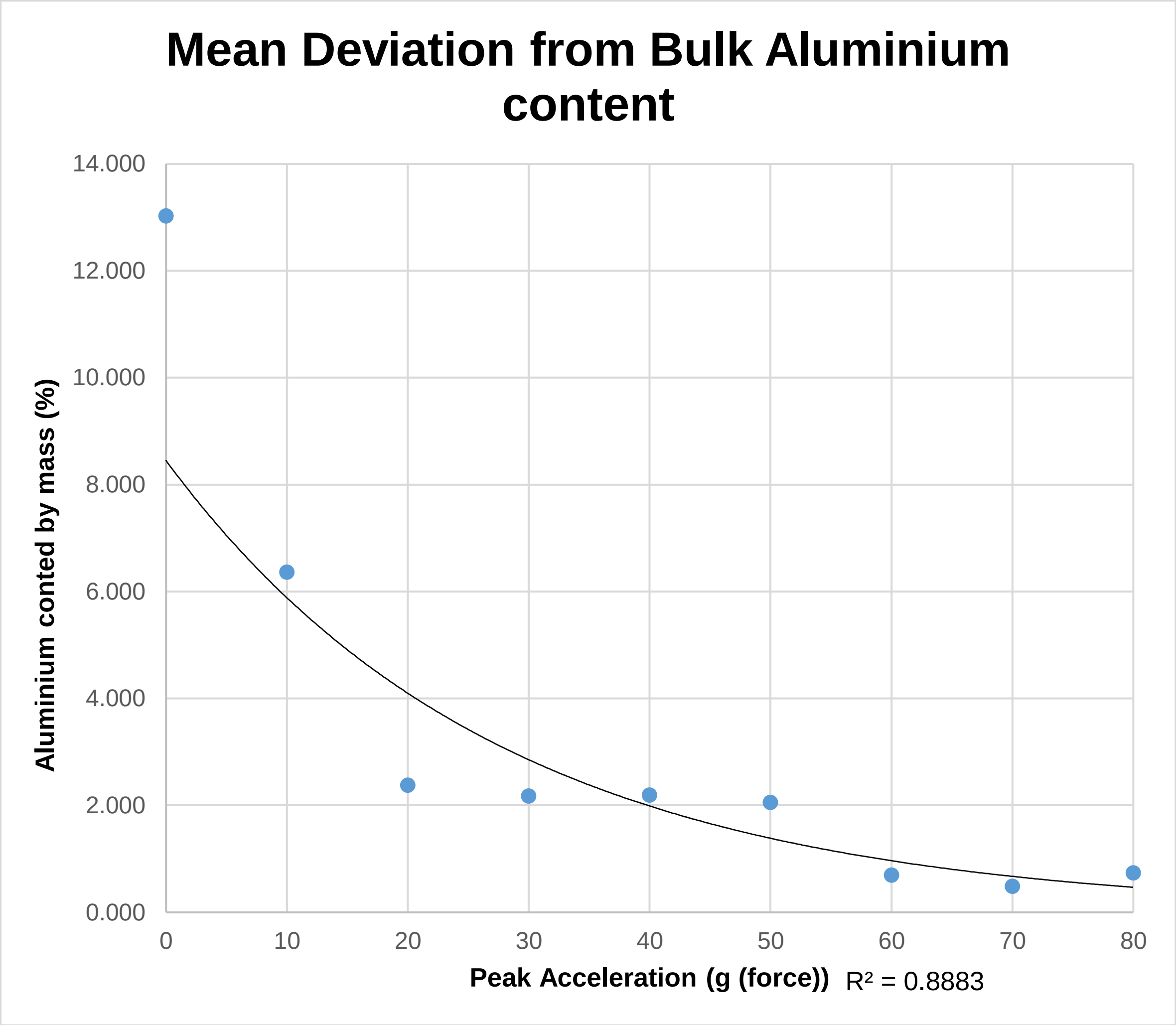
- High Deviation from Bulk content for unmixed sample
- Significantly lower deviation for mixed samples regardless of duration.
- Agreement with Davey, Wilgeroth and Burn.
  - Mix duration irrelevant beyond a given mix intensity (50g)
- Low correlation between mix duration and homogeneity..
- Some anomalous readings at 1800s leading to greater mean deviation





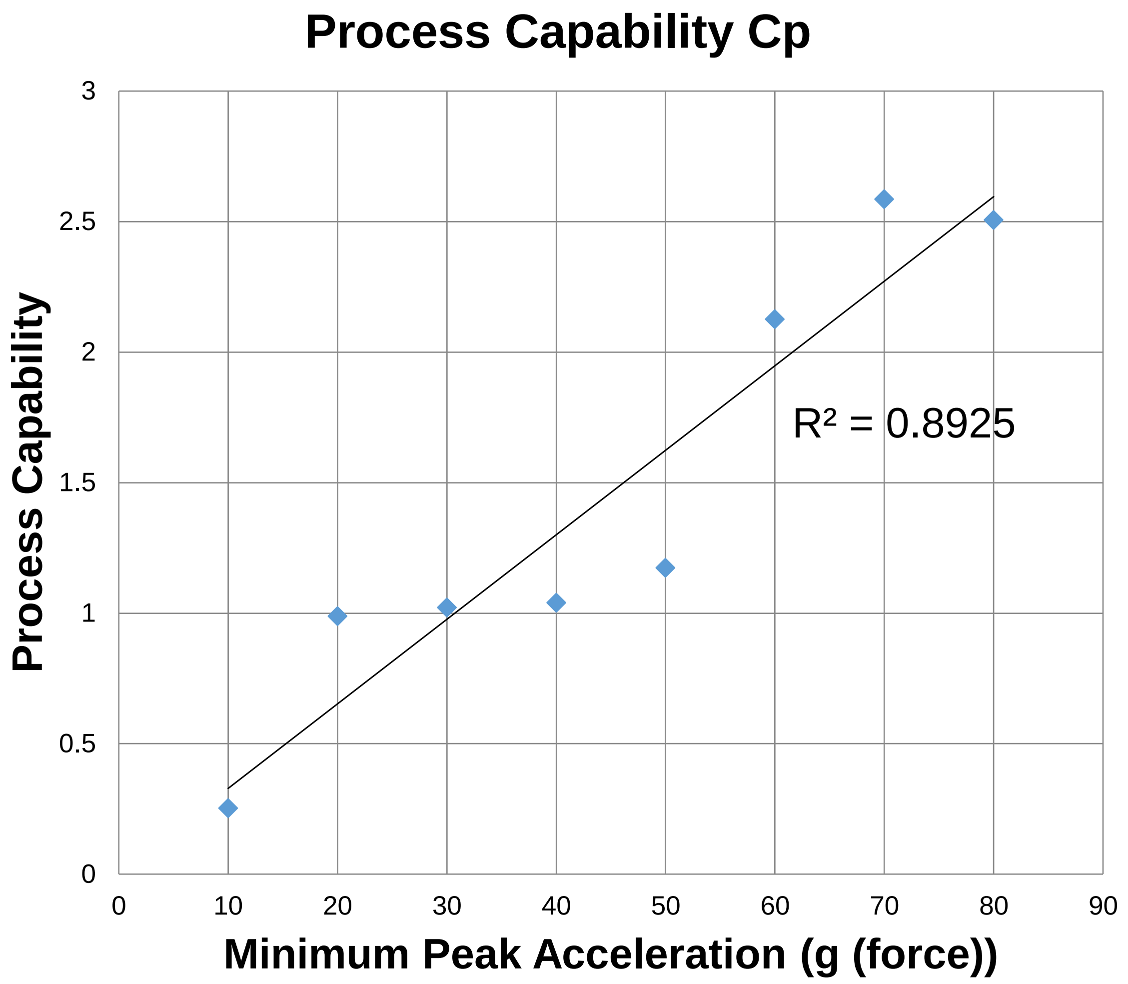
Results (Mix intensity)

- High Deviation for unmixed samples
- Reduced Deviation with increasing intensity
  - Coeficient of correlation = 0.8722
- Optimum mix intensity between 50g and 60g
- Deviations from the trend line likely due to primitive sampling methodology.



Process Capability Cp

- For a normally distributed set of data, all values can be assumed to be within 3σ of the mean
- CP is a simple quotient of the Specification range over the process range. Therefore
  - $Cp = \frac{(USL - LSL)}{6\sigma}$
- USL = Upper Spec Limit, LSL == Lower Spec Limit
- Assuming a LSL of 0% and USL of 5%
  - $Cp = \frac{(5 - 0)}{6\sigma} = 0.253$
  - Cp = 1 is considered barely adequate
- BY rearranging the above we can see that to achieve Cp=1 we must significantly increase the tolerance band.
  - $1 * (6 * 3.291) = (USL - LSL) = 19.746$

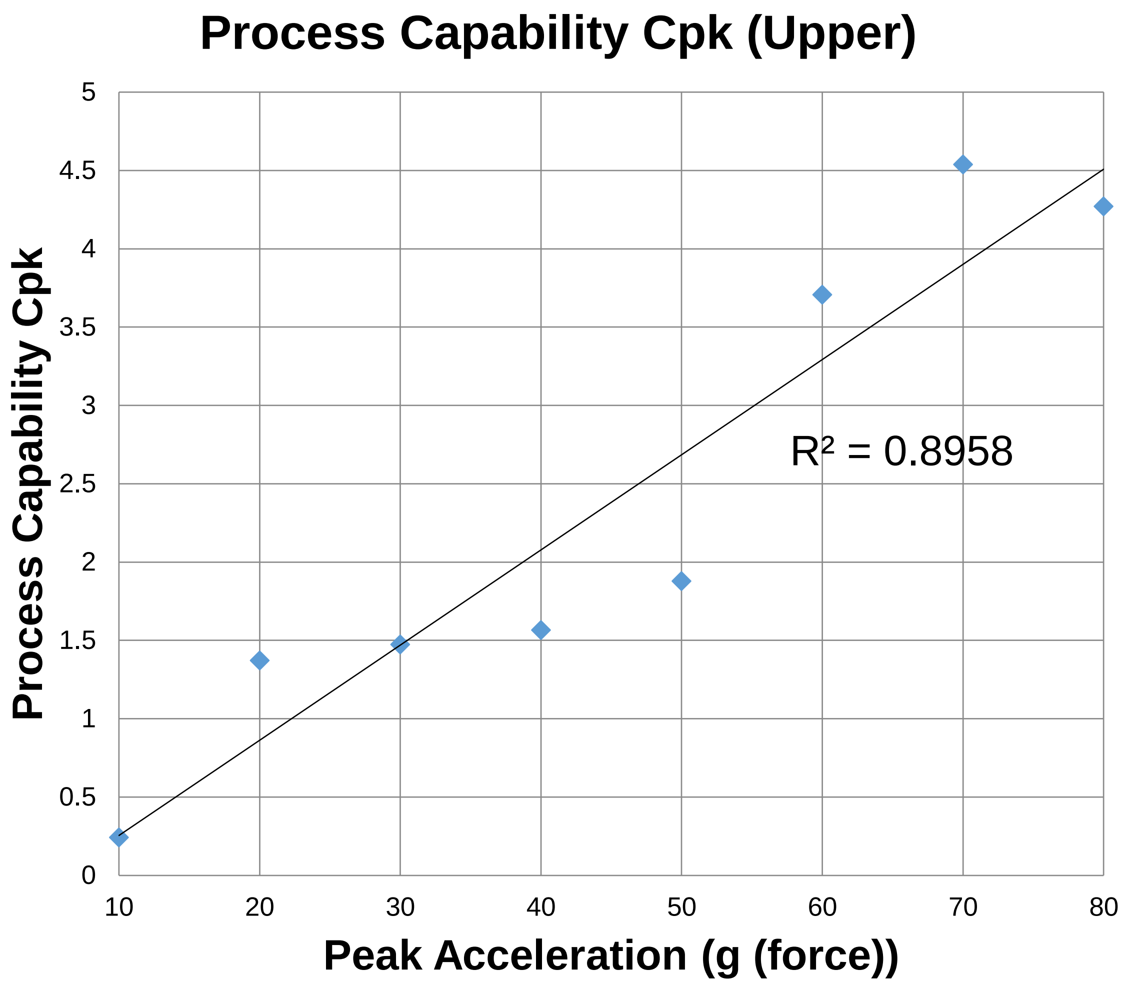


Process Capability Cpk

- Cpk compares the proximity of the process to the nearest specification limit, against the variability of the process
- Predicts failure rate
- $Cpk = min\left\{\frac{\mu - LSL}{3\sigma} \middle| \frac{USL - \mu}{3\sigma}\right\}$
    - USL = 5%, μ = mean
    - For minimum peak acceleration of 10g:
    - $Cpk = \frac{5 - 2.606}{3\sigma}$
    - $Cpk = 0.242$

CPK	Process Yield	DPMO
0.33	68.27%	317300
0.67	95.45%	45500
1	99.73%	2700
1.33	99.99%	100
1.67	99.9999%	1
2	99.999998%	0.02

Alkubaisi M. (2013)



Conclusion

- Homogeneity largely independent of mix duration.
- Homogeneity improves with Mix intensity
- Process capability can be improved with mix intensity
- Mix intensity can therefore be used as a primary indicator of compliant mixes
- Some to be done work to understand:
  - Process capability of RAM mixing overall
  - Factors at which mix duration becomes a limiting metric
- Good understanding of process capability can:
  - Allow reduced inspection rates
  - Allow greater insight into expected batch performance
  - Allow for optimisation of production processes
  - Form part of the argument for the qualification of RAM compositions.

Cranfield University

www.cranfield.ac.uk

T: +44 (0)1234 750111

@cranfieldduni

@cranfieldduni

/cranfieldduni