Failure Analysis of Hand Sanitizing Wipe Packaging by Pyrolysis-GC-MS

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Application Note

Polymer, Packaging

Abstract

This application note investigates the difference on packaging between normal and inferior hand sanitizing wipes using EGA and multi-step pyrolysis GC-MS techniques.

Introduction

In 2020, the WHO declared the coronavirus a pandemic. Since then, there has been unprecedented demand for disinfecting products including hand disinfecting wipes that outpaced the maximum manufacturing capacity by reputable suppliers. This global shortage created an opportunity for the inferior and even counterfeit product to enter the market. One of the noticeable differences between the normal and inferior products is the packaging. Normal individually wrapped wipe has a packaging that contains multiple layers, where the center layer is designed to withstand ingredients and chemical solutions, and the outer layer is suitable for ink-labeling. As a comparison, the packaging of an inferior product could have smudged labeling due to the moisture and chemical which has leaked from inside the packaging. This leak is often caused by quality issue from the center layer. Pyroprobe 6150 is a perfect tool to study the root cause of such packaging failure.

Experiment Setup

Two individually wrapped hand sanitizing wipe samples were commercially acquired. One is a P brand product and the other is a no brand product. The ink on the outside of the P brand product was in perfect condition, but the packaging on the no brand had smudged ink. For analysis, one 1 mm hole punch of each sample was first added into DISC (Drop-In-Sample Chamber) tubes and then analyzed using Evolved Gas Analysis (EGA) as an initial screening step. Using the information from this step, multi-step pyrolysis was followed. Then, a reproducibility study was also performed on the P brand packaging at a setpoint of 400°C.

EGA

Pyroprobe with DISC	
Initial:	100°C
Final:	800°C
Ramp Rate:	100°C per min
DISC Interface:	300°C
Transfer Line:	300°C
Valve Oven:	300°C

GC/MS	
Column:	fused silica 1m x 0.10mm
Carrier:	Helium 1.25mL/min 80:1 split
Oven:	isothermal 300°C
Ion Source:	230°C
Mass Range:	35-600amu

Multi-step Pyrolysis Pyroprobe with DISC	
DISC:	400°C 1 min 550°C 1 min
Interface:	300°C
Transfer Line:	300°C
Valve Oven:	300°C

GC-MS	
Column:	5% phenyl 30m x 0.25mm
Carrier:	Helium 1.25mL/min 80:1 split
Injector:	360°C
Oven:	40°C for 2 minutes 12°C/min to 300°C hold 10 min
Mass Range:	35-600amu

Results and Discussion

Using the Application Roadmap as a guide, EGA was first performed on both the P brand and no brand product packaging to help choose temperatures for multi-step pyrolysis. With this fast screening technique, the DISC temperature was ramped up at 100 °C/min from 100 °C to 800 °C and the GC oven was kept isothermal at 300°.

The overall EGA results of both P brand and no brand packaging appeared similar. Both EGA had a single peak at 550°C as shown in Figure 1. However, the ion compositions from the mass spectra were different, where the EGA spectrum on the P brand contained significant amount of m/z 250. When isolating m/z 250, the P brand product showed additional peak at 400°C in Figure 2.

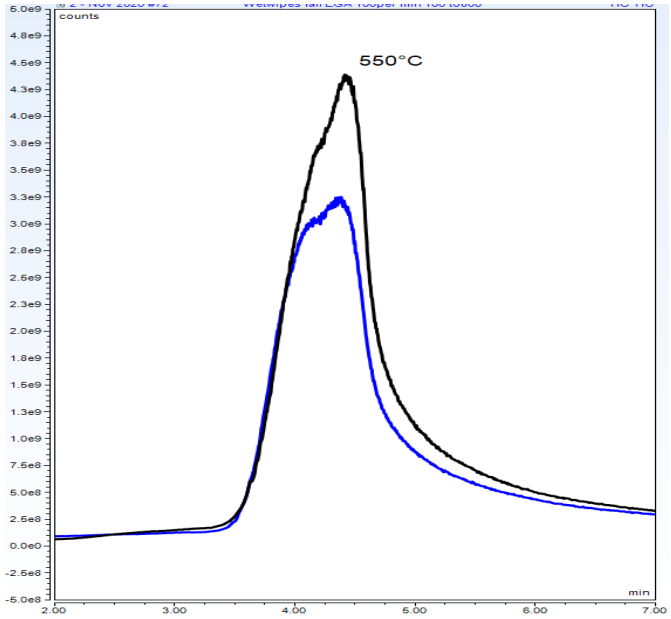

Figure 1. Evolved Gas Analysis of P brand (black) and no brand (blue) packaging.

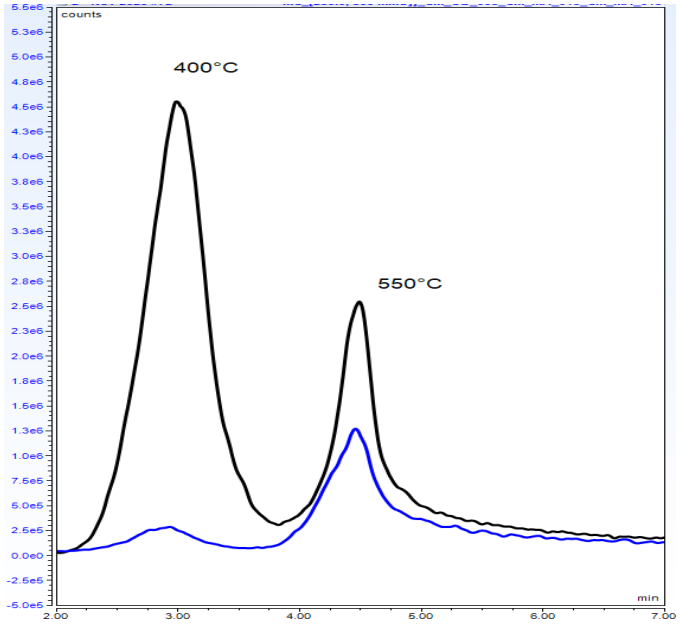

Figure 2. m/z 250 EGA run of P brand and no brand packaging.

To investigate this key difference seen in the EGA, temperatures of 400°C and 550°C were chosen for multi-step analysis. Each of these runs provided information about the composition of the packaging. Isomers of isopherone diisocyanate (IPDI) in each package represents a polyurethane component at 400°C (Figure 3), and 550°C (Figure 4), a repeating pattern of oligomers represented polyethylene, and pyrolysates of the PET layer can be seen amongst this.

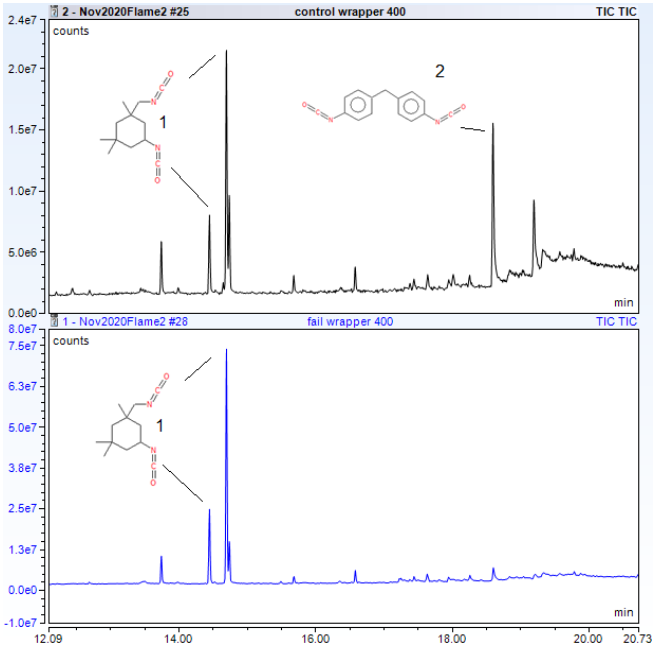

Figure 3. TIC of P brand (top) and no brand (bottom) packaging chromatogram at 400°C. Peak # Identification: 1 Isopherone Diisocyanate (IPDI), 2 Methylene Diisocyanate (MDI).

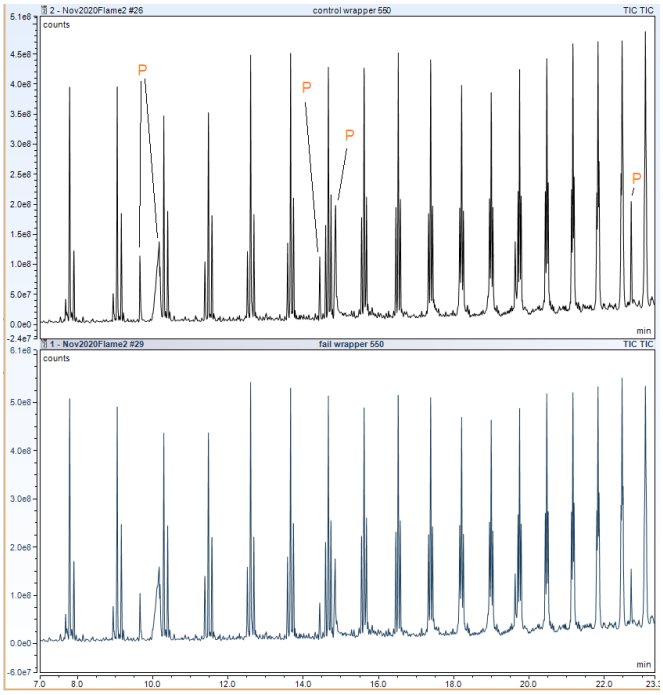

Figure 4. P brand (top) and no brand (bottom) packaging chromatogram at 550°C, after 400°C. Peaks labeled “P” are pyrolysis products of Polyethylene terephthalate (PET).

Consistent with the EGA results, differences between P brand and no brand packaging was most evident at 400°C. While both packages had IPDI, the P brand package had a larger peak for Methylene diphenyl diisocyanate (MDI), whose base peak in its mass spectrum was 250. The decrease in MDI in the no brand package indicated a lack of lamination processing.

The Pyroprobe is a great quantitative tool in polymer analysis. While a sample amount under 100 μg with a high split ratio (100:1) is generally recommended for pyrolysis, only the polyurethane layer pyrolyzed at 400°C, so a larger sample size, 230 μg (1.5mm hole punch), and a lower split ratio (30:1) was used to increase sensitivity. Ten replicates of the P brand packaging pyrolyzed at 400°C are shown in Figure 5. A Peak area ratio on m/z 110 of the IPDI isomer peaks was found to be around 2% as Table 1, which qualifies the Pyroprobe for quantitative studies.

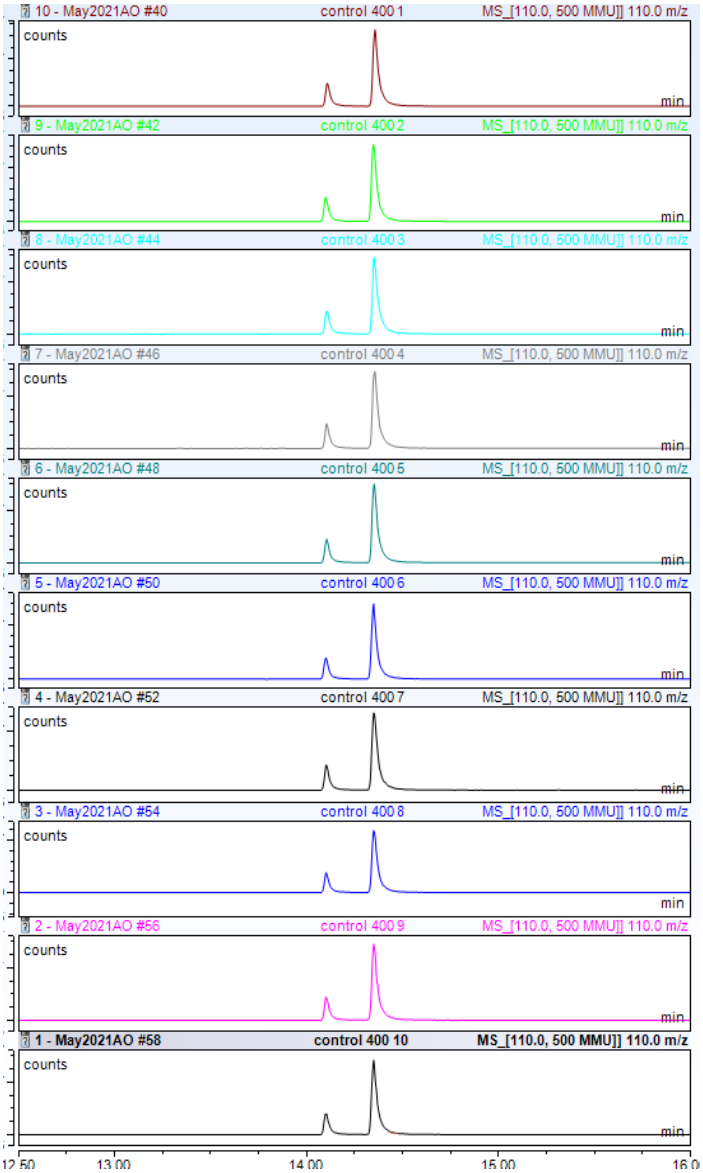

Figure 5. Replicates m/z 110 in P brand packaging for IPDI at 400°C.

Table 1. Peak area ratios of IPDI isomers in P brand packaging at 400°C.

Replicates	IPDI Peak Area Ratio
Rep 1	3.73
Rep 2	3.68
Rep 3	3.62
Rep 4	3.81
Rep 5	3.87
Rep 6	3.74
Rep 7	3.65
Rep 8	3.81
Rep 9	3.72
Rep 10	3.64
Average	3.73
RSD (%)	**2.20**

Conclusion

EGA together with Multi-step pyrolysis disclosed differences between packagings from a branded and non branded hand sanitizing product. The packaging failure in the non branded product was identified as lack of a polyurethane chemical, signaling a problem in the lamination process. The data shown is highly reproducible for quantification studies, and so provides an effective solution for various polymer applications including failure analysis.