National Textile Center

FY 2003 (Year 12) Continuing Project Proposal

Project No.

C01-NS08

Competency: Chemistry

Improving the Thermal Stability of Textile Processing Aids

Project Team:

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Objective:

The overall objective of this project is to investigate the thermal behavior of textile finishes and lubricating agents in order to improve their thermal stability at high temperature processes.

Progress Statement:

A schematic diagram of the total experimental system is shown in Figure 1. It includes a stainless steel heating chamber, a high temperature QCM system, an on-line GC system and an off-line gel permeation chromatograph (GPC) system. The QCM is used to measure in situ, real-time lubricant buildup on the metal surfaces. The GC on-line analytical system, equipped with both thermal conductivity (TCD) and flame ionization (FID) detectors, is designed to monitor the gas phase components produced by thermally stressing of the lubricants at elevated temperatures. The molecular weights and molecular weight distributions of the liquid and solid phase products are determined using GPC. Their composition is determined by a gas chromatograph-mass spectrometry (GC/MS). Fourier transform infrared spectroscopy (FTIR) is used to identify the new chemical bonds formed in the degraded lubricants. Also thermogravimetric analysis (TGA) is employed to determine the amount and the rate of weightloss during the lubricants heating.

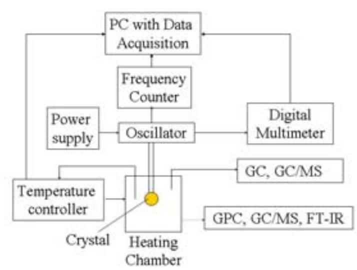
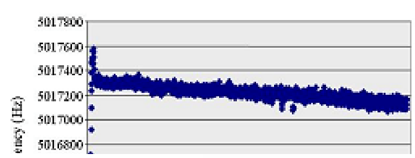
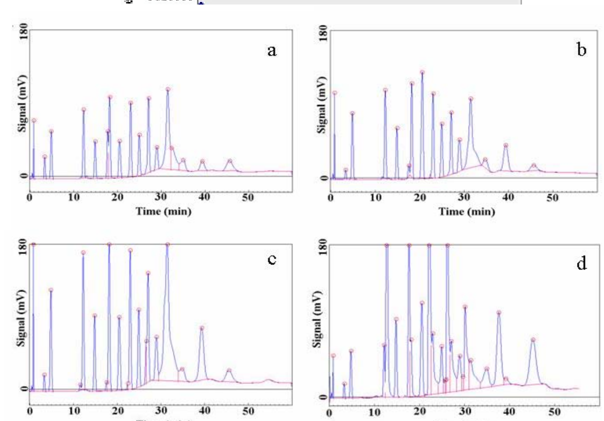


Figure 1. Schematic diagram of the experimental

. Figure 2 shows a typical QCM result for the polyol ester lubricant degradation at constant temperature period. The decrease of frequency with heating time is caused by both the mass loading on the electrode surfaces and the increase in the lubricant viscosity with increasing the heating time.



. Figure 3 shows the representative gas chromatograms of Liquid 322. a, b and c represent the volatilized phase compositions after four hours of heating the lubricant at 180, 200 and 220°C, respectively. The composition of initially volatilized phase when the temperature reached 200 °C is shown in figure 3-d. Analyzing the results indicates that the components with lower boiling point vaporized as soon as the liquid reached the higher temperature. When comparing the chromatograms of 180 (a), 200(b) and 220 °C (c), we find that higher temperatures increase the rate of lubricant



breakdown, as seen by the increase in peak height, while the degraded products, as shown by the retention times, changed little.

The GC/MS results indicate that more than sixty organic compounds were included in the cooling condensed samples of volatile gas phase generated by heating the lubricants at 200 °C for seven hours. The major products were shown to be paraffin C1-C9, aldehydes C1-C9, methylketones C3-C9, carboxylic acid C1-C9 and carboxylic acid, methyl esters C4-C11. Some small amounts of alcohol and olefin were also detected.

…The TGA tests were conducted both in air and in nitrogen atmospheres to investigate the decomposition and evaporation behaviors of lubricants, respectively. Although the lubricants were of the same base component, the differences in the rates of evaporation (in nitrogen) and decomposition (in air) were obvious. This is in a good agreement with the results obtained from the weight loss tests in a conventional oven (Figure 4).

Figure 3. Chromatograms of Liquid 332 from GC

. As the lubricants are processed at high temperature, an increase in viscosity has been observed. Viscosity increases with heating time; this increase gets smaller when the viscosity is measured at higher temperatures. The trend of change in the viscosity indicates a very good correlation with a exponential curve. Extrapolation of the trend line to 180ºC gives an estimation of the viscosity at this temperature.

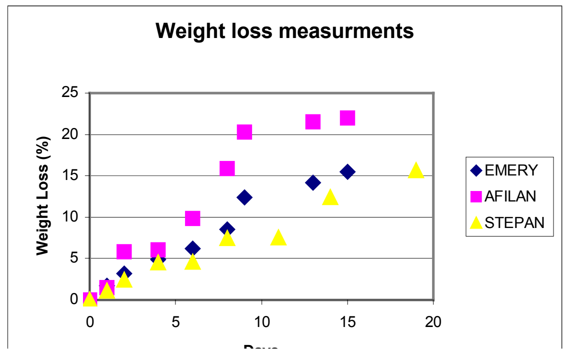


Figure 4. Weight loss of the lubricants heated in a conventional oven at 200°C for different exposure times.

Samples of the three above-mentioned lubricants were heated at 200° in a conventional oven for different exposure times; the IR spectra of the cooled samples were collected. Comparing the spectra of the heated samples with the original ones shows the growth of a broad peak at 3528 cm^{-1} and a slight growth of the peak around 1050 cm^{-1}. The peak at 3528 cm^{-1}corresponds to the presence of hydroxyl group in the compound and the peak at 1050 cm^{-1}relates to the C-O stretch of primary alcohols. The area under the peak at 3528 cm^{-1} was measured and an increase in the area under the peak with increasing the heating time was observed. The increase in the hydroxyl content of the lubricant apparently has a direct relationship with the exposure time and can be used as an indicator for the degree of degradation.

Next Year's Goals:

Our research goals for the upcoming year have will be to (1) utilize the newly installed analytical equipment to continue the development of the kinetic model of thermal degradation; (2) collect real time QCM and GC data from the degradation products (solid, liquid, and vapor) of commercial samples; and (3) determine any effects from incorporating common lubricant additives with the base fluids in the degradation experiments.

Approach:

The proposed research is an interdisciplinary effort between a textile chemist, a chemical engineer and an expert in textile processing. Our combined expertise in surface/interfacial science, transport phenomena, textile processing and an in depth understanding of the fundamental chemistries of both fibers and processing aids will enable us to conduct a comprehensive investigation of this problem.

While there are several different classes of textile processing aids; the first step in our research is to identify a class of compounds to work with, and to classify the critical features of the degradation materials and their associated impact on the fiber and the equipment. This is underway through our interactions with textile manufacturers focused on various stages of fiber and fabric production.

Our research will be a systematic study to identify the driving force for the thermal breakdown of processing aids. We will provide a real time physical and chemical analysis of the degradation using a unique combination of analytical tools. Initial degradation studies are performed on a series of polyol esters based synthetic finishes that have a high thermal and thermo-oxidative stability. A detailed study of role of inclusion of various additives on thermal degradations will enable us to establish a protocol for subsequent assessment studies. We will test the role of various additives and controlling parameters such as concentration, type and temperature in modifying/improving finish behaviors.

High temperature, static and dynamic experiments will be conducted using a specially designed quartz crystal microbalance (QCM) apparatus

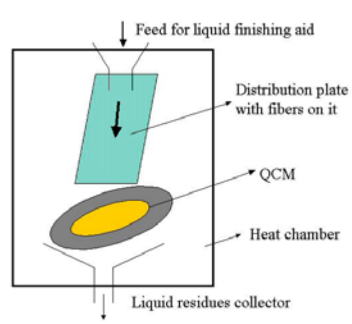


Fig. 5 Liquid finishing aids flow system

equipped with an on-line GC analytical system. This state of the art approach will enable a continuous in-situ evaluation of the rates of solid deposition and gas volatilization due to thermal breakdown. In particularly, to mimic the actual industrial processes, we will incorporate textile fibers into the experimental system as shown in Fig. 5. Our approach is novel because it will be the first comprehensive in-situ evaluation of the dynamic thermal degradation of textile chemicals and fibers. Determining the underlying interfacial, chemical, and transport mechanisms will provide detailed information about the interactions of the additives, base chemical and fiber at elevated temperatures.

The ultimate goal is to be able to predict thermal degradation behavior in processing aids based on a set of fundamental chemical and physical property measurements. These include: viscosity changes, rate of solid deposition, chemical reaction rates, chemical byproduct generation, and particle size analysis. This insight will improve the selection and use of new classes of processing aids and associated formulations.

Outreach to Industry:

Our research group has already had interactions with researchers in the Finishing Area within Dupont Textiles in Chattanooga, Tennessee. They have been supportive and encouraging of this approach to address thermal degradation in textile processing. We have interacted also with our industrial partners including Boehme Filatex and Goulston Technologies, the two leader companies in processing aids industry. Especially, we visited Boehme Filatex in Watlington. The technical discusses with them have provided a critical starting point in our assessment of textile processing aids. Regarding the actual textile system, it is suggested that the volatilization, decomposition, and deposition of aids at high temperature should be focused on. Future interactions with other textile and fabric manufacturers will enable us to expand the application of the technology from fiber finishes to aids used in weaving, knitting and other areas where high speed, thermal breakdown can inhibit economic production of textile products.

The proprietary nature of the additive systems further emphasizes the need to conduct this research in the context of the NTC where the partner companies can provide an important perspective on the end use of the finishing aids. A first step in this research is a survey of the thermal decomposition issues in the industries represented in the consortium. This would provide important information in the selection of a class of aids to focus on in our research. It would also insure the broadest impact of the work on the U.S. industry. The ability to know a priori the thermal degradation profile of processing aids will give the current U.S. textile industry a competitive edge in terms of worker safety, environmental issues (e.g., reduced smoke generation). Furthermore, the investment in a detailed study to assess a variety of processing aids will support fiber producers, textile mills, and garment manufacturers.

New Resources Required:

This project will require support for the graduate and undergraduate student researchers, faculty time, travel to facilitate interactions with industry and some funding for experimental equipment.