Distributive mixing analysis of a dense, multi-component food paste using Thermogravimetric Analysis (TGA)

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Introduction

Industrial processes which rely on the mechanical mixing of components, be it continuous mixing or batch mixing, often require that a product be "sufficiently mixed" or "sufficiently homogenous" in order to satisfy quality control. How can these criteria be defined quantitatively for dense pasty materials and how can the performance of one mixing process be compared with another?

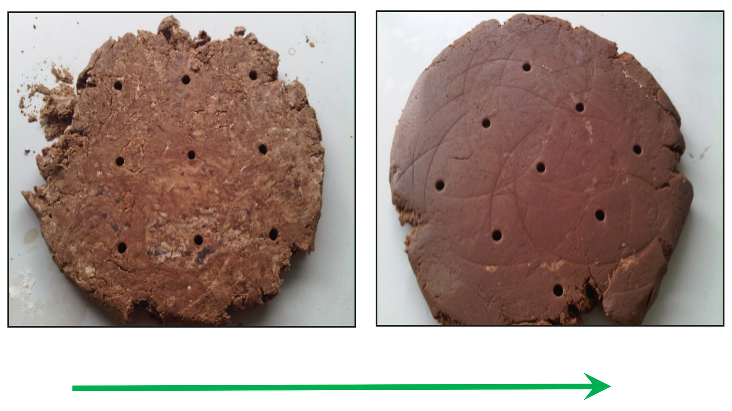


Figure 1. A batch mixed confectionary paste clearly becomes more homogenous with mixing time, but by how much? Which ingredients disperse most efficiently?

Materials and methods

Thermogravimetric Analysis (TGA) measures the mass change of a sample as it is heated and may be used to determine the concentration of individual components which thermally decompose at known temperatures. The standard deviation, SD, of concentrations observed from multiple samples then provides the most basic measure of homogeneity in a mixture.

But what if the mixture contains many complex, organic ingredients which do not decompose entirely at discrete temperatures and ingredient concentrations cannot be determined accurately? A 6 ingredient, batch mixed confectionary paste made from icing sugar, powdered dairy ingredients, molten cocoa fats and water has been sampled during batch mixing in a planetary mixer to develop a new TGA mixing analysis for this scenario.

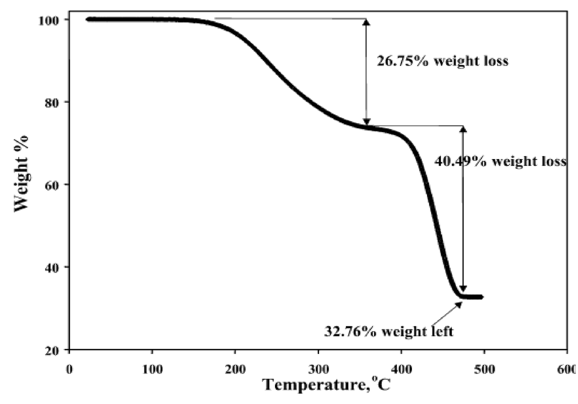


Figure 2. A well defined thermogram for a 3 component mixture. All component concentrations are determinable (Kalyon et al, 2006)

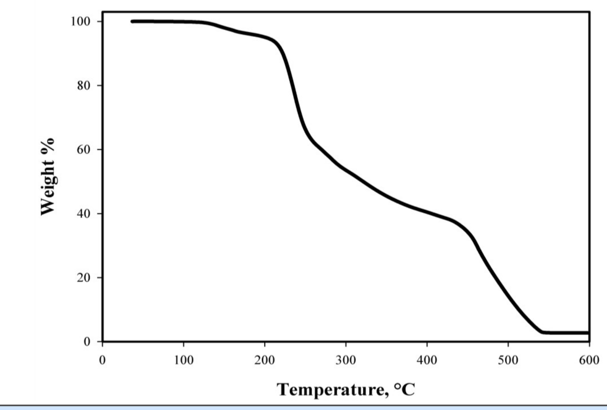


Figure 3. A poorly defined thermogram for a 6 component mixture. Component concentrations are undeterminable.

New analysis method

- Consider each sample as being composed of an infinite number of components which all thermally decompose at discrete temperatures.
- Each ingredient decomposes over a temperature range of ΔT_i and is present in the quantity ΔM_i.
- The gradient of a thermogram, $-dM_i/dT_i$ therefore gives the % mass present of each infinitesimal ingredient.
- The standard deviation of thermogram gradients for multiple samples plotted as a function of temperature can then be integrated over the entire temperature range as a mixing index of sample homogeneity:

$$\text{Mixing Index} = \int_{T_{min}}^{T_{max}} SD(T)\, dT$$

- If peaks in inhomogeneities occur at temperatures corresponding to peaks in the thermal decomposition rate of raw ingredients furnaced alone (see Figure 4 for example), the magnitude of the inhomogeneity peaks may be used to track the mixing behaviour of individual ingredients.

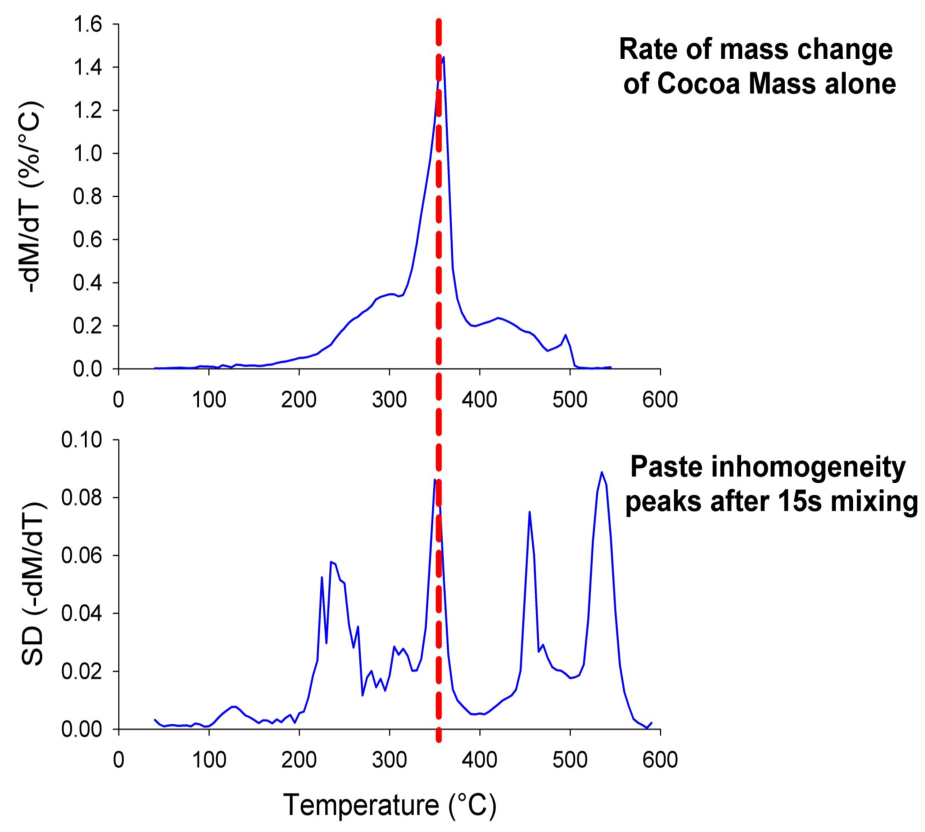
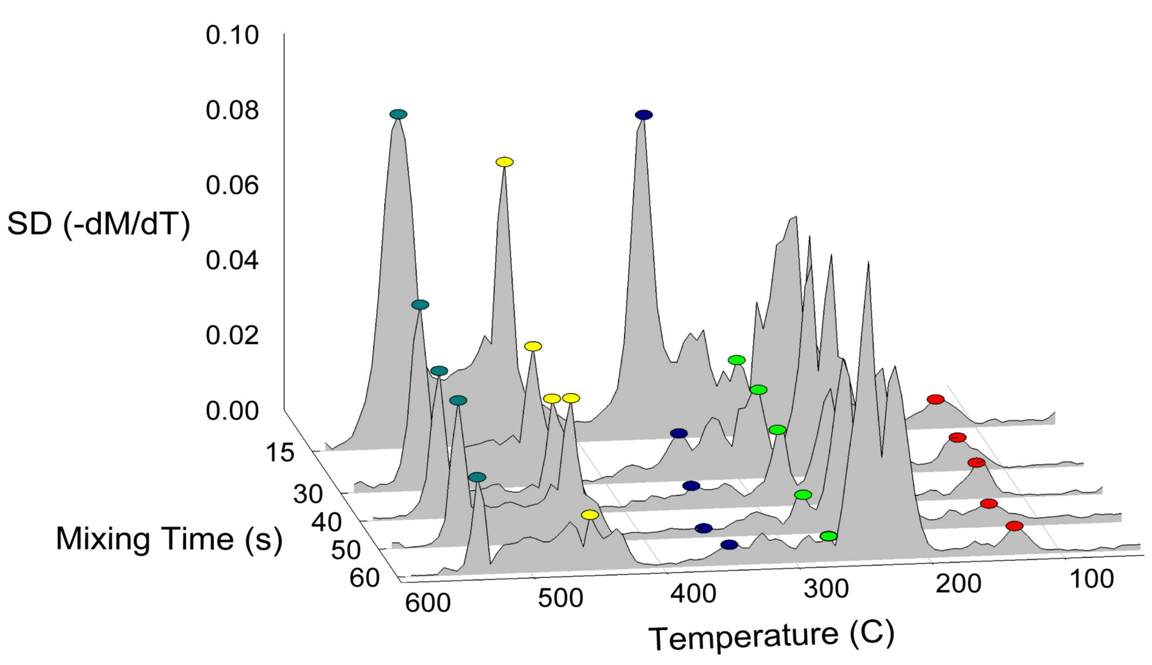


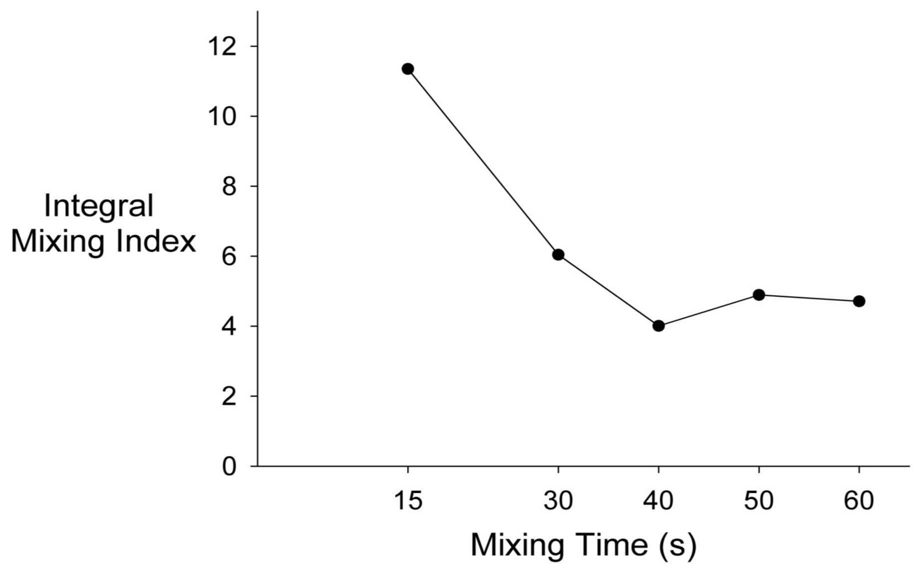
Figure 4. A paste inhomogeneity peak (bottom) shown to correspond with thermal decomposition peak of cocoa mass alone (top).

Results

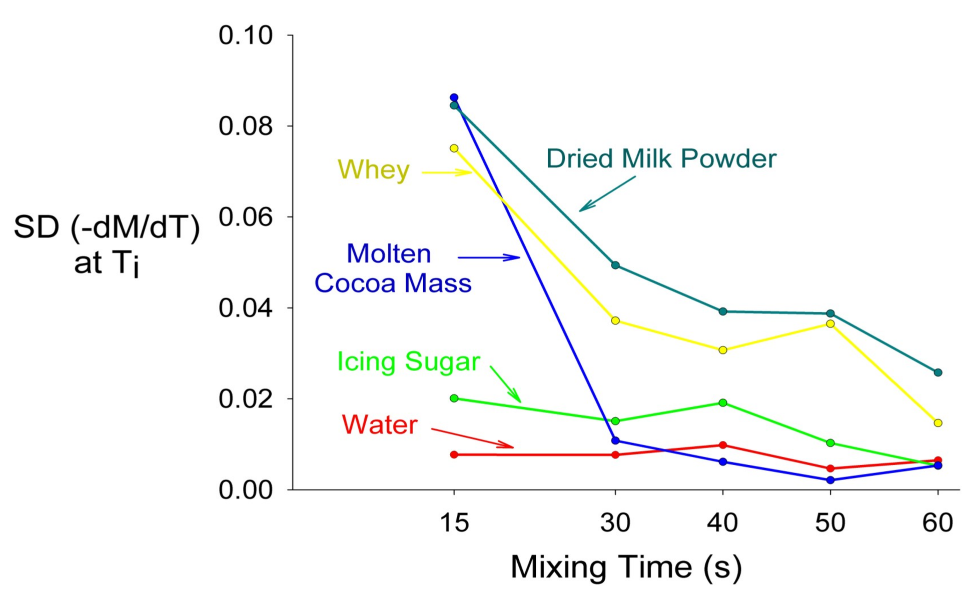
Changing distribution of inhomogeneities with mixing time



Improvement in homogeneity with increased mixing



Individual ingredient mixing



Observations

- A decreasing integral mixing index shows an expected increase in overall homogeneity with mixing time.
- Inhomogeneity peaks appear to occur at temperatures corresponding to peaks in mass change rate of some raw ingredients, giving insight into the relative distributive mixing of individual components (or subcomponents thereof).
- Identifiable liquid ingredients appear to disperse more readily than powdered ingredients.
- High viscosity cocoa mass takes longer to disperse than low viscosity water.
- Identifiable powdered ingredients appear to disperse at differing rates, for as yet unstudied reasons.

Conclusions

- Thermogravimetric data for a batch mixed confectionary food paste has been analysed in a new way to quantitatively observe distributive mixing of the bulk and individual components with time.
- Standard deviations of rate of mass change for multiple samples plotted against temperature can be integrated to generate an overall homogeneity index.
- The standard deviation functions of temperature reveal peaks in inhomogeneities within particular temperature ranges.
- Inhomogeneity peaks appear characteristic of individual raw ingredients allowing peak magnitudes to give insight into the relative distributive mixing performance of components.

Acknowledgments

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Kalyon et al (2006), "Rheological behavior of concentrated suspensions as affected by the dynamics of the mixing process", Rheological Acta 45: 641 - 658