

# Morphological and chemical characterization of a non-woven fiber using electron and infrared microscopy

## Authors

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## Abstract

In this application note, the morphological and chemical characterization of a non-woven fiber sample is described. The SEM images suggest a fiber blend of at least two types of fiber, one of which has a possible sheath core structure. Through the library searching of the FTIR spectra, the non-woven fibers were determined to contain cellulose, PET and PE. The correlation profiles of the fibers confirm the presence of a sheath core structure, where the PET core is surrounded by the PE sheath.

## Application Benefit

The example demonstrates the complementarity between SEM and FTIR microscopy in material characterization: SEM excels in spatial resolution to understand materials' morphology, whereas FTIR microscopy offers molecular level insight into the underlying chemistry.

## Our solutions

- Nicolet iN10 MX Imaging Infrared Microscope, OMNIC Software,
- Phenom ProX Desktop Scanning Electron Microscope

## Introduction

Non-wovens are one of the fastest-growing segments of the textile industry and constitute a significant portion of the fiber industry. Multi-layer non-woven composites, laminates, and three-dimensional non-woven fabrics are commercially produced and used in a wide variety of industrial engineering, consumer, and healthcare products. The complexity of these fibrous materials mandates the use of multiple analytical techniques for their full characterization.

Scanning electron microscopy (SEM) and Fourier transform infrared (FTIR) microscopy are two widely used microscopy techniques for the characterization of non-woven materials. Using electrons as the radiation source, SEM offers higher spatial resolution (in nm scale) than other optical techniques. The large depth-of-field of SEM also yields images with a characteristic three-dimensional appearance beneficial for understanding the surface structure of a sample. While the difference in chemical composition at elemental level is manifested by the contrast in SEM images, the exact chemical identity cannot be readily determined. On the other hand, by leveraging the spatial specificity of microscopy and the chemical specificity of spectroscopy, FTIR microscopy can provide molecular level chemical annotation to sample morphology. The spatial resolution of FTIR microscopy, however, is limited by the diffraction limit of the infrared light to approximately 10  $\mu\text{m}$ . The combination of FTIR microscopy and desktop SEM can provide a holistic insight into materials' structure-function relationship from both the chemical and the morphological standpoints.



Thermo Scientific™ Nicolet™ iN10 MX Imaging Infrared Microscope



Thermo Scientific™ Phenom ProX Desktop Scanning Electron Microscope

In this application note, the structural and chemical characterization of a non-woven fiber sample using both FTIR microscopy and desktop SEM is illustrated. While SEM allowed for a quick visual discernment of the different constituents in the sample, FTIR microscopy offered chemical identification of the constituents, and thereby shed light on the associated production process.

## Experimental

**Sample:** The non-woven sample used in this study is an off-the-shelf hygiene wipe.

**Fourier Transform Infrared Microscopy:** A bundle of non-woven fibers was isolated under a stereoscope and flattened with the back end of a roller knife onto an aluminum mirror. They were analyzed with the Thermo Scientific™ Nicolet™ iN10 MX Imaging Infrared Microscope using a 15X objective. Two images were acquired with a linear array detector and another one with a single-point MCT-A detector. Spectra on all maps were acquired in reflection mode with 16 scans co-added at a 16 cm<sup>-1</sup> resolution.

**Scanning Electron Microscopy:** Images were acquired using a Thermo Scientific™ Phenom ProX Desktop Scanning Electron Microscope. A small piece of the non-woven fibers was cut from the bulk sample and mounted onto a standard ½ inch pin-mount SEM stub using double-sided carbon tape. For the acquisition of the SEM images, the standard backscattered electron (BSE) detector of the Phenom ProX Microscope was utilized. The main contrast mechanism on such images is based on elemental differences. To maximize the elemental contrast of the

organic samples, a relatively low electron beam voltage should be applied, which could result in lower quality images. However, due to the high-brightness electron source with which the Phenom ProX Desktop SEM is equipped, high-resolution images at 5 kV beam voltage were acquired without compromising their quality.

In this experiment, the inherent charge-reduction mode (low-vacuum operation) of the Phenom ProX Desktop SEM was utilized to prevent the non-conductive samples from charging. Using this approach combined with the advantage of the high-brightness electron source, sputter coating the sample with a metallic layer could be avoided, allowing investigation of the sample in its original form.

## Results and discussion

Figure 1 shows the SEM images of two different sections of the fiber sample using a backscattered electrons (BSE) detector. Heavy elements (high atomic number) scatter electrons more strongly than light elements (low atomic number), and thus appear brighter in the image.

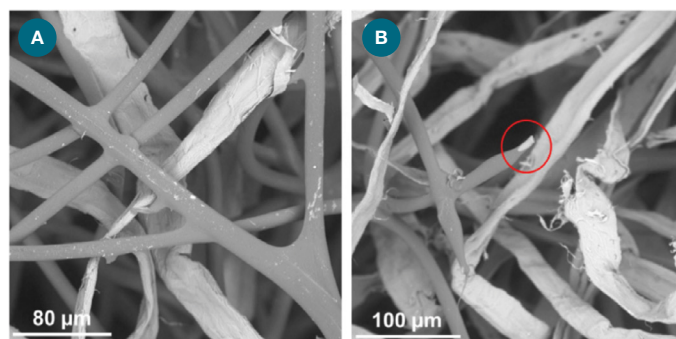


Figure 1: SEM images of (a) non-woven fiber bundle; and (b) an area where possible sheath core structure was observed (red circle).

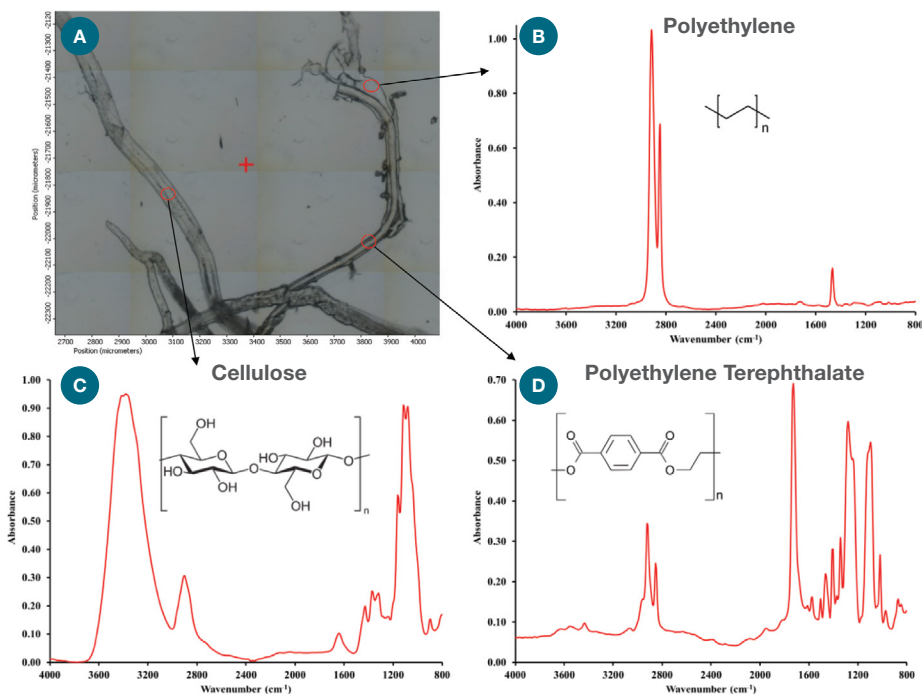


Figure 2: (a) Video image of the fibers obtained by Nicolet iN10 MX Microscope. Representative spectra and chemical structure of (b) polyethylene, (c) cellulose, and (d) polyethylene terephthalate. Red circles show the locations where the representative spectra were taken.



The contrast in the grayscale image signifies different chemical compositions. Figure 1a indicates that at least two types of fibers are present in the sample: one with smooth textures in dark color and one with wrinkled texture in white color. Closer examination (red circle in Figure 1b) further reveals that the dark-colored fibers are likely comprised of two different chemical compositions: a possible sheath core structure with a dark outer layer and a white inner core.

To investigate the chemical origins of the different types of fibers observed by SEM, the sample was analyzed by FTIR microscopy. Figure 2 shows the video image (approx. 1.2×1.5 mm) and the representative spectra taken at three different spots of the sample. A library search indicates that the selected spectra (Figures 2 b–d) match polyethylene (PE), cellulose and polyethylene terephthalate (PET), respectively.

The representative spectra shown in Figures 2 b–d were used as the reference spectra to construct the correlation profiles, wherein the red color represents a high degree of correlation, i.e., a greater similarity, with the respective reference spectra. Figure 3 shows the correlation images overlaid with the video images. Of particular interest is Figure 3c where a noticeable PE moiety resides adjacent to the PET fiber, supporting the hypothesis of a sheath core structure suggested by SEM (Figure 1b): a high

melting point (~250 °C) PET core surrounded by a low melting-point (~120 °C) PE sheath. During production when the fibers are heated, the sheath layer melts and adheres to each other at the junctions. A crosslinking network is formed to achieve the desired mechanical strength while maintaining the structural integrity.

## Conclusions

This application note describes the characterization of a non-woven fiber sample. The contrast in the SEM images suggests a fiber blend of at least two types of fiber, one of which has a possible sheath core structure. FTIR microscopy provides corroborating evidence from the chemistry perspective to support the observations by SEM. Through the library searching of the FTIR spectra, the non-woven fibers were determined to contain cellulose, PET and PE. The correlation profiles of the fibers confirm the presence of a sheath core structure, where the PET core is surrounded by the PE sheath. The example clearly demonstrates the complementarity between SEM and FTIR microscopy in material characterization: SEM excels in spatial resolution to understand materials' morphology, whereas FTIR microscopy offers molecular level insight into the underlying chemistry. The great analytical power unleashed from the combination of these two microscopy techniques should be welcomed by those in research and development as well as quality control/quality assurance across many industries.

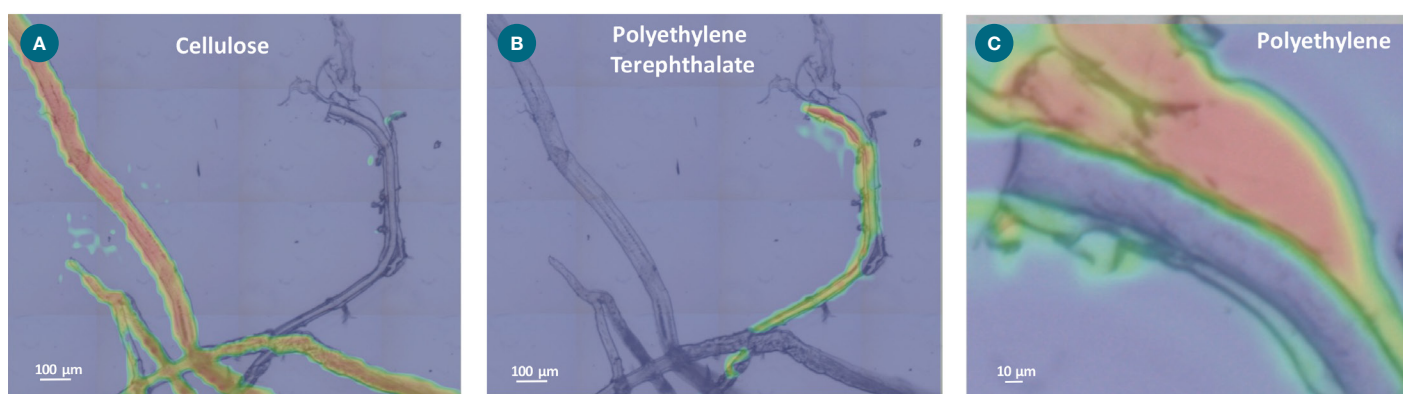


Figure 3: Chemical correlation maps overlaid with video image of fibers. Correlation maps correspond to where (a) cellulose, (b) polyethylene terephthalate and (c) polyethylene are located across the visual image.

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