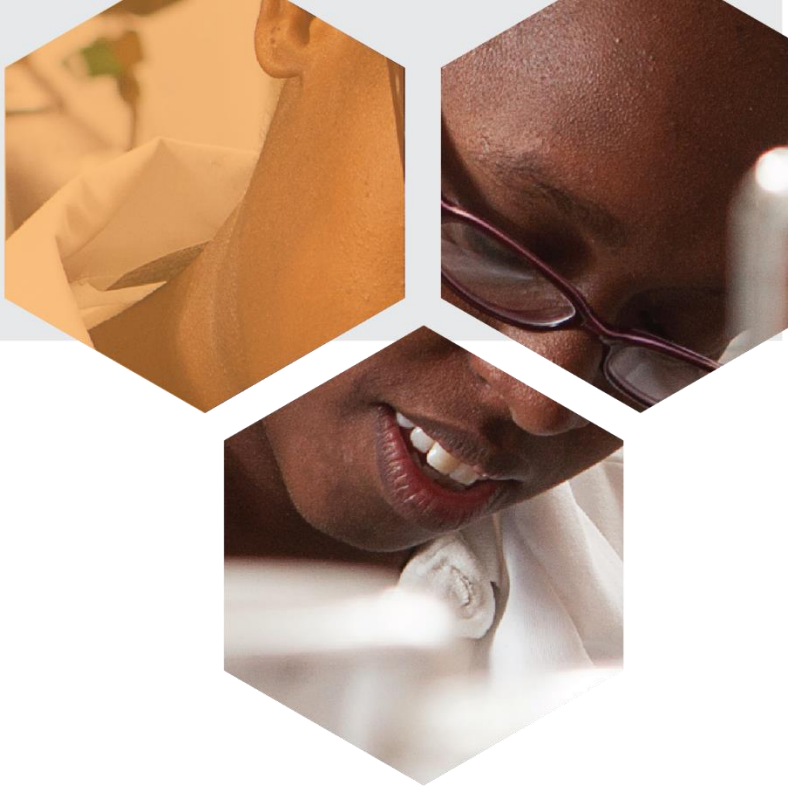
Cambridge Polymer Group



Pyrolysis GCMS of Adhesive

By Dr. Rebecca Bader

Summary

Deformulating low molecular weight and inorganic additives and fillers is relatively routine, but understanding the polymeric composition of a formulation is substantially harder. For composition analysis, more specialized techniques are required, such as Pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS). In this case study, Py-GC-MS was used to examine two silicone adhesives. One adhesive reportedly had good adhesion quality while the second adhesive had poor adhesion quality. Testing revealed that the adhesives had two different siloxane structure types: linear and cyclic, likely influencing the bond properties.

Introduction

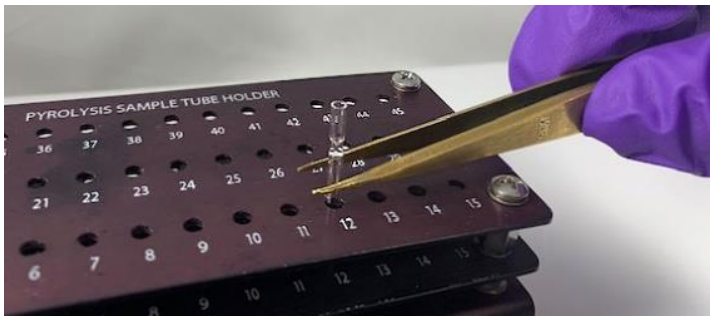
Pyrolysis-gas chromatography-mass spectrometry (Py-GC-MS) uses elevated temperatures to initiate thermal decomposition of a material. This process allows large, complex molecules to be fragmented by rapid heat into characteristic small fragments that can be run on a conventional GC-MS for analysis. These "building blocks" of the polymer can be complex. However, with experience one can "rebuild" the molecule from these oligomers and reconstruct the starting polymer(s).

Advantages of Py-GC-MS include the ability to bypass complex, time-consuming sample preparation procedures, minimal sample size requirements, and multi-step analysis on single samples. Multi-step analysis allows sample separation into various classes of compounds such as; volatiles, semi-volatiles, and non-volatiles. Sample sizes between 5-50 micrograms are placed in quartz analysis tubes, pyrolyzed, and compounds of interest are directly transferred to the GC for analysis.

Polymeric materials often present challenges during analysis, including particle dispersion and poor solubility due to crosslinking. Pyrolysis-gas chromatography-mass spectrometry is an especially useful technique for overcoming these challenges. Py-GC-MS analysis serves polymer research needs in many industries including pharmaceuticals, aerospace, environmental, textile, and consumer products. This study describes a single-step analysis procedure that resulted in thermal degradation compounds of two silicone adhesive materials. Analysis into these compounds offered insight about the performance differences between two adhesives.

Experimental

Two client adhesive samples (good adhesion quality and poor adhesion quality) were prepared by adding representative samples of approximately 5-10 micrograms to a quartz tube. Each tube was pyrolyzed at 800°C for 20 seconds and the evolved gas transferred to the GC-MS for analysis. The GC was equipped with a non-polar capillary column for retention and separation of typical polymeric pyrolysis products.



Results & Conclusions

Peaks from the total ion chromatogram of the samples were screened against several mass spectral libraries. Analysis of the samples revealed differences in the structure of the thermal degradation compounds between the samples. One adhesive sample revealed cyclic structure while the other adhesive revealed linear structure (Figure 1). The details of this structural difference would have been very difficult, if not impossible, to obtain using conventional chromatographic techniques, such as Gel Permeation Chromatography or Liquid Chromatography.

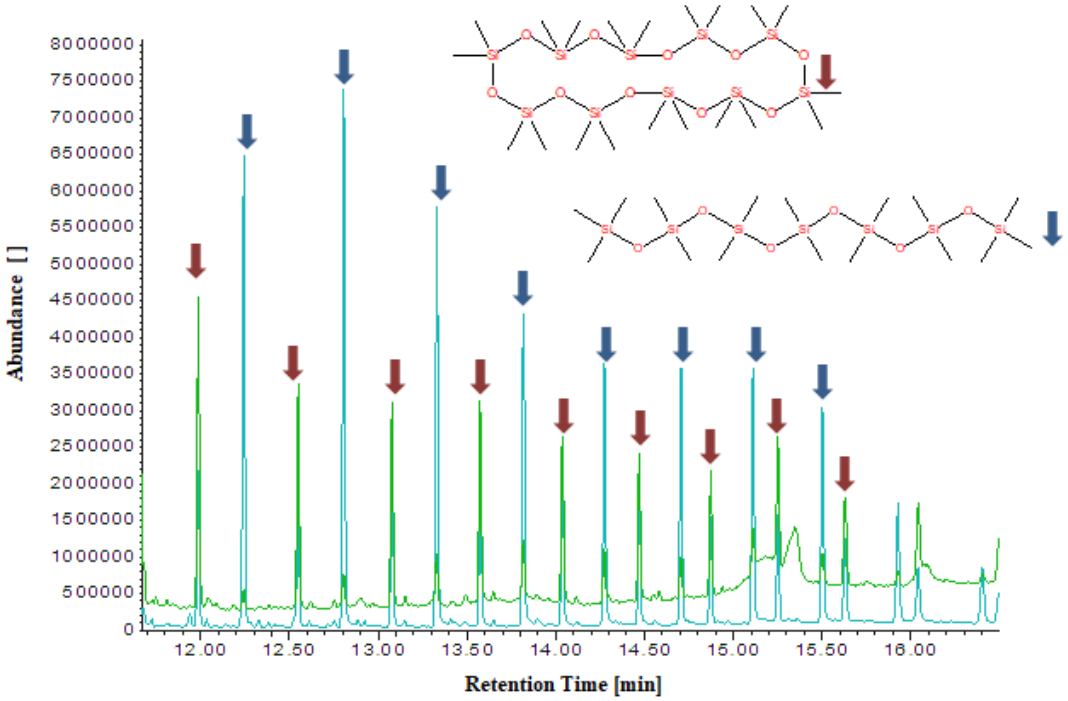


Figure 1: Zoomed total ion chromatogram showing structural differences between two adhesives. Blue arrows indicate linear siloxanes and red arrows indicate cyclic siloxanes.

About Dr. Rebecca Bader

Dr. Rebecca (Becky) Bader is the Associate Director of Chromatography and biocompatibility specialist at Cambridge Polymer Group. Prior to returning in 2023 after initially working for Cambridge Polymer Group from 2017-2020, Becky was Biocompatibility Engineering Manager at ZOLL Medical Corporation where she ensured biocompatibility and material compliance with harmonized standards and global regulatory requirements. Becky has over 20 years of experience in polymeric materials, drug delivery, medical device, and analytical chemistry. She is currently advancing analytical techniques for materials characterization at Cambridge Polymer Group.

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