

Author:

Karen Sam





# Characterizing Textiles using Evolved Gas Analysis and an MSChrom Database

#### **Application Note**

**Textiles** 

#### **Abstract**

This application note demonstrates characterization of woven jean fabric using Evolved Gas Analysis coupled with Multi-Step Pyrolysis GC-MS.

#### Introduction

Fiber identification can be important for many different fields such as the textile industry, fashion design, heritage science, and forensic science. Here, evolved gas analysis (EGA) and multi-step pyrolysis were used to help characterize woven jean fabric. The process of fabric identification was performed by studying quick (10 minute screening) EGA in Mestrelab's Mnova MSChrom with CDSPlugin and the CDS EGA database. Then, multi-step pyrolysis provided additional confirmation and details.such as the presence of organic dyes.

#### **Experiment Setup**

A small piece of woven jean fabric was trimmed down with scissors to 100  $\mu$ g and loaded into a DISC tube for analysis. A fused silica transfer line was used to connect the GC inlet to the MS detector in a preliminary EGA run. After which, a 30 meter long 5% phenyl capillary column was used for multi-step pyrolysis. A vent-free adapter was installed to enable a fast switch between the fused silica and the column without losing vacuum in the mass spectrometer.

#### **EGA**

# Pyroprobe 6150 Autosampler GC-MS

Initial: 50°C Column: Fused silica (1m x 0.10mm)
Final: 1000°C Carrier: He 1.25mL/min 75:1 spl

Ramp Rate: 100°C per min Injector: 360°C Oven: 300°C
Transfer Line: 325°C Ion Source: 230°C Valve Oven: 300°C Mass Range: 35-600amu

**Multi-Step Pyrolysis** 

**Pyroprobe 6150 Autosampler GC-MS** Column:

425°C 60 sec 5% phenyl (30m x 0.25mm x 0.25μm)

600°C 60 sec Carrier:

He 1.25mL/min, 75:1 spl

Interface: 300°C Injector: 360°C

Transfer Line: 325°C Oven: 40°C for 2 minutes

Valve Oven: 300°C 12°C/min to 320°C (10min)

Ion Source: 230°C Mass Range: 35-600amu

## **Results and Discussion**

In the first step, the DISC temperature was ramped up at 100 °C/min from 50°C to 1000 °C and the GC oven was kept at 300°C for a 10 minute screening EGA. Data analysis was performed in Mestrelab Mnova's MSChrom with the CDS Plugin. The results of the EGA are shown in Figure 1. Two regions of interest were seen, between 350°C and 425°C, and between 425°C at 600°C. Here the CDS EGA database (p/n 10A1-3025) was used to identify the fiber types by investigating the two EGA regions separately. When the mass spectrum of the first region was combined (Figure 2) and compared against the CDS EGA database, top matches included cellulose (which makes up cotton), and cotton (Figure 3). Matches for polyester products, including Faux Silk, were found for the second region (Figure 4).

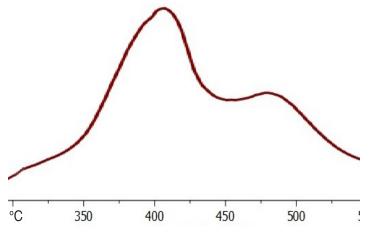


Figure 1. EGA of Woven Jean Fabric.

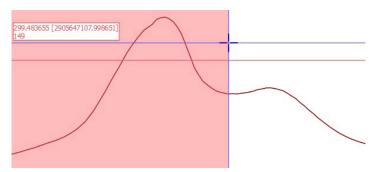


Figure 2. Combining Mass Spectra of EGA.

	Server	Database	Recordid	Item Type ^	Score	Polymer Name Cellulose
1	localhost:5531	CDS-EGA	27	Mass Spectrum	984	Cellulose
2	localhost:5531	CDS-EGA	176	Mass Spectrum	972	Corn Starch
3	localhost:5531	CDS-EGA	192	Mass Spectrum	966	Bamboo Fabric
4	localhost:5531	CDS-EGA	400	Mass Spectrum	950	Maltodextrin
5	localhost:5531	CDS-EGA	58	Mass Spectrum	943	Rayon
6	localhost:5531	CDS-EGA	196	Mass Spectrum	936	Cotton

Figure 3. CDS EGA database search results for first region of EGA in Figure 1.

	Server	Database	RecordId	Item Type	Score ~	Polymer Name	
1	localhost:5531	CDS-EGA	223	Mass Spectrum	985	Faux Silk	
2	localhost:5531	CDS-EGA	580	Mass Spectrum	982	Amorphous Pellet Recycle PET	
3	localhost:5531	CDS-EGA	589	Mass Spectrum	982	Carpet, polyester	,

Figure 4. CDS EGA database search results for second region of EGA in Figure 1.

When the matches were stacked with the woven jean fabric for a comparison, each temperature region aligns with its respective match (Figure 5).

Then, using the EGA as a guide, multi-step pyrolysis was performed to both confirm the EGA result and to gain additional information. Temperatures at the end of each region of interest, 425°C and 600°C, were chosen to be sure adequate thermal energy was used for multi-step pyrolysis GC-MS (Figure 6). At 425°C, levoglucosan, a characteristic peak for cellulose, was identified by the NIST library. At 600°C, both benzoic acid and vinyl benzoate, pyrolysis products for polyester, were identified.

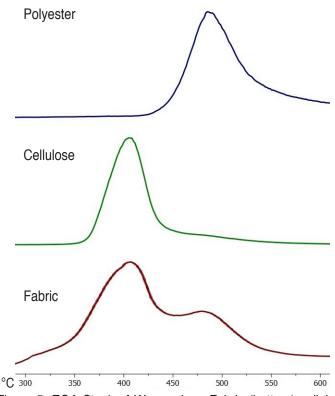


Figure 5. EGA Stack of Woven Jean Fabric (bottom) cellulose database match (center), and polyester database match (top).

Additionally, indigo dye, used to give fabric a deep blue color, was found in both pyrograms.

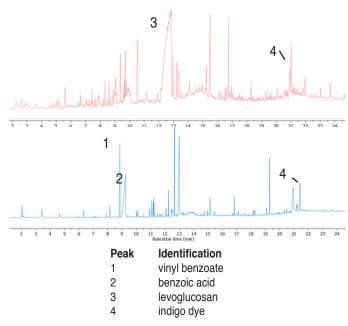


Figure 6. Woven Jean Fabric at 425°C (top), and 600°C(bottom).

### Conclusion

EGA with the CDS EGA database and Mnova's MSChrom plus multi-step pyrolysis GC-MS was useful in determining that the woven jean fabric contains both cotton and polyester. These two techniques provide essential information for polymer identification. An EGA used with the CDS EGA database, provides quick information and serves as a guide for multi-step pyrolysis, which in turn provides in-depth information.