PARTICLE SIZE ANALYSIS OF POLYMERS

Measuring the particle size distribution of polymeric materials provides a high degree of control over a range of product performance characteristics. Particle size determination using static light scattering (laser diffraction) is currently the most versatile and popular technique for the majority of polymeric materials. Since extruded polymer pellets can exceed the upper size limitation of laser diffraction these materials are typically measured via dynamic image analysis. The combination of laser diffraction and dynamic image analysis systems supplies a wide dynamic range (0.01 μm to 30 mm), fast material analysis, and easy data interpretation.

Introduction

Most consumers probably only associate polymers with "plastic" materials, but this greatly underestimates the many origins and uses of polymer-based products. Any chemical which consists of regularly repeating units (called *monomers*) is classified as a polymer (see Figure 1 for PVC for example). Rubber, proteins, shellac, nylon, DNA, PVC tubing, GORE-TEX fabrics, Kevlar ballistic armor, the "non-stick" in non-stick cookware… these are all examples of better living through polymerization.
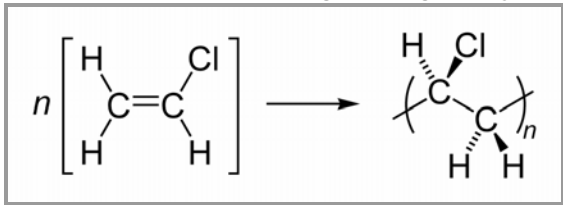

Figure 1 : PVC Polymerization Example

Measurement and control of particle size is important regardless of where the production process stops - whether directly after the polymerization reaction, extrusion into fibers (Figure 2), or after dried and rolled into sheets. Particle characterization correlates to scrap rate for several applications - creating a critical need for measurement and control of final product size.
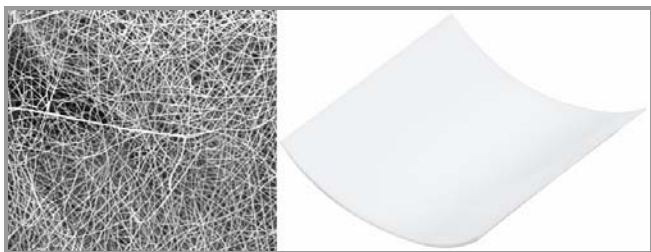

Figure 2 : Polymer Fibers and Sheets

Measurement Techniques

The two most popular sizing techniques for polymers are static light scattering (a.k.a. laser diffraction) and image analysis.

Static Light Scatter instruments measure the intensity and angle of light after interaction with the particle and transform this information into the particle size distribution. The HORIBA LA-950 features the fastest measurement time on the market (less than 1 minute for setup and analysis), widest measurement range (10nm to 3mm), and most flexible accessory set with samplers to fit most polymer applications. Speed, range, and flexibility are the hallmarks of laser diffraction size measurement and account for its ubiquity.

Dynamic Image Analysis systems contain one or more cameras capturing 2-D images of the dispersed, moving particles. Size and shape parameters are then assigned and calculated for all of the particles inspected by the system. The HORIBA Camsizer developed by Retsch Technology utilizes dynamic image analysis to analyze a wide range of materials from 30 μm to 30 mm, making this tool valuable to companies manufacturing polymer beads and pellets.

The polymer industry includes a wide range of applications ranging from sub-100nm polystyrene latex emulsions to millimeter sized extruded pellets, requiring the full range of HORIBA technologies for complete characterization of these various materials.

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Experimental Results

Accurate, reproducible measurement of polymeric materials requires a range of technologies and methodologies. Polystyrene latex emulsions present little challenge owing to excellent stability and dispersion characteristics. Fluorinated polymers (e.g. PTFE, PVF, PFA) are hydrophilic thus making dispersion a concern. PLGA, a biodegradable and biocompatible polymer used in pharmaceuticals, often has pockets of trapped air which cause buoyancy problems. This leads to dry powder measurement of PLGA as the analysis of choice.

The results presented here were collected on the LA-950 and Camsizer particle characterization analyzers.

Example 1
Polystyrene latex (PSL) serves a very important role in particle size instrumentation as certifiable and traceable standard reference materials. PSL standards are available from 20 nm to 1000 μm as either (typically) suspensions or dry powders from a variety of sources.

The LA-950 can measure these materials in either wet (i.e. water) or dry dispersion with very little effort. The example in Figure 3 was measured dry.

Example 2
Polypropylene (PP) possesses a variety of uses including injection molding, weather-modified clothing, waterproofing, and product packaging.

The Camsizer data presented in Figure 4 was obtained from extruded polypropylene pellets.

Example 3
Polytetrafluoroethylene (PTFE) is more famously known as Teflon® and has a well-earned reputation as a miracle chemical. High resistances to corrosion and wetting, a low coefficient of friction, excellent dielectric properties, and chemical inertness allow PTFE to be used in numerous applications.

The data shown in Figure 5, generated by the LA-950, comes from powdered PTFE used for industrial lubrication.
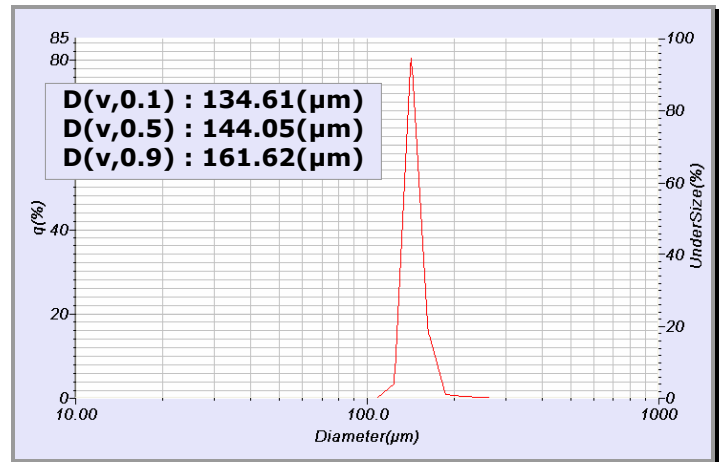

D(v,0.1) : 134.61(μm)
D(v,0.5) : 144.05(μm)
D(v,0.9) : 161.62(μm)

Figure 3 : Polystyrene latex result from LA-950

D(v,0.1) : 1863(μm)
D(v,0.5) : 2729(μm)
D(v,0.9) : 3586(μm)

Figure 4 : Polypropylene result from Camsizer

D(v,0.1) : 4.16(μm)
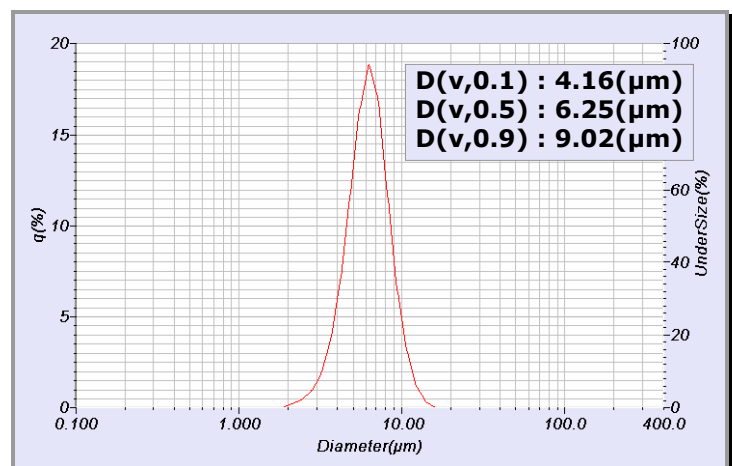D(v,0.5) : 6.25(μm)
D(v,0.9) : 9.02(μm)

Figure 5 : Polytetrafluoroethylene result from LA-950

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Example 4

Polyvinyl chloride (IUPAC Polychloroethene), abbreviated PVC is another classic example of a polymer performing many jobs. PVC is featured in construction the world over because of its low cost, ease of use, and durability. Other uses of PVC include pipes, electric wire insulation, magnetic stripe cards, vinyl siding, and conduit fixtures.

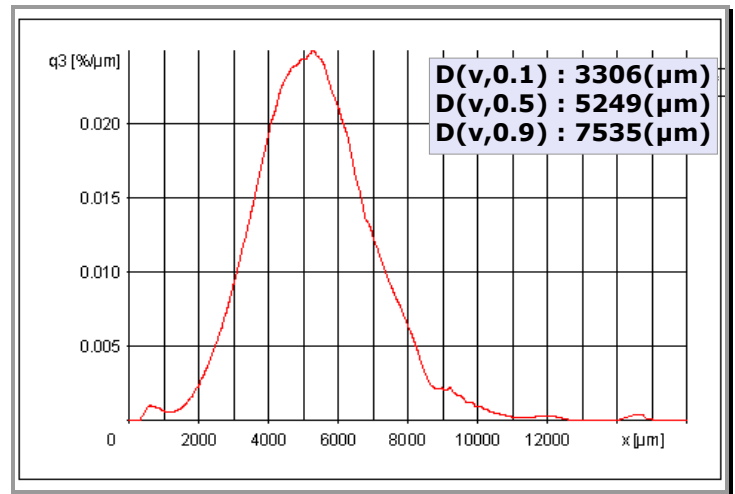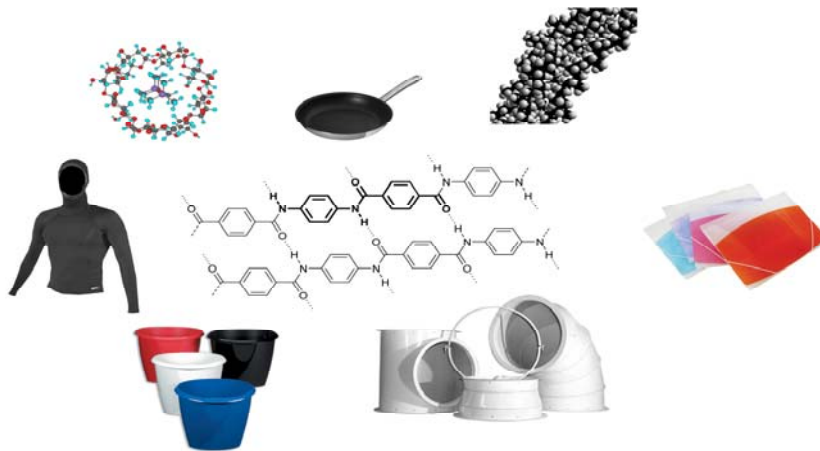Figure 6 is an example of PVC flakes measured on the Camsizer.

D(v,0.1) : 3306(µm)
D(v,0.5) : 5249(µm)
D(v,0.9) : 7535(µm)

Figure 6 : Polyvinyl chloride result from Camsizer

Conclusions

As the needs of the chemical industry change and products require more detailed inspection to maximize quality and quantity, the analytical instrumentation available must adapt to fill the space. Both the LA-950 and Camsizer successfully meet the needs for research and routine quality control through the provision of speed, ease of use, repeatability, reproducibility, and reliability. Laser diffraction analyzers measure from nanometer to millimeter sized wet or dry dispersed polymers. Dynamic image analysis systems are preferred when product size grows and shape factors become important.

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