**Understanding the effect of manufacture process variation on polymer properties**

Coatings are applied to a substrate to impart decorative and functional properties which will extend the lifetime of the asset. Polymers are large molecules made up of repeating units of smaller molecules, chemically bonded together. The polymer is the main constituent of a coating and is the film-forming element of the product. It provides adhesion to a substrate, binds pigments and other additives together, and determines important properties such as colour stability on exposure to sunlight, corrosion protection or resistance to chemicals.

Control of the manufacturing process is key to ensuring the polymer has the desired properties to provide adequate coating performance for our customers. There are several factors to process control which show variation from batch to batch, such as temperature, speed of addition of raw materials (feed rate), speed of stirring (agitation) and pressure. In addition, materials can be made across different manufacturing sites. The properties of the polymer are measured as part of our quality control (QC) process. This ensures that the polymers subsequently used in paint manufacture are suitable for use. Properties measured include:

- Non-volatile content – the weight percent of final material that is not volatile (has a higher boiling point and therefore is not at risk of being lost to the environment via evaporation)
- Viscosity – determination of how the material flows under stress (the higher the viscosity, the slower the flow)
- Specific gravity – density of the material
- Residual material A – the weight percent of raw material A that remains unreacted in the polymer once manufacture is complete
- Molecular weight – a measure of the size of the individual polymer molecules – this is calculated in three ways, giving the parameters Mn, Mw and Mz.
- Polydispersity index (PDI) – a measure of the variation in the size of the individual polymer molecules

The manufacturing process is split into a number of defined steps:

1) Heat the reactor to 85$^{o}$C;
2) Feed in set weight of material A and simultaneously feed in a set weight of material B;
3) Heat the reactor to 95$^{o}$C;
4) Feed in a set weight of material B;
5) Hold the reactor at 95$^{o}$C;
6) Cool the reactor to 62$^{o}$C.

The polymer is then discharged from the reactor and QC checks are done to determine whether key properties are within the product specification.

At AkzoNobel we would like to improve our understanding of the effect of variability in the manufacturing process on the polymer properties. This would enable us to better define our process control limits, set the polymer QC specification and speed up the scale-up process from small, lab-scale batches to several-tonne factory batches.

Objectives

- Analysis of batch log data to provide summary information of key process and property metrics for each batch:

    - Time taken for each feed (for both materials A and B in step 2 and the second feed of material B in step 4);
    - Temperature variation from the specified temperature for each step;
    - Maximum and minimum temperature for each step;
    - Average stirrer speed for each step and across the whole process;
    - Whether a batch is in or out of specification based on the QC results;
- Analysis of the properties and process datasets to show the overall variability in both process metrics and polymer properties.
- Use of statistical modelling to give an understanding of the relationships between process metrics and polymer properties.

Understanding the dataset

A sample batch log and property QC analysis have been provided for a single batch of material. All data relates to a single manufacturing site.

**Batch Log**

- Batch Number
- Timestamp (dd/mm/yyyy hr:min:sec AM/PM)
- Reactor Temperature ($^{o}$C)
- Reactor Pressure (bar)
- Material A weight (kg) – cumulative weight added of material A (used to calculate the time over which material A is being fed into the reactor)
- Material B weight (kg) – cumulative weight added of material B (used to calculate the time over which material B is being fed into the reactor)
- Agitator (amp) – measure of the current flowing to the stirrer in the reactor. The assumption is that this is synonymous with stirrer speed (agitation) as all batches for this dataset were made on the same reactor.
- Step – steps in the manufacturing process as outlined above

**QC metrics**

- Batch Number
- Solids (wt%)
- Viscosity (@25deg, Poise)
- Specific Gravity (g/ml)
- Residual monomer (wt%)
- Mn (g/mol) – measure of molecular weight (the size of the polymer molecules) based on the number of individual molecules
- Mw (g/mol) – measure of molecular weight (the size of the polymer molecules) based on the weight of individual molecules
- Mz (g/mol) – measure of the molecular weight (the size of the polymer molecules) in relation to the molecular weight of the molecules of the raw materials
- PDI (no units) – polydispersity index - a measure of the variation in the size of the individual polymer molecules