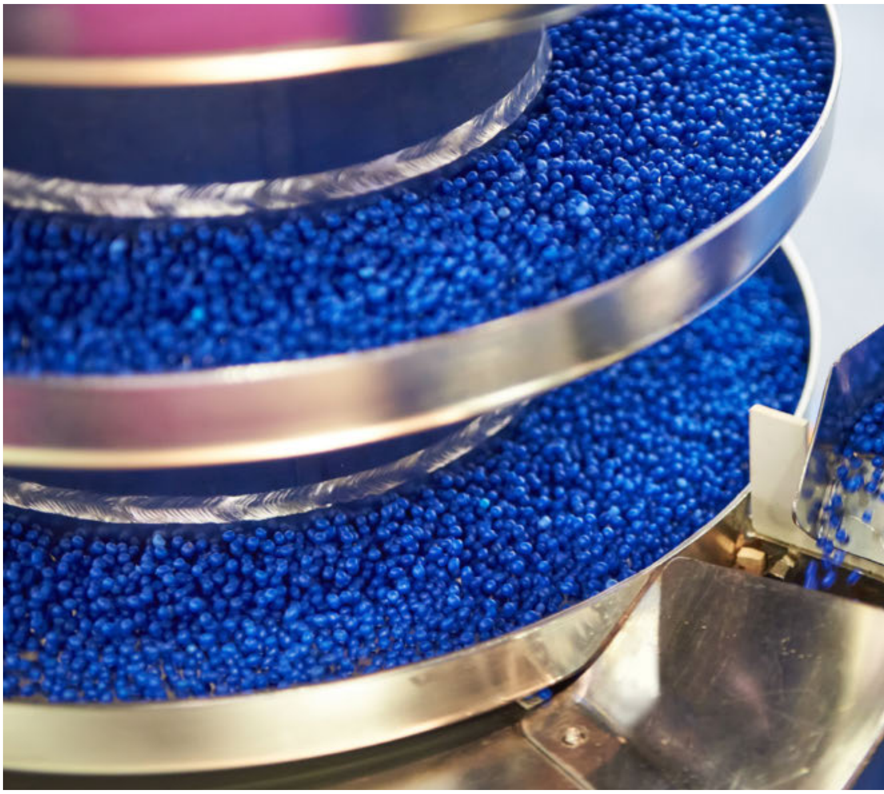

APPLICATION NOTE

Materials Characterization

Author:

Kieran Evans

PerkinElmer Inc.
Seer Green, UK

Determination of Important Physical Parameters of PPE for Effective Quality Control

Introduction

Quality control of personal protective equipment (PPE) is an incredibly important aspect of the manufacturing process. A lack of quality control can lead to an increase in failures in the products and in turn a loss of revenue for the manufacturer. It is therefore important to have a means to test materials quickly while also obtaining as much information as possible.

There are three materials characterization techniques which can provide fast and simple methods for quality control of PPE products – infrared spectroscopy (IR), differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). Each of these can provide valuable insight into the physical and chemical properties of the material used to produce PPE.

Infrared spectroscopy may be used to obtain data pertinent to the chemical composition of the material e.g. which polymer has been used in a glove. IR spectroscopy has the huge advantage of being non-destructive as well as being fast and easy to use – providing results in as little as 30 seconds.

DSC can provide data on in depth information about the physical properties of the materials such as the crystallinity and melting point. TGA offers the further benefit of giving the user an understanding of the composition of the material.

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There are also a wide variety of regulations and directives guiding manufacturers on the checks which must be carried out on most PPE materials, namely gloves. For ASTM international, the most important of these standards are:

- ASTM D6319 – Nitrile Gloves
- ASTM D3578 – Rubber Examination Gloves
- ASTM D5250 – PVC Gloves
- ASTM D6977 – Polychloroprene Gloves

Each of these designations details not only physical criteria which must be met by the glove (size, lack of holes etc.) but also the materials which must be used to produce the gloves such that the final product can easily meet the other requirements.

The stringent regulations imposed on manufacturers of PPE mean that it is crucial for the manufacturer to investigate the materials used to produce these products thoroughly. PerkinElmer provide a full suite of materials characterization techniques allowing the user to thoroughly investigate their product, ensuring it is of the highest quality.

Infrared Spectroscopy

IR spectroscopy provides a fast method for identification of polymeric materials. The PerkinElmer Spectrum™ Two+ with universal attenuated total reflectance (UATR, Figure 1) provides a small-footprint, integrated IR platform allowing analysts to collect high-quality data with no sample preparation.

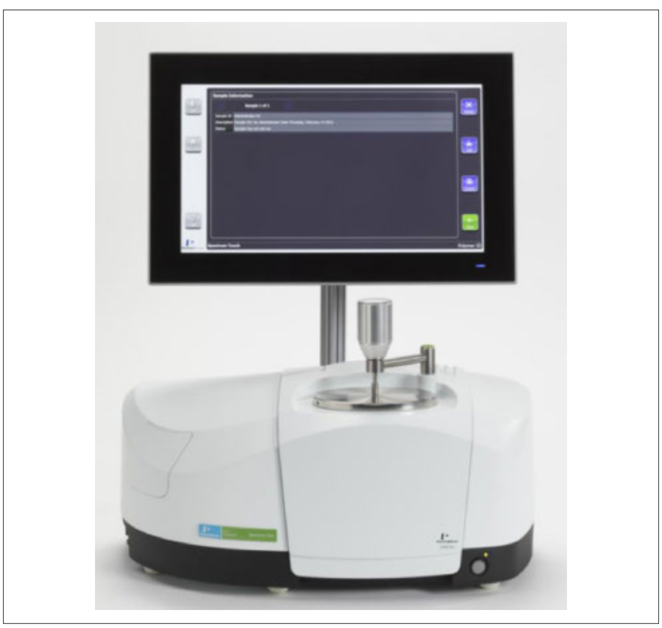

Figure 1. PerkinElmer Spectrum Two+ with UATR Accessory.

Experimental

Spectra of three different types of PPE (gloves, face masks and a face shield) were collected using the spectral parameters shown in Table 1.

Results and Discussion

Spectra of four glove samples, produced using each of the four materials mentioned in the ASTM documentation, are shown in Figure 2.

Table 1. Spectral parameters used for measurement of PPE samples.

Parameter	Value
Spectral Range	4000 – 450 cm^{-1}
Resolution	4 cm^{-1}
Number of Scans	16
Corrections	Atmospheric Vapour Compensation (AVC)

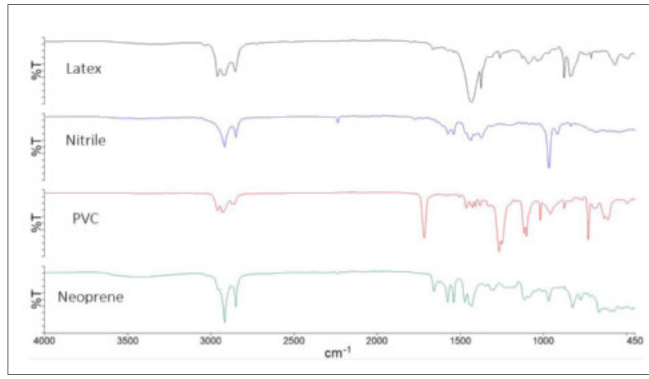

Figure 2. IR spectra of the four different glove materials covered by ASTM designations.

IR spectra can provide the user with detailed information about the chemical structure of the material as well as additives which may have been added to give the polymer to make it more fit for use. For example, the PVC spectrum shows a strong peak at 1717 cm^{-1} which is commonly assigned to a C=O stretch in a carbonyl. However, PVC does not contain carbonyl groups. This peak could be assigned to dioctyl phthalate, a plasticizer which is commonly added to PVC to increase the flexibility of the polymer.

In addition to visual investigation of polymer spectra, it is also possible to use the library search function in PerkinElmer's Spectrum 10 software to identify polymers using commercially available IR polymer libraries. For example, Figure 3 shows the overlaid spectra of a surgical face mask and the 'best hit' spectrum from the PerkinElmer ATR polymer library. In this instance the best hit was polypropylene with a search score of 0.95 indicating a good match between the sample (red) and reference (black) spectrum.

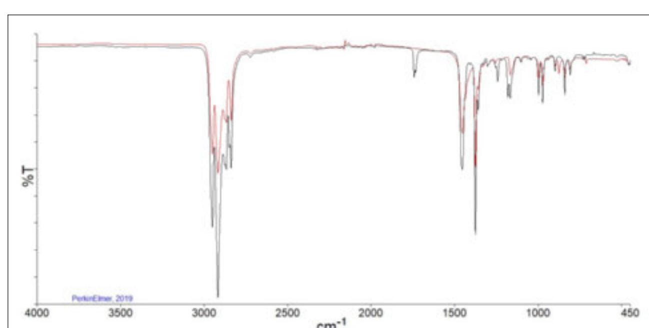

Figure 2. IR spectra of the four different glove materials covered by ASTM designations.

It is also possible to use the 'Compare' function in Spectrum 10 to determine how closely a material matches a specific known reference by producing a correlation value.

Thermogravimetric Analysis

Thermogravimetric Analysis (TGA) provides information about the weight loss of the sample as it is subjected to controlled temperature ramping. This allows users to see the weight loss events at a particular temperature which occur due to evaporation of volatile components or decomposition of materials. TGA also offers the ability to control the purge gas used allowing the sample to be heated under an inert environment (usually nitrogen), in air or pure oxygen or even using a reactive gas such as HCl.

Experimental

Samples were measured using the PerkinElmer TGA 8000™ (Figure 4) in tared ceramic crucibles. The temperature program used was dependant on the material being analyzed. For example, the polypropylene face masks had entirely decomposed at 520 °C whereas a neoprene glove did not completely decompose until around 800 °C. All experiments involved heating the material to 550-600 °C under nitrogen then switching the purge gas to oxygen to allow for complete combustion.

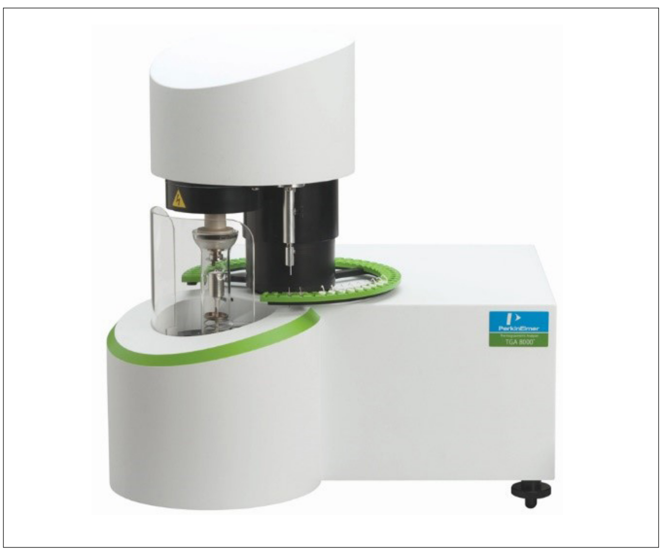

Figure 4. PerkinElmer TGA 8000 Thermogravimetric Analyzer.

Results and Discussion

The results from different pieces of PPE varied in complexity due to their respective compositions. Figure 5 shows the result from heating of a KN95 face mask (determined by IR spectroscopy to be polypropylene).

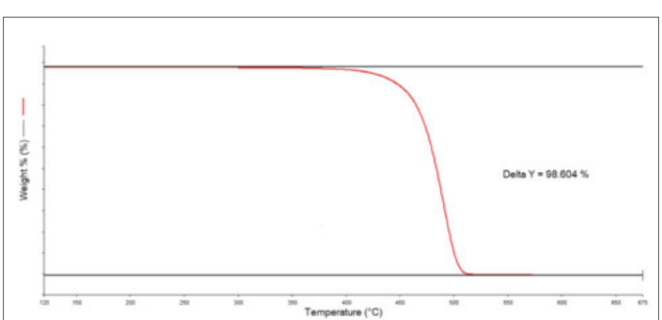

Figure 5. Thermogram of an N95 face mask. Conditions: Hold at 120 °C for three minutes under N_2, heat from 120 °C to 550 °C at 25 °C/min under N_2, heat from 550 °C to 675 °C under O_2.

Although the resulting thermogram is simple, it still provides useful data such as the onset temperature for the pyrolysis of the mask (390 °C) and the ash content (1.40%). On the other hand, materials such as neoprene and PVC produce slightly more complex thermograms. The results obtained from a sample taken from a neoprene glove are shown in Figure 6.

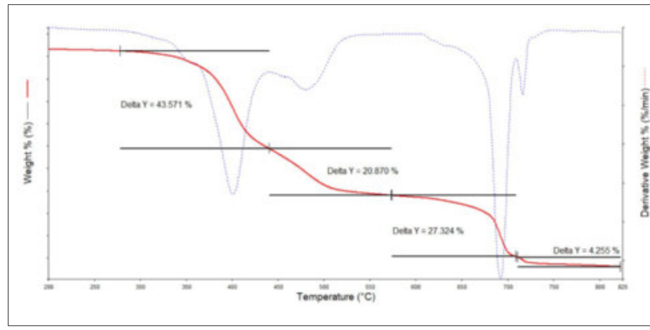

Figure 6. Thermogram of neoprene rubber. Conditions: Hold at 200 °C for three minutes under N_2, heat from 200 °C to 600 °C at 25 °C/min under N_2, heat from 600 °C to 825 °C under O_2.

The results from the experiment using neoprene shows several weight-loss steps, each corresponding to different degradation mechanisms. The first loss of 43.6% around 400 °C is likely due to the elimination of HCl from the polymer. The next weight loss of 20.9% at 480 °C is the decomposition of the remaining polymer. At 600 °C, the purge gas is switched to oxygen which allows the carbon formed during the degradation process to burn and form CO_2. It appears from the thermogram that this occurs in two separate steps, as can be seen from the derivative weight curve.

Finally, material from a plastic face shield was analyzed using the TGA 8000. In this case, two distinct weight loss events occurred, as can be seen in Figure 7.

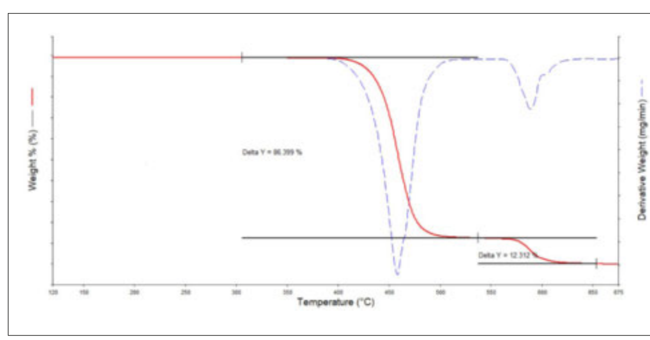

Figure 7. Thermogram of a PMMA face shield. Conditions: Hold at 120 °C for three minutes under N_2, heat from 120 °C to 550 °C at 25 °C/min under N_2, heat from 550 °C to 675 °C under O_2.

The first weight loss is due to the initial pyrolysis of the polymer used to produce the face shield – poly(methyl methacrylate) or PMMA. The second weight loss occurs after the purge gas is switched to oxygen and so corresponds to the burning of the residual carbon to form CO_2. The ash content of this polymer was determined to be 1.29%.

Differential Scanning Calorimetry

DSC measures the amount of heat required to increase the temperature of a sample relative to a reference. Due to common physical processes such as melting and crystallization being endothermic i.e. requiring energy, the temperature at which these occur can be accurately determined using DSC.

Experimental

Samples of nitrile gloves a surgical face masks were measured using the PerkinElmer DSC 6000 (Figure 8). As with TGA, the parameters used to measure the materials differed slightly based on the information required and the physical properties of the samples.

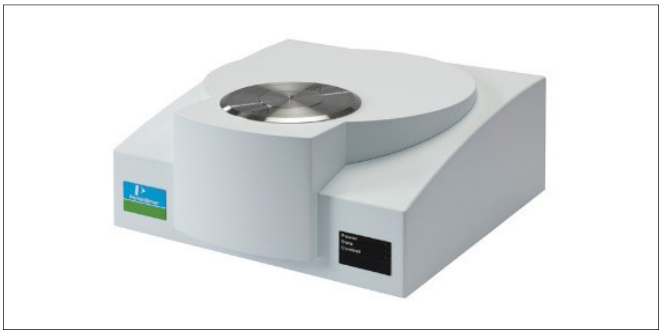

Figure 8. PerkinElmer DSC 6000.

Results and Discussion

The DSC analysis of the two different types of nitrile gloves gave the heat flow curves shown in Figure 9.

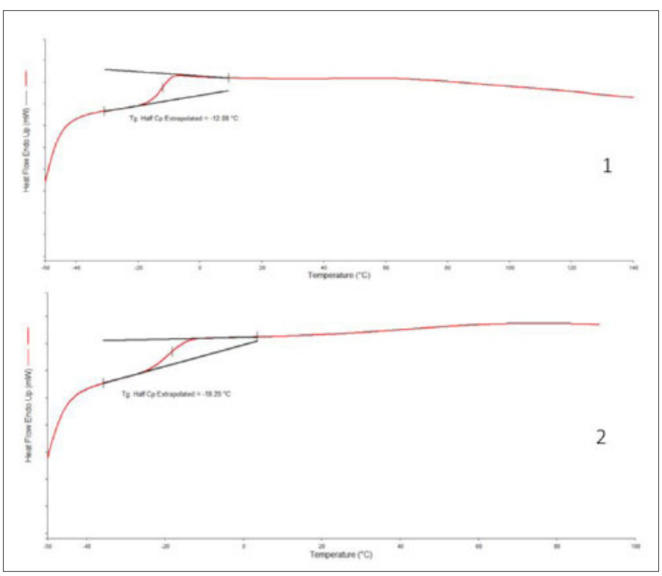

Figure 9. Heat flow curves of two different brands of nitrile glove. Conditions: Hold at -50 °C for five minutes under N_2, heat from -50 °C to 100 °C at 20 °C/min under N_2.

It can be seen from the measured heat flow curves that the glass transition temperature (Tg) of the samples does differ slightly, with sample 1 demonstrating a Tg of -12 °C and sample two demonstrating a Tg of -18 °C. This parameter can be determined simply using the calculations tab of PerkinElmer Pyris™ data analysis software.

In addition to measurement of the glass transition temperature, the melting point can also provide information allowing the user to positively identify a sample. For example, the heat flow curve from a face mask is shown in Figure 10.

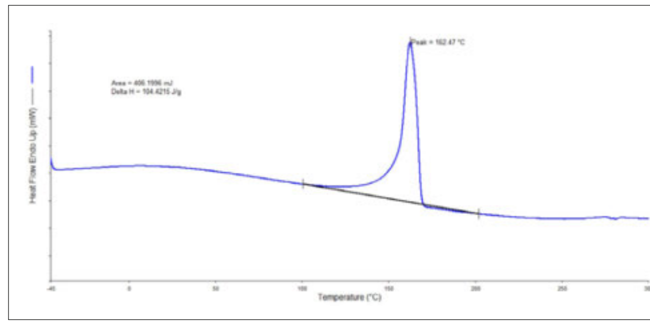

Figure 10. Heat flow curve of a surgical facemask. Conditions: Hold at -50 °C for one minute under N_2, heat from -50 °C to 300 °C at 20 °C/min under N_2.

It can be seen from this heat flow curve that the melting point occurs at 162.47 °C. This can be used to further confirm information obtained using IR spectroscopy (Figure 3) about the identity of the sample. The IR spectrum had a good match with a polypropylene reference spectrum and the DSC heat flow curve shows a melt occurring at 162 °C, consistent with polypropylene.

Conclusion

PerkinElmer offer a comprehensive material characterization solution portfolio, providing a one-stop-shop for the complete suite of instrumentation. Infrared spectroscopy and thermal analysis allow users to collect incredibly important information about their samples. In addition to the hardware on offer, the software offering (Spectrum 10 and Pyris for infrared and thermal analysis respectively) provide powerful data collection and analysis tools allowing the analyst to thoroughly investigate samples.

PerkinElmer, Inc.
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
www.perkinelmer.com

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