APPLICATION NOTE

Hyphenated Technology

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Detection of Additive Used in Gloves by TG-GC/MS Hyphenation System

Introduction

Producing consistently high quality gloves with minimum cost has always been the top mission for glove manufacturers. The quality of gloves is associated with the raw materials used and its manufacturing processes. For this reason, understanding the various rubber additives and raw materials used and its application advantages has becoming very important.

Glove manufacturing processes begin by mixing latex with latex compounding ingredients which include sulfur, zinc oxide, accelerators, pigments, stabilizers, dewebbing agent and antioxidant. The mixture is then leave to cure, before it undergoes glove dipping, vulcanization, and leaching process that produce stretchable gloves.

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One of the additives commonly used in glove manufacturing is dispersing agent, which keep solid particles in latex dispersion from agglomerating or joining with other particles to form a cluster[1]. In this study, two glove samples formulated with and without dispersing agent are analyzed to see if a difference can be seen.

Figure 1. TG-GC/MS (Clarus SQ 8 GC/MS, TL8500, TGA 8000).

Experimental

The experiment was conducted using PerkinElmer TGA 8000™ hyphenated with Clarus® SQ 8 GC/MS. The hyphenation transfer line system used is TL-8500. A survey run was performed using TGA system to characterize the thermal degradation of dispersing agent.

The percent weight loss (TG) and derivative percent weight loss (DTG) curves of dispersing agent were plotted against temperature in Figure 2. As sample is in liquid form, the weight loss plot has shown immediate weight loss due to the drying or vaporization of volatile content of about 30% between room temperature to 200 °C. From the TG-DTG curves, it was determined that dispersing agent has shown greatest decomposition rate at approximately 455 °C, 28%/min.

Therefore, in subsequent runs, the evolved gas from each glove samples were analyzed at the same temperature region using TG-GC/MS hyphenation system. The setting of the experiment is shown in Table 1.

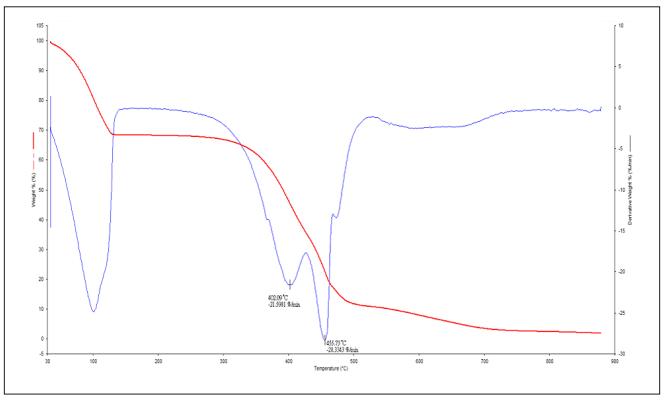

Figure 2. Dispersing agent survey run – Percent weight loss (in red) and derivative weight loss (in blue) curves versus temperature.

Table 1. Hyphenation system experimental setup.

TGA Program		
TGA 8000	Purge gas	Helium at 40 ml/min
	Temperature profile	Heat from 30 to 650 °C at 50 °C/min
	Sample size	10 ~ 15 mg
Transfer Line Program		
TL 8500	TL Temperature	280 °C
	Pump rate	70 mL/min
GC/MS Program		
Clarus 680 GC	GC column	Elite-5 MS, 30 m * 0.25 mm * 0.25 µm
SQ8 MS	Temperature profile	50 °C for 1 min, 10 °C/min to 300 °C, hold for 10 min
	Scan range	40-300 amu, SIR 44, SIR 54
	Ionization mode	EI, 70eV
	Ion source Temp	230 °C

Results

Glove samples A and B, one formulated with dispersing agent, another one without are analyzed. Figure 3 (a) and (b) showed the weight loss profile of both samples, with very similar trend at the temperature region of interest. A more sensitive instrument is in need to differentiate the two.

The evolved gas from each sample was analyzed by GC/MS to determine if hyphenation technique would confirm the presence or absence of trace amount of dispersing agent in glove samples. Figure 4 demonstrates the MS data obtained from the analysis of each sample.

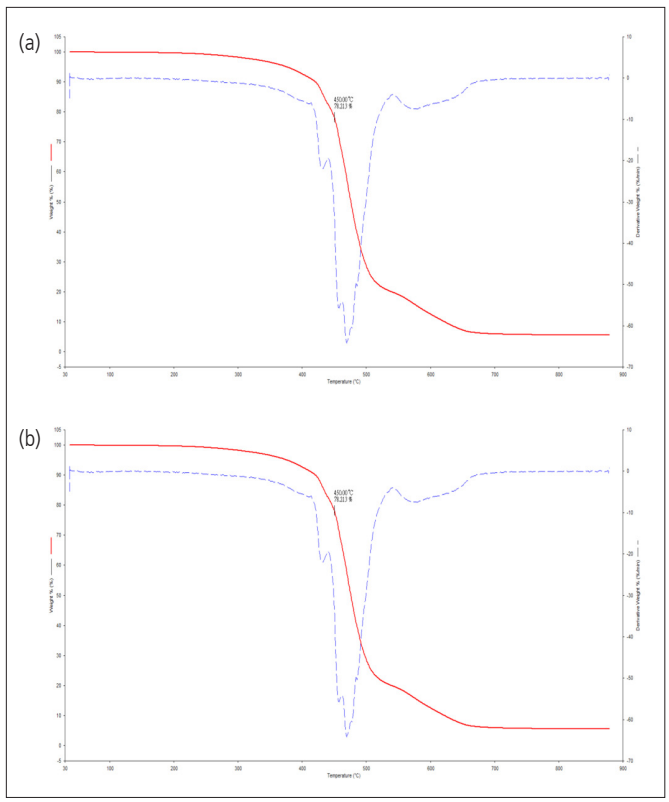

Figure 3. Weight loss profile of (a) Glove A and (b) Glove B.

It can be seen that the gas evolved are very complex, with major compounds detected at Retention Time (RT) between two to four minutes for both glove samples. However, it is not comparable to those eluted out from dispersive agent. This is because the major compounds detected in glove samples are mainly contributed by the decomposition of latex. As the content of dispersing agent used is very low, it becomes challenging to resolve the peaks coming from dispersing agent from those of latex gloves.

Hence, two other peaks detected between RT between 11 to 12 minutes in the TIC diagram of dispersing agent (Figure 5) were identified and used for further interpretation.

Figure 6 demonstrated three major mass to ion charge ratio (m/z) detected at RT 11.205 minute and 11.563 minute which are being monitored. Profile searching via MS software, has shown evidence of low levels of dispersing agent detected in Glove B, that is not seen in Glove A, refer to Figure 7.

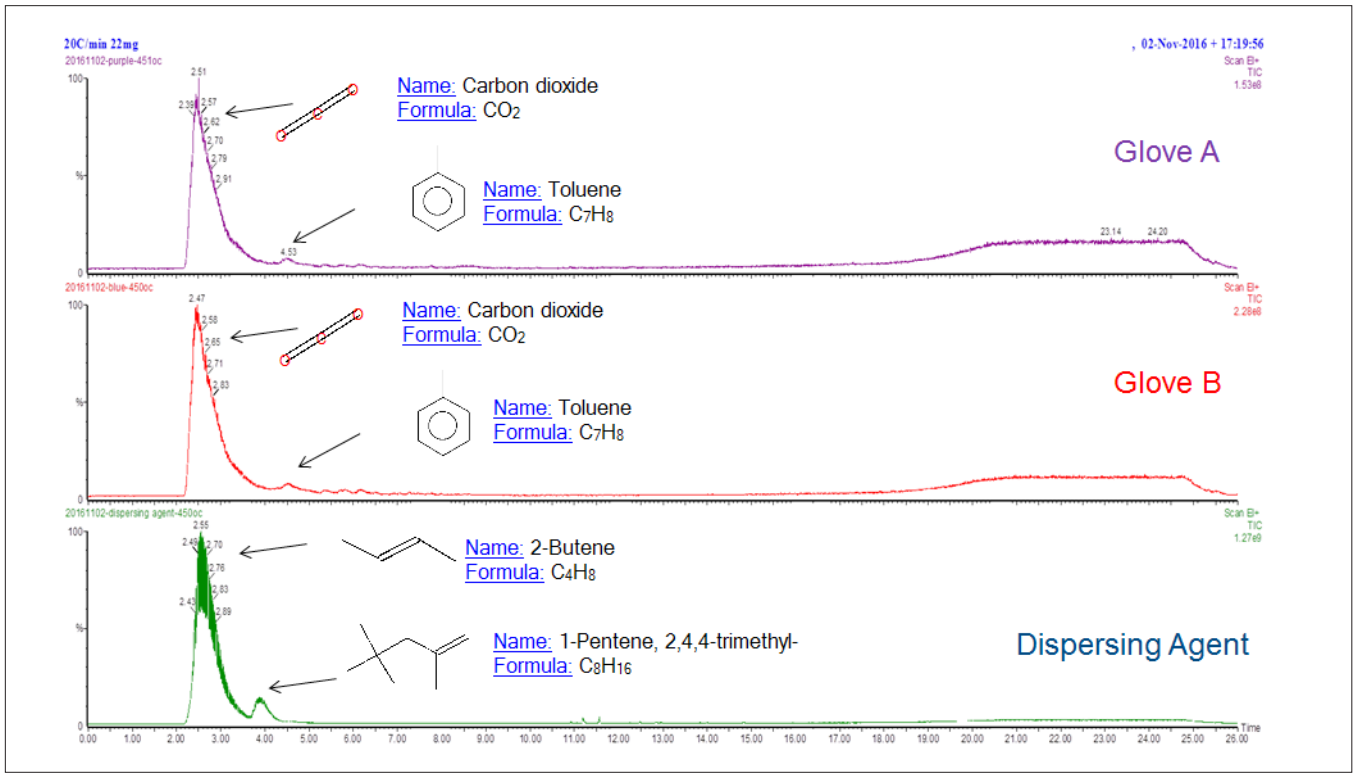

Figure 4. Comparison of GC/MS TIC Diagram of the evolved gas of Glove A, Glove B and dispersing agent injected at 450°C.

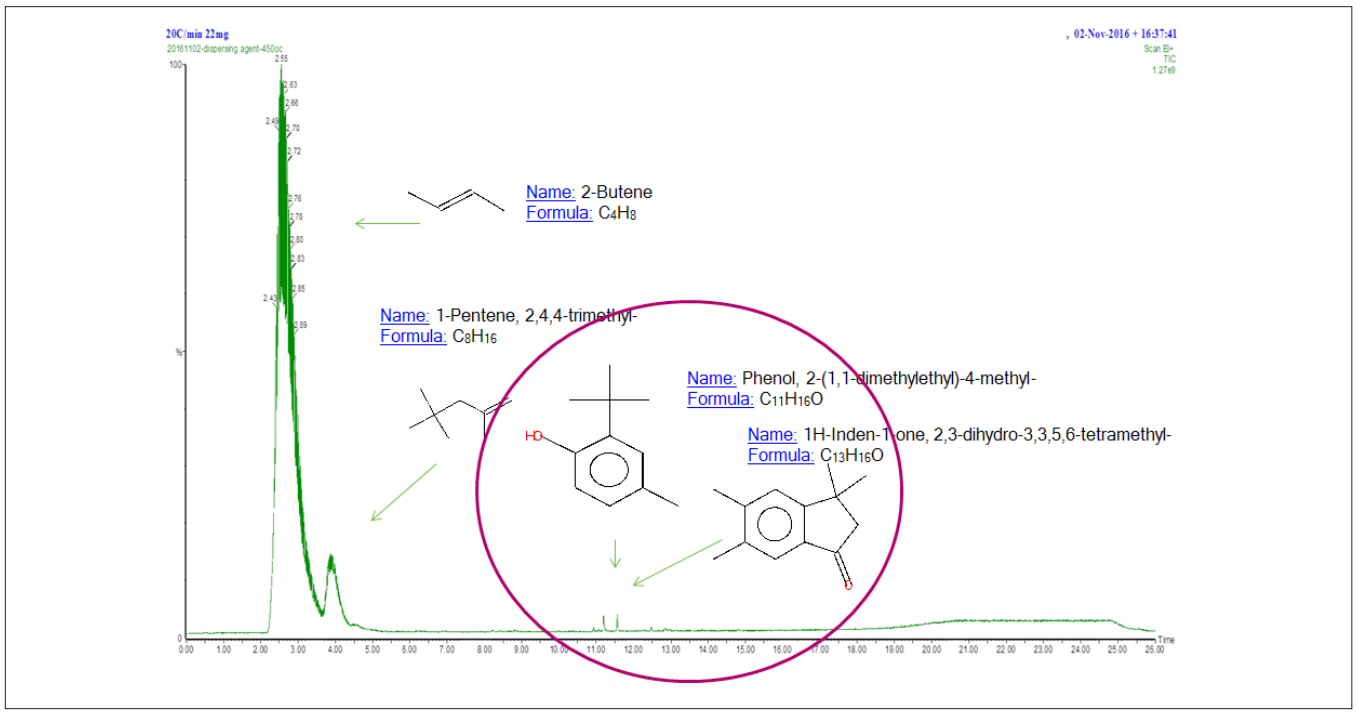

Figure 5. Dispersive Agent – GC/MS TIC Diagram after injection of evolved gas at 450°C.

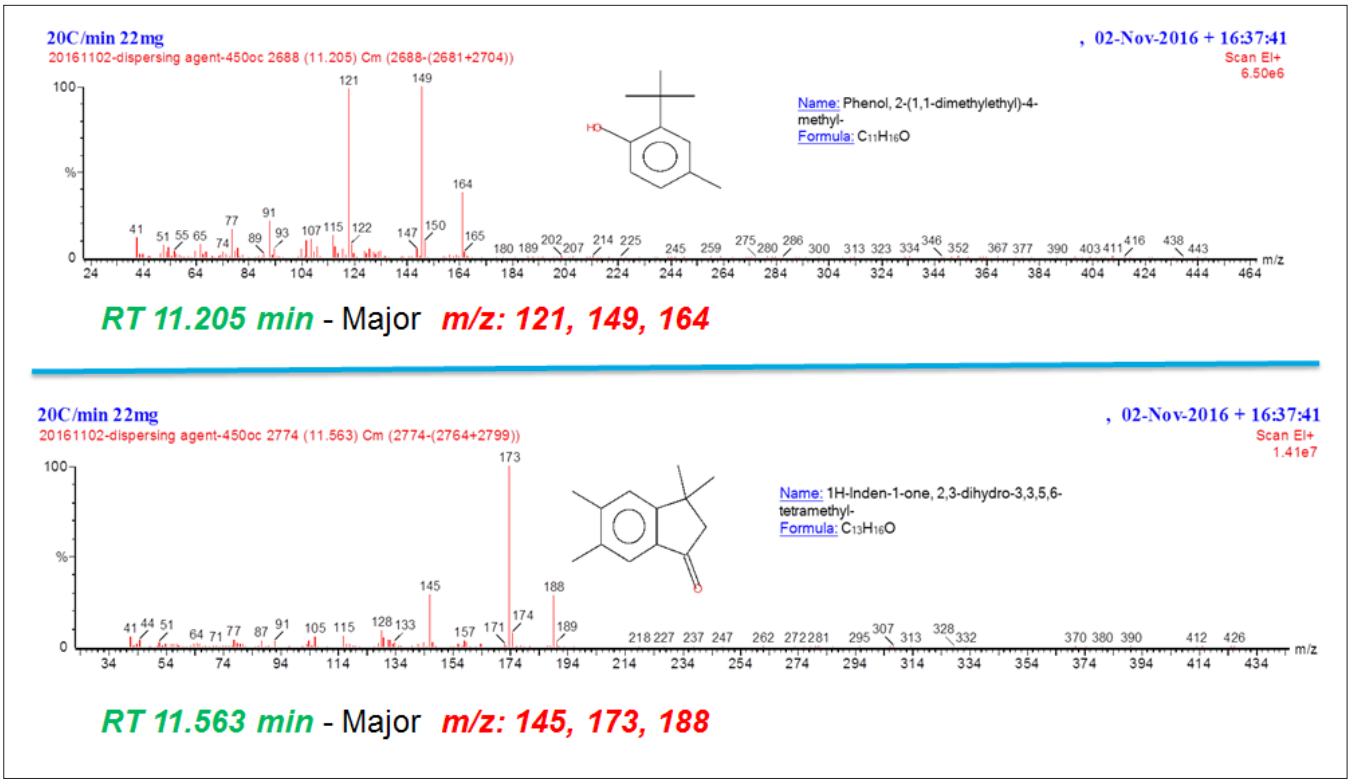

Figure 6. Dispersive Agent – Mass spectrum of selected peaks at RT 11.205 min and 11.563 min.

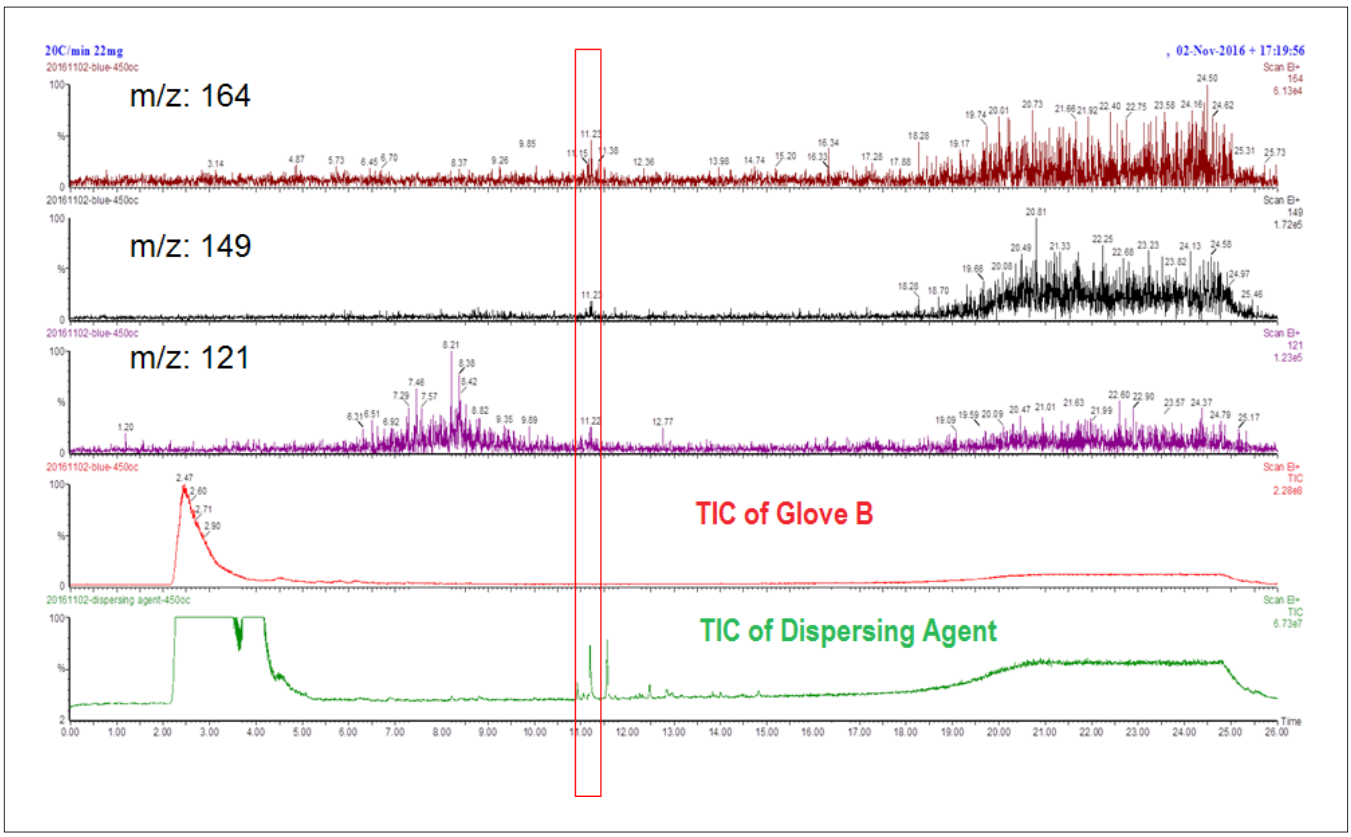

Figure 7. Blue glove – Peak monitoring at RT 11.205 min, m/z 121, 149 & 164, showing trace amount of dispersing agent detected.

Conclusions

TGA is a useful tool when it comes to compositional analysis as it allows quantification of weight loss of a material at targeted temperature region. By hyphenating TGA to a GC/MS system, further identification of evolved gases can be carried out.

In this case, TG-GC/MS hyphenation system successfully detect trace amount of additive used in glove manufacturing process. With minimum sample preparation, the TG-GC/MS serves as a much faster approach when it comes to sample differentiation, at the same time allowing a more complete thermal characterization of material.

References

1. Vanderbilt Chemicals Product Information, Latex Glossary.

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