

August 9, 2007

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### Report: 07-38720

### Introduction

A sample of debris from the Sort Pro dry-brush sheet cleaning system was received at Food Safety Net Services for characterization of the chemical and physical components of the material. The material is recovered from the dry brush process as sheets are returned to the facility for cleaning prior to reuse. The sheets were described as separators used in the processing and shipment of empty blank beverage cans. The sample, as received, is shown in Figure 1.



Figure 1. Debris Sample as Received.

### **Macroscopic Observations**

The sample was evaluated by visual means to detect and remove larger fragmented pieces of material that would not require additional analytical methods to establish the character

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of the material. The evaluation detected pieces of plastic from a minimum of four separate sources. These included several long green pieces of plastic strapping that were dissimilar to any other pieces in overall mass. Also recovered were numerous elastic strings, glass, pieces of gray and green plastic, portions of plastic film, wood chips, and numerous synthetic fibers of varying diameter and length. Representative examples of these components are shown in Figures 2 through 7.

Figure 2. Glass, Plastic and Fibers. Figure 3. Elastic Strings and Fibers.



Figure 4. Plastic Film and Fibers.

Figure 5. Gray and Green Plastic, Wood Chip and Elastic Strings.





Figure 6. Elastic Strings, Plastic Film, and Fibers.

Figure 7. Green Plastic Strapping Material

### **Microscopic Observations**

After removal of macro-sized debris noted above, the sample was subjected to analysis by stereoscopic microscopy at 10 and 30 X magnifications, and light and phase contrast microscopy at 100, 400 and 1000 X magnifications. The stereoscopic evaluation further showed the detail of the macroscopic debris and revealed the presence of insect fragments and plant material. Figures found at Appendix A, show the macroscopic debris in greater detail and show the presence of insect parts. Each left-right pair is at 10 and 30 X magnifications respectively. Light microscopy showed the additional presence of pollen, microbes and rodent or insect hairs as seen in Figures 8, 9 and 10.

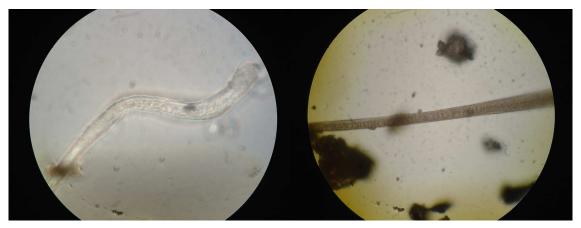


Figure 8. Rodent/Insect Hair 1000 Magnification - Light Microscopy.

Figure 9. Rodent/Insect Hair 1000 Magnification - Phase Contrast Microscopy.

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Figure 10. Pollen at 1000 Magnification - Light Microscopy.

# **Microbiologic Evaluation**

The debris sample was cultured on to Standard Methods Agar (SMA) supplemented with chloramphenicol (to suppress bacterial growth) to determine if mold spores were present. Mold was detected at 2,000 Colony Forming Units per gram of debris (CFU/g) which was subsequently identified by slide culture and standard light microscopy. The mold recovered was identified as *Aspergillus* species and is shown in figure 11.

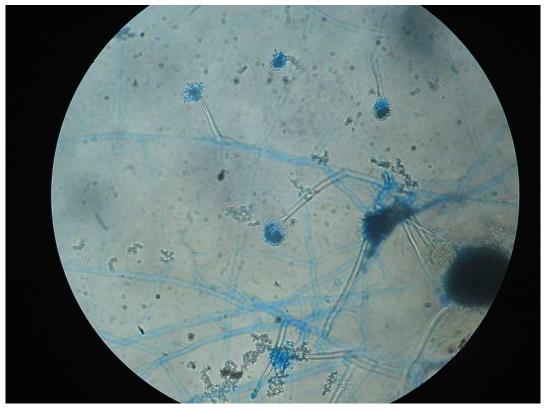


Figure 11. Slide Culture of Aspergillus spp. Recovered from Debris

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#### **Physical Evaluations**

Physical evaluation of the material was subcontracted for characterization by x-ray diffraction (XRD), Scanning Electron Microscopy with Energy Dispersive X-ray Analysis (SEM/EDS) and Fourier Transform Infra-red Spectroscopy (FT-IR). The report submitted to Food Safety Net Services by the subcontractor is included at Appendix B.

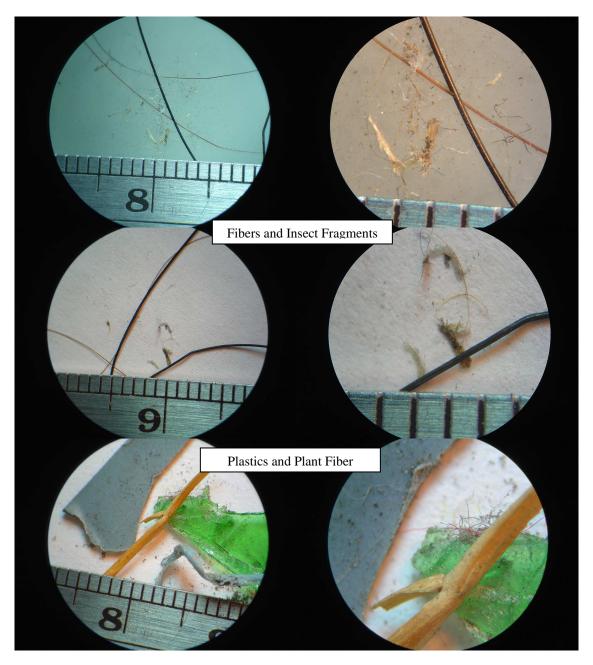
#### Conclusions

Macroscopic, microscopic, microbial and physical evaluations of the submitted sample indicate that the debris is an amorphous mixture of many materials ranging from large pieces of glass and plastics to microscopic fragments of rock, metals, minerals, insect and rodent fragments, pollen, mold spores and many unknown materials. Further, the mold spores detected were identified as an *Aspergillus* species many of which are capable of producing mycotoxins and many of which are implicated as allergens. The material contains numerous minerals and metals, amongst which were found high levels of oxygen (29.1%), carbon (24.5%) and silica (21.2%). These elements are most commonly found in terrestrial materials (rock etc.) such as feldspars and also found in glass. The carbon was detected primarily in the cotton fibers found through out the debris.

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Robert M. Levy, Technical Supervisor

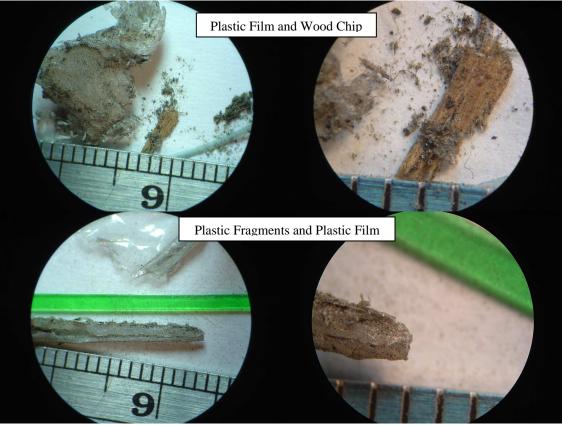




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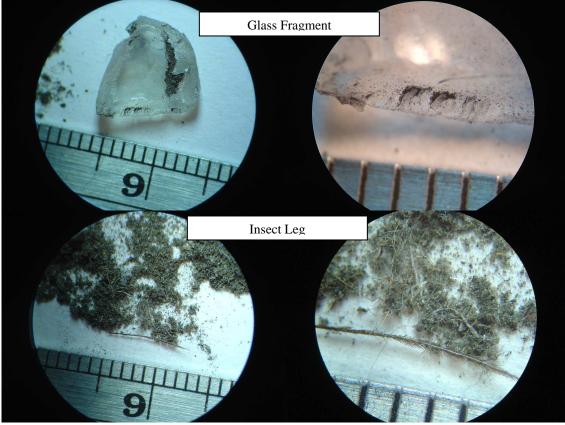
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### **Characterization and Analysis of a Fibrous Powder Sample**

### **Introduction:**

A powder sample was received at the laboratory. It was characterized to study their phase and chemical make-up. It was analyzed by x-ray diffraction (XRD), Scanning Electron Microscopy with Energy Dispersive X-ray Analysis (SEM/EDS) and Fourier Transform Infra-red Spectroscopy (FT-IR). The following report summarizes the findings:

### **Materials and Methods:**

The following sample was analyzed:

#### 1) 07-38720-001

The following two techniques were used to characterize the powder samples. **1) X-ray Diffraction Analysis**:

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X-ray diffraction (XRD) is a crystal structure analysis method using the atomic arrays within the crystals as a three-dimensional diffraction grating to diffract a monochromatic beam of x-rays. The angles at which the beam is diffracted are used to calculate the interplanar atomic spacings (d-spacings) giving information about how the atoms are arranged within the crystalline compounds. Even if materials are chemically similar, they can be differentiated by their crystallographic structures. These patterns (and their d-spacings) are compared to over 65000 data entries in the International Powder Diffraction File (PDF) data base.

The powders materials were gently ground in an agate mortar and pestle. The powder sample, packed in a holder, was analyzed in a Scintag powder diffractometer, equipped with a solid state detector, using copper K-alpha radiation at 45 kV and 35 ma. It was scanned from 5 to 55 degrees two-theta at 1.5 degrees/minute.

# 2) Scanning Electron Microscopy /Energy Dispersive X-ray Analysis (SEM/EDS)

In this technique, an electron microscope with an energy dispersive X-ray spectrometer is used for analysis. The electron beam in the microscope causes specimens to emit x-rays including those from the k, l and m atomic shells. Spectrometer counts of these x-rays, which are said to be "characteristic" of the elements present in the specimen, can be used to calculate composition for a full qualitative analysis. The analysis is non-destructive and is accurate to  $\sim 1$  %.

The powder was sprinkled on a carbon tape and mounted on a holder for analysis by Scanning Electron Microscopy (SEM). The sample was bombarded with 20 kV electrons, in a Phillips SEM microscope attached with a EDS, causing the emission of characteristic X-rays for each element. These X-rays were detected and identified by the integral energy dispersive optics of the X-ray spectrometer.

# **Results and Conclusions:**

X-ray diffraction (XRD) pattern shows that the powder is mostly quartz (SiO2) and Feldspars (Figure 1).

SEM Photographs are shown in attached figures. All well crystallized plates and crystals are quartz. The fibers are mostly carbon containing cotton fibers.

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EDXRF pattern is shown in Figure 2 and elemental concentrations are also tabulated. It contains mostly silicon (Si) with calcium and iron

FT-IR analysis (Figure 3) shows that most of it (1000 cm-1 band) is from Si-O-Si bend from feldspars, silicates or glass. There is very little C-H, may be from some type of hydrocarbons

Elements	07-38720-001
(wt. %)	
Oxygen	29.1
Carbon	24.5
Magnesium	1.1
Sodium	0.9
Aluminum	4.6
Silicon	21.2
Phosphorous	<0.2
Sulfur	<0.2
Calcium	7.4
Potassium	2.1
Iron	9.1

 Table 1: Chemical composition of powder Sample

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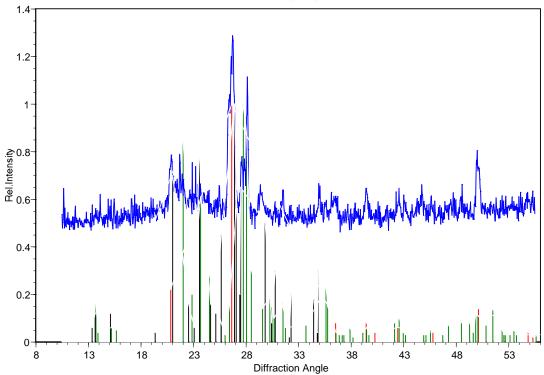
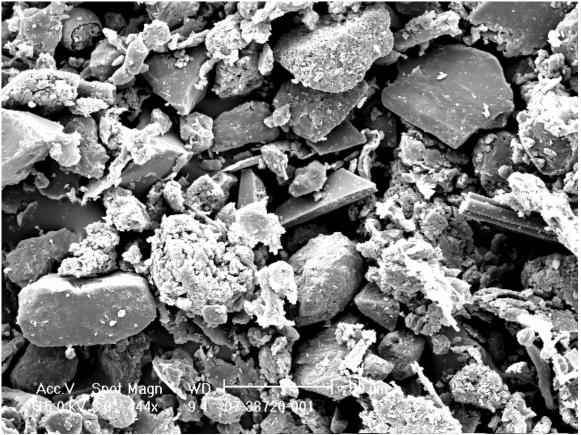


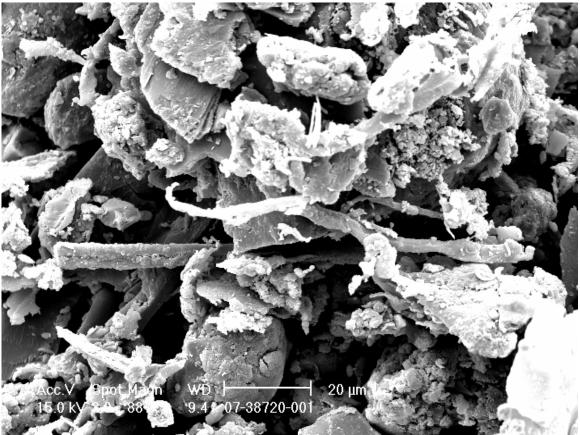
Figure 1: XRD pattern for sample 07-38720-001 with stick patterns for quartz (red) and feldspars (green and black)





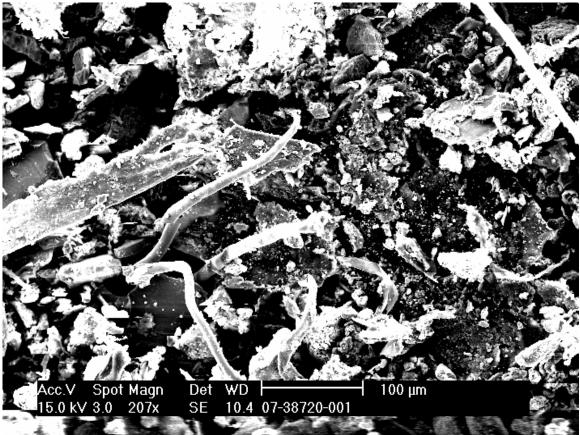
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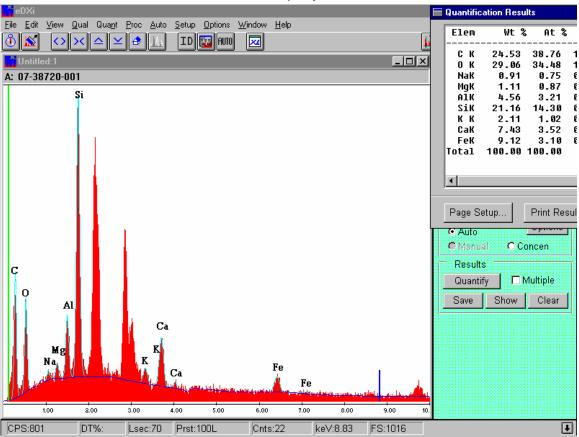


Figure 2: ED X-ray Fluorescence Pattern for the powder sample FT-IR for 07-38720-001

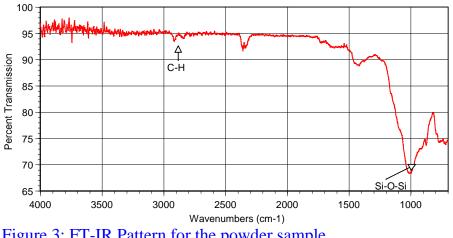


Figure 3: FT-IR Pattern for the powder sample

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