High Throughput Polymer Latex Synthesis and Purification Process

"Size Does Matter"

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Abstract: Polymer latexes are essential components in a wide range of commercial products and formulations such as, paints, cosmetics, coatings, biotechnology and functionalised supports. Many difficulties are intrinsic in the implementation of polymer latex research, particularly in purification of the latex dispersions, making high throughput research in this area especially challenging. By the use of experimental design, automated synthesis and the new developed high throughput purification process, the investigation of the influential factors on polymer latexes can be swiftly and reproducibly achieved. This allows fully characterised libraries of functionally diverse polymer latexes to be produced and screened for a wide range of applications.

1) Background

A flexible process to conduct high throughput investigations into polymer latex synthesis via dispersion and emulsion polymerisations has been developed[1] (see figure 1). The process uses Design of Experiments (DoE) to establish synergistic and antagonistic effects of reaction conditions (temperature, reaction time, reactor size, surfactant, etc…) on the final polymer latex's physical properties (molecular weight, particle size, etc…). Also, a high throughput latex purification and analytical sample preparation process was also developed to allow the production of fully characterised libraries of functionalised polymer latexes in a parallel fashion.

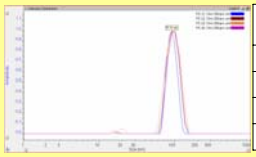
Figure 1. Chemspeed[2] SLT 106 set up with 13ml, 27ml and 75ml reactors to conduct HT latex synthesis investigations via experimental design.

2) Synthesis and Purification

Multiple "onion layer" DoE models were used to identify and quantify the influential synthesis parameters used during the production of latexes in a range of reactor sizes using a Chemspeed SLT 106 synthesis platform. One such investigation model discussed below measures the effects of shear rates on latex synthesis in different reactor sizes (13ml – 75ml) caused by increasing vortex mixing speeds. This allows us to assess reproducibility and control of Mw and particle size over a range of scales.

Figure 2. Polystyrene latexes (including repeat reactions to measure reproducibility) synthesised via dispersion polymerisation in 13ml, 27ml and 75ml reactors (left, centre and right images respectively) at increasing vortexing speeds (from left to right).

Latexes produced in 13ml, 27ml and 75ml reactors at increasing vortexing speeds in figure 2 (200rpm – 800rpm left to right) show that high vortexing speeds cause the reaction to undergo bulk polymerisation rather than a forming a latex dispersion (figure 2). It was also shown how this effect is exagerated in larger reactor sizes thereby highlighting how crucial the effects of mixing speeds and reactor scale are on latex synthesis.



Sample	Vortex speed (rpm)	GPC Mw
PS (1)	200	579,013
PS (2)	200	596,389
PS (3)	200	623,630
PS (4)	200	576,596

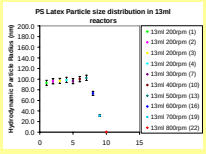


Figure 3. The process showed good reproducibility in both particle size distribution and Mw of repeat reactions via DLS and GPC (left). DLS was also used to illustrate the influence of mixing speed on particle size (centre). A similar dependence of mixing speed and reactor size was also observed when evaluating the mechanical stirred 100ml Chemspeed mini plant reactors (right).

3) Semi-Automated Batch Latex Purification Process

The synthesized latexes are then fed into a newly developed automated purification process in batches of up to 96 samples. These are first centrifuged before using the Infrared level detection camera on an Eppendorf epMotion 5075LH liquid handler[3] to measure the liquid level in each centrifuge tube to remove the supernatant liquid without disturbing the latex, thus extracting excess surfactant and unreacted monomer (figure 6). Fresh medium is then added and the polymer redispersed before the process is repeated until the polymer is purified. To ensure the surfactant or stabiliser has been removed by these repeat washings, a sample of the supernatant is tested using a Kibron Delta-8 surface tension plate reader[4] and rapidly compared to the surface tension of the pure solvent medium[5].

Figure 4. The samples are delivered from the Chemspeed automated synthesis platforms and placed directly onto the deck of an Eppendorf epMotion 5075LH liquid handling platform where the centrifuged samples are rapidly prepared and processed.

This purification process is over 50 times faster than traditional manual methods and is scalable up to 100ml allowing researchers to rapidly produce fully characterised libraries of functionally diverse polymer latexes as shown in figures 4 and 5.

Figure 5. The latexes are centrifuged in batches of 48 samples for 20 minutes (left) and the pelleted samples returned to the liquid handler (centre). The process if also fully scalable for samples from larger reactors sizes up to 100ml (right).

4) Results

Once the latexes have been purified, the process prepares analytical samples for Dynamic Light Scattering (DLS) and Gel Permeation Chromatography (GPC) and the final samples are stored as a 2D bar-coded library in a robotically compatible format for future application screening.

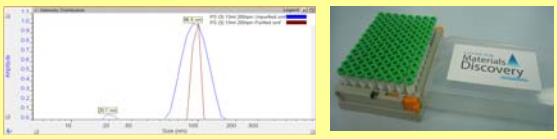


Figure 6. Particle sizing via DLS (left) shows particle distributions of a latex sample before (blue) and after (red) ten purification washings. Also the removal of small impurities can clearly be observed. The final library of multiple replicates of each latex sample is stored in robotically compatible 2D bar-coded vials and exported for application screening.

The results of these experiments are then fed into the DoE software to produce a model evaluating the influential synthesis factors on the physical properties of the resultant latex product. A number of these models can be combined to produce a fundamental and quantitative understanding of latex synthesis. This allows researchers to predict and produce new latexes with the desired physical properties for particular applications.

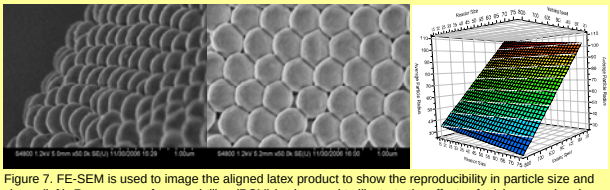


Figure 7. FE-SEM is used to image the aligned latex product to show the reproducibility in particle size and shape (left). Response surface modelling (RSM) is also used to illustrate the effects of mixing speed and reactor volume on latex particles size (right).

5) Conclusions

A reproducible automated high throughput process for polymer latex synthesis, purification and characterisation has been established. By the use of DoE it was possible to gain an understanding of the influential parameters effecting latex synthesis and their physical properties. This control allows libaries of diverse functionalised polymer latexes to be rapidly produced and screened for wide range of applications.

References:

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