

## 5.0 Recommendations

- Determine whether this reclamation process can be used on other thermotropic liquid crystalline polymer / thermoplastic composite systems. The use of organic peroxides to breakdown aliphatic polymers can be explored on such thermoplastics as low density polyethylene (LDPE) or on different molecular weight polypropylenes. However, the reactive extrusion of linear low density and high density polyethylene with organic peroxides preferentially leads to cross-linking and not to degradation. Therefore, the degradation of any PE matrix would be best carried out using thermal degradation. In the case of other polyolefins, as long as the polymeric backbone contains aliphatic linkages with tertiary carbons, this type of reactive extrusion should result in the breakdown of the backbone. Instead of the recombination with other carbon radicals (cross-linking), the tertiary carbon radical prefers to undergo chain scission. Concurrent with this degradation, the matrix material must be soluble in the mineral oil, or at least partially soluble.

Furthermore, it would be important to determine if this reclamation process could be used to reclaim other thermotropic liquid crystalline polymers from different TLCP/TP composite systems. The use of this reclamation system can be tried with such TLCPs as HX1000, HX6000, Vectran A950, or even Vectran B950. As long as these thermotropic liquid crystalline polymers do not contain any aliphatic linkages, are not susceptible to peroxide attack, and are not soluble in the mineral oil, this

reclamation process should result in the collection of reclaimed TLCP. Furthermore, it would also require that the matrix of these composite materials fit with the above criteria for degradation and dissolution.

- Determine the optimum processing conditions such as extrusion temperatures, screw speed, peroxide concentration, bath temperature, stirring speed, and length of dissolution for the reclamation process. Examine what the effects of changing the heating zone temperatures of the extruder have on the amount of degradation and phase separation of the product, which could be monitored by viscosity changes. Determine how fast the reactive extrusion could be run by examining the level of degradation at multiple speeds. Examine the minimum level of degradation of polypropylene needed in order to completely dissolve the PP into the mineral oil. Determine what the maximum solubility of the light mineral oil is and how many times the mineral oil can be reused to dissolve the degraded polypropylene. Examine the effect of varying the bath temperature and the speed of mixing on the solubility of the degraded polypropylene. Also, it would be important to determine the minimum amount of time needed to effectively dissolve the degraded polypropylene in order to maximize efficiency. By optimizing these variables, the overall production cost could be reduced and the effect of the thermal and chemical histories on the materials could be minimized. Furthermore, if the degradation of the PP could be minimized, recovery of the degraded PP could lead to further uses for this resin in such applications as non-wovens.

- Determine the maximum loading of the reclaimed HX8000 that can be used to make more in situ composites of polypropylene and HX8000. By changing the injection molding conditions (such as nozzle and mold temperatures), higher loadings of the reclaimed HX8000 could be processed into plaques. Furthermore, by utilizing the super-cooling behavior of TLCPs a pure plaque of the reclaimed HX8000 could be generated. Once this has been accomplished, then the next step would be to determine if the reclaimed plaque demonstrated the same mechanical properties of the pure HX8000 at the same processing conditions.
- Determine if the reclaimed HX8000 can be blended with pure HX8000 and then be used in the dual extrusion process to generate fiber composites. The mechanical properties could be tested in order to determine if any losses in properties would be realized. Furthermore, it would also be important to investigate whether or not the reclaimed HX8000 could be used by itself to generate fiber composites in the dual extrusion process.
- Finally, determine how many times the HX8000 could be reclaimed before any substantial losses in mechanical properties were realized. This could be accomplished by reclaiming the HX8000 from in situ composites, reprocessing the reclaimed resin into second generation composites, and then determining the mechanical properties of the recycled composites. After the mechanical properties were determined, the same

composites could be passed through the reclamation process, and the recycled HX8000 could be reclaimed again for third generation composites. This process could be repeated over and over again in order to determine when a loss in mechanical properties would be realized. This determination could lead to a further reduction in the cost of the reclaimed material.

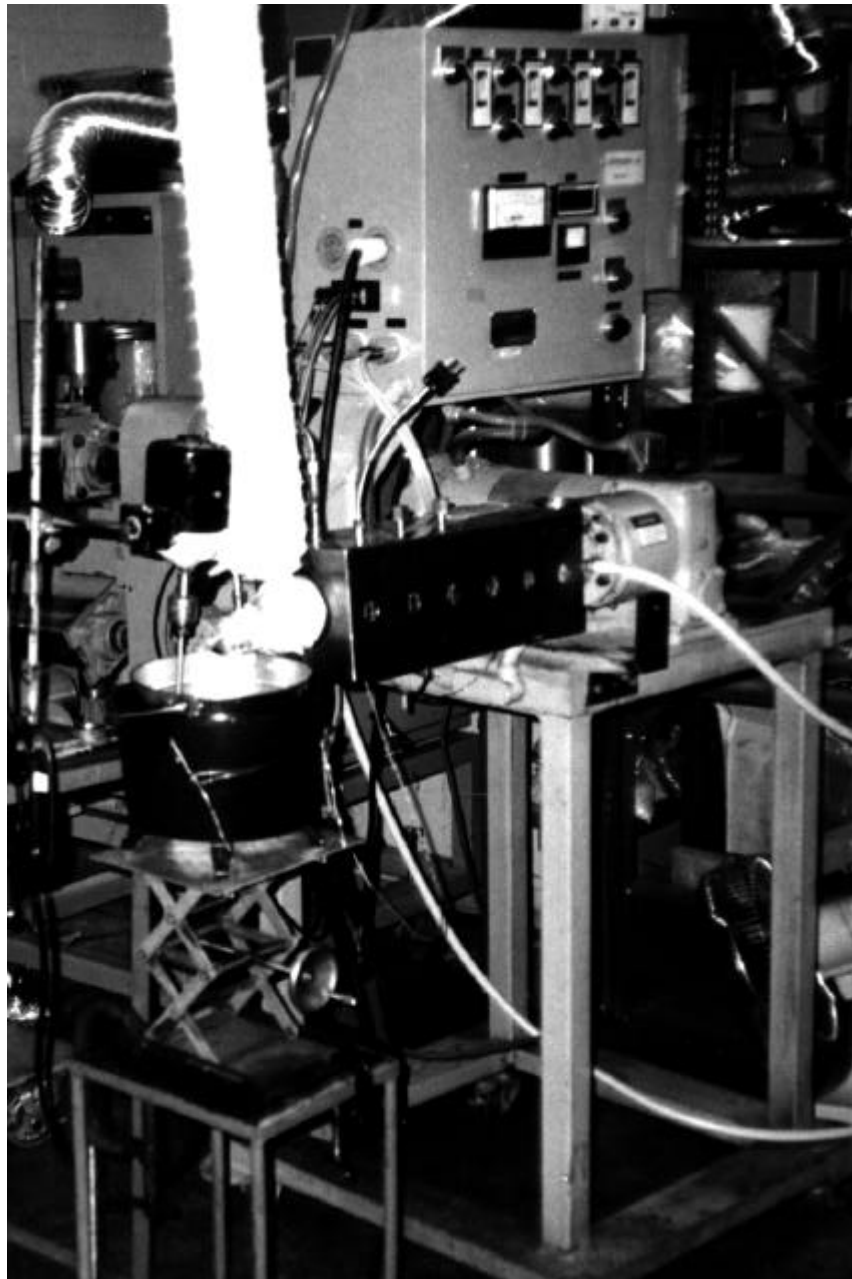
**Appendix A:**  
**Reclamation Process Pictures**



**Figure A.1** Cumberland Polymeric Material Granulator used to render the In situ Composites (40 wt% HX8000 / 60 wt% PP 6523) into Pieces.



**Figure A.2** Spool of Composite Fiber (40 wt% HX8000 / 60 wt% PP 6523) that was generated in the Dual Extrusion Process and the Granulated Pieces for Reactive Extrusion.



**Figure A.3** Reclamation Process which consists of the Single Screw Extruder for Reactive Extrusion and the Light Mineral Oil Bath for Polypropylene Dissolution.

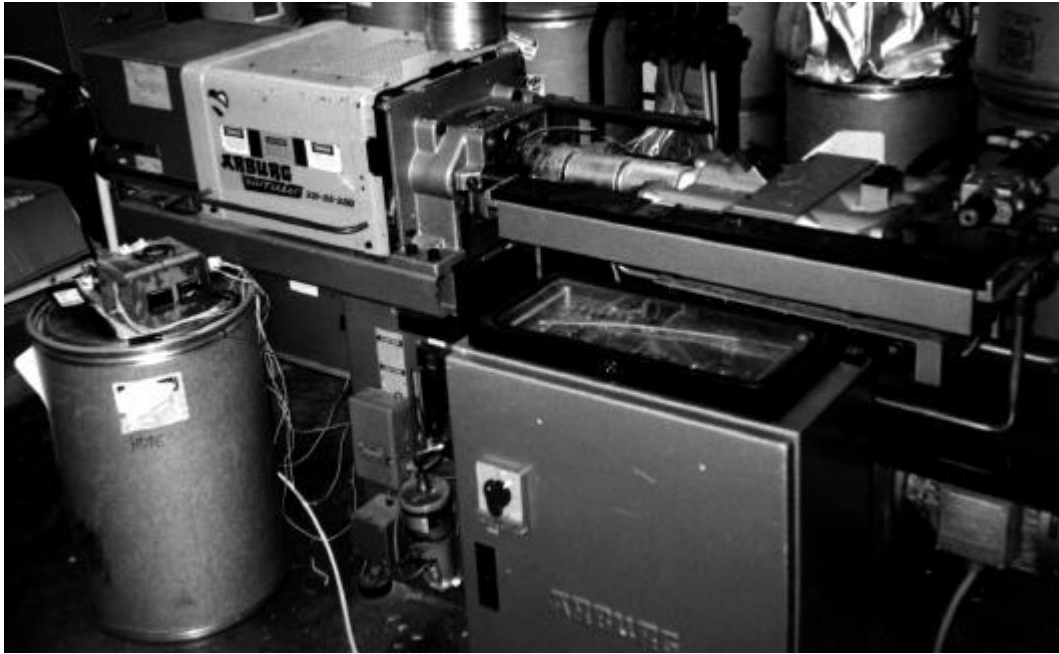




**Figure A.4** Close-up of the Mineral Oil Dissolution Process that shows the Impeller and the Screening System with leftover Reclaimed HX8000 Pieces.

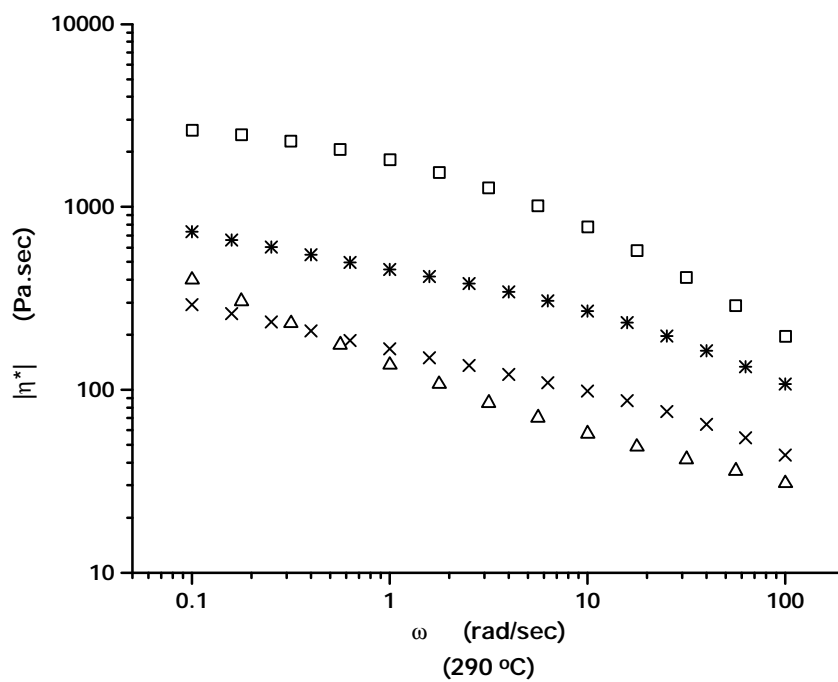


**Figure A.5** Fisher Scientific Marathon 21K/R Centrifuge used to separate the pieces of Reclaimed HX8000 from the Mineral Oil, the Kerosene, and the Hexanes.

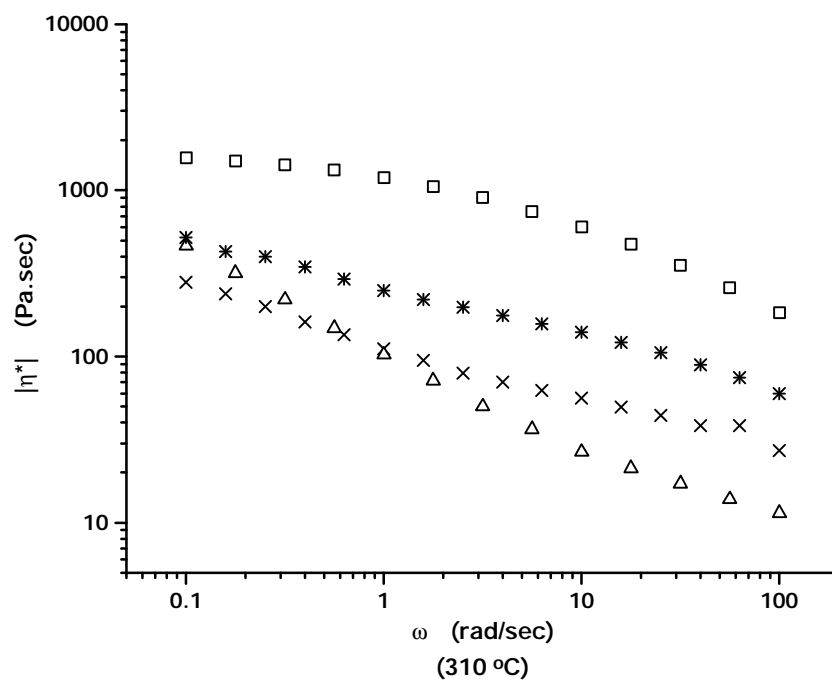


**Figure A.6** Arburg Allrounder Injection Molder (Model 221-55-250) used to generate the Injection Molded In situ Composites and Plaques.

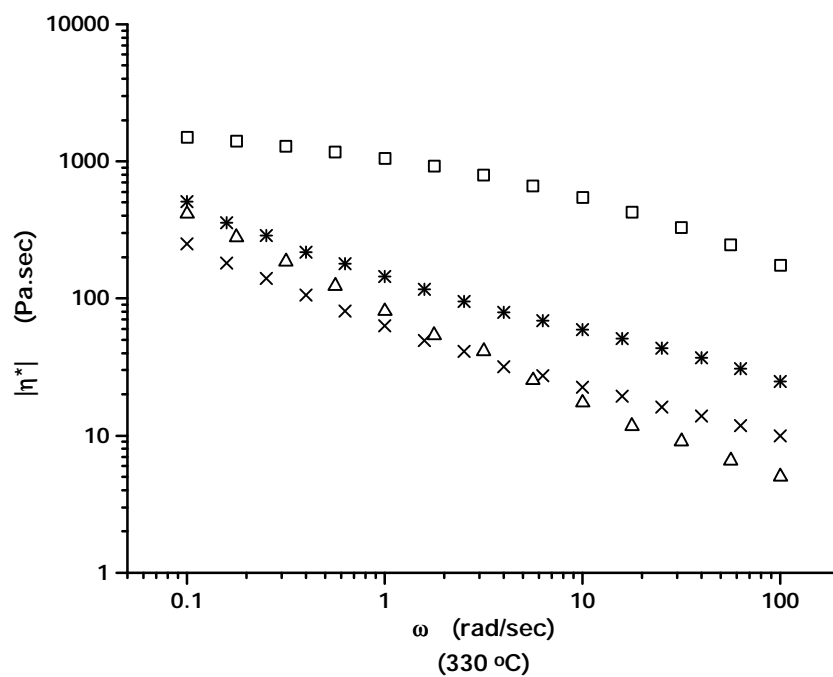
**Appendix B:**  
**Rheological Data**



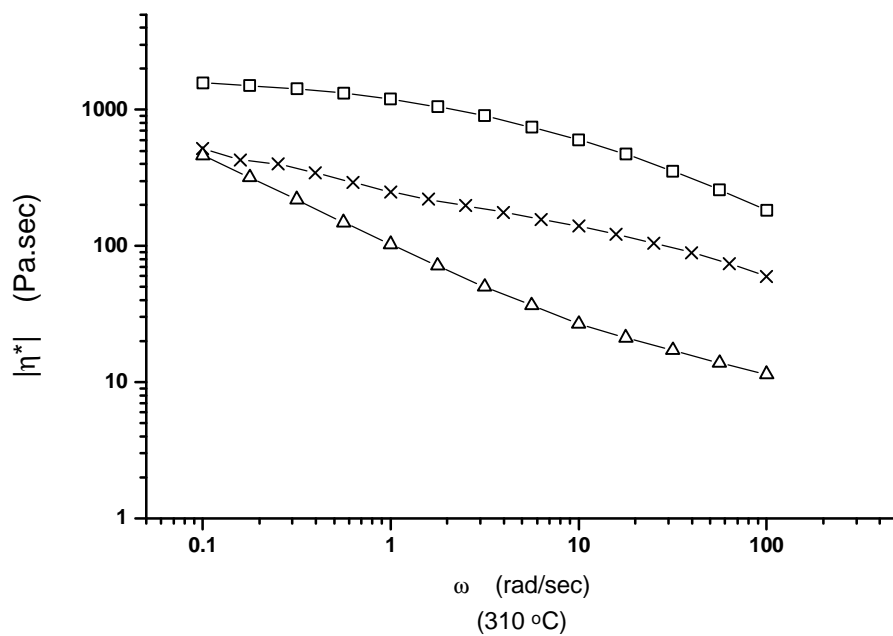
**Figure B.1** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) Comparison among Pure HX8000 = ( $\square$ ), Pure HX8000 run through the Reactive Extrusion Process (x), 40 wt% Reclaimed HX8000 / 60 wt% Pure HX8000 = (\*), and 97 wt% Pure Reclaimed HX8000 = ( $\Delta$ ). Run at 290°C, 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.



**Figure B.2** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) Comparison among Pure HX8000 = (□), Pure HX8000 run through the Reactive Extrusion Process (x), 40 wt% Reclaimed HX8000 / 60 wt% Pure HX8000 = (\*), and 97 wt% Pure Reclaimed HX8000 = (Δ). Run at 310°C, 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.

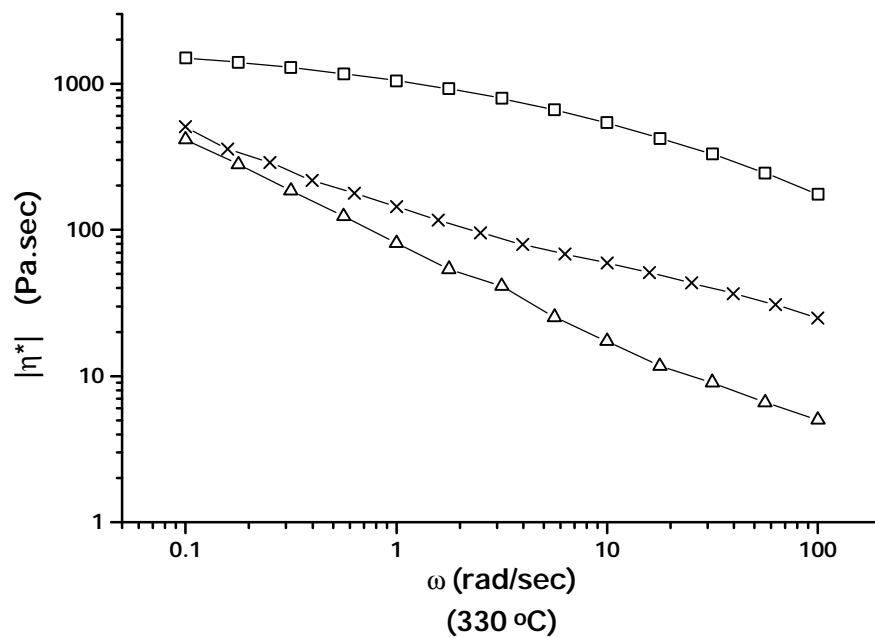


**Figure B.3** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) Comparison among Pure HX8000 = ( $\square$ ), Pure HX8000 run through the Reactive Extrusion Process (x), 40 wt% Reclaimed HX8000 / 60 wt% Pure HX8000 = (\*), and 97 wt% Pure Reclaimed HX8000 = ( $\Delta$ ). Run at 330°C, 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.

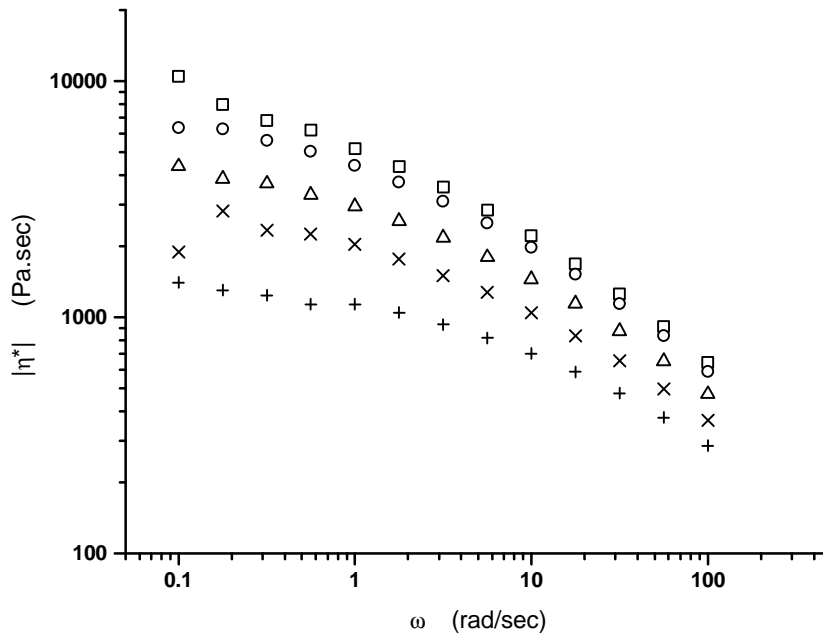


**Figure B.4** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) Comparison among Pure HX8000 = (—□—), 40 wt% Reclaimed HX8000 / 60 wt% Pure HX8000 = (—x—), and 97 wt% Pure Reclaimed HX8000 = (—△—). Run at 310°C, 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.

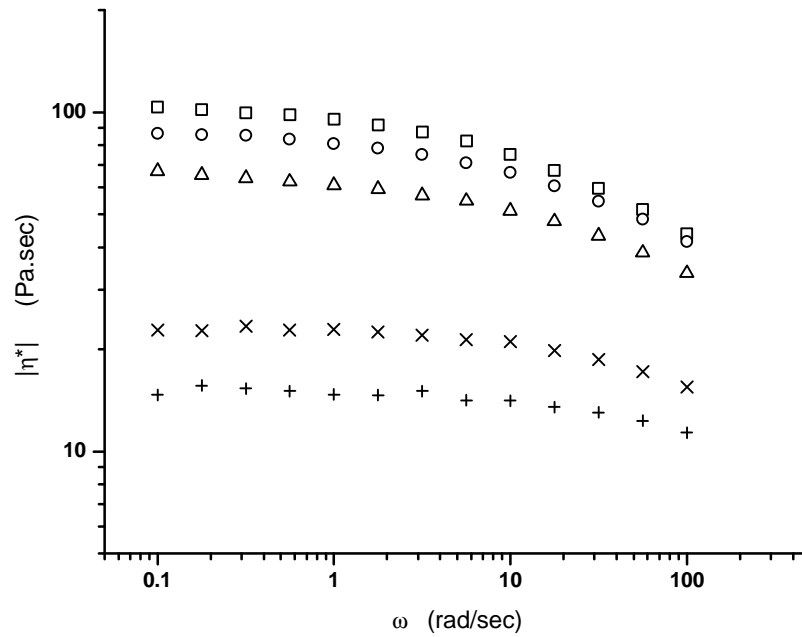




**Figure B.5** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) Comparison among Pure HX8000 = (—□—), 40 wt% Reclaimed HX8000 / 60 wt% Pure HX8000 = (—x—), and 97 wt% Pure Reclaimed HX8000 = (—△—). Run at 330°C, 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.

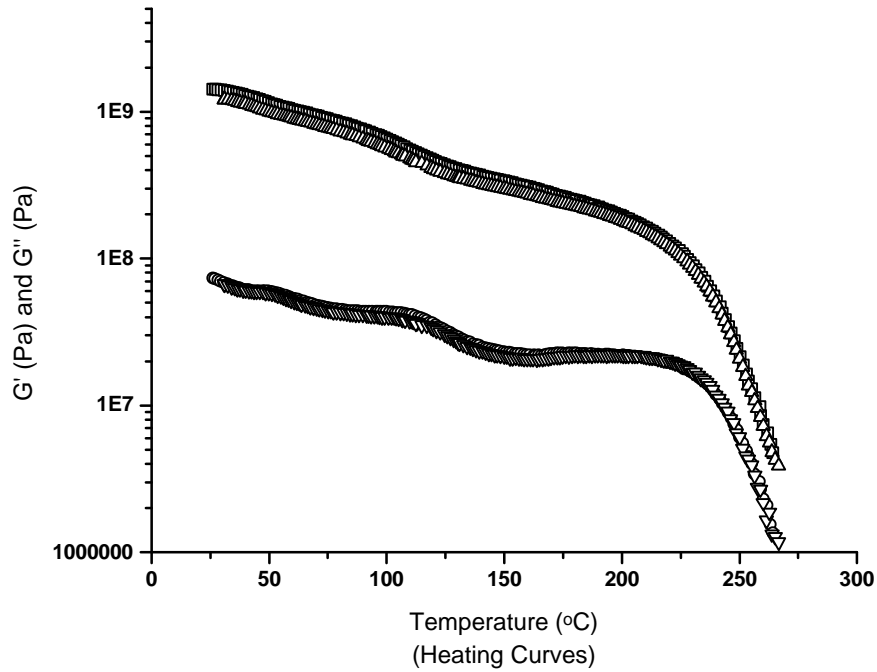


**Figure B.6** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) for Pure Polypropylene, Montell PP6523 at 5 Different Temperatures. 190°C = (□), 210°C = (○), 230°C = (Δ), 250°C = (x), and 270°C = (+). Run at 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.

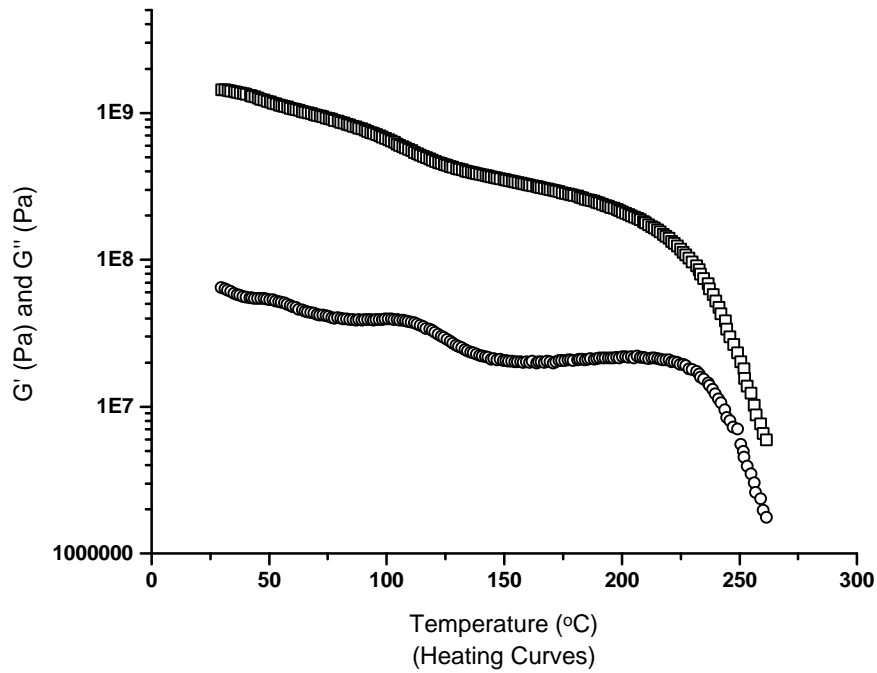


**Figure B.7** Complex Viscosity ( $|\eta^*|$ ) vs. Angular Frequency ( $\omega$ ) for 1.0 wt% Dicumyl Peroxide Reacted, Polypropylene (Montell PP6523) at 5 Different Temperatures. 190°C = ( $\square$ ), 210°C = ( $\circ$ ), 230°C = ( $\Delta$ ), 250°C = ( $\times$ ), and 270°C = ( $+$ ). Run at 5.0% strain, 25 mm parallel plates, and a 1.0 mm gap.

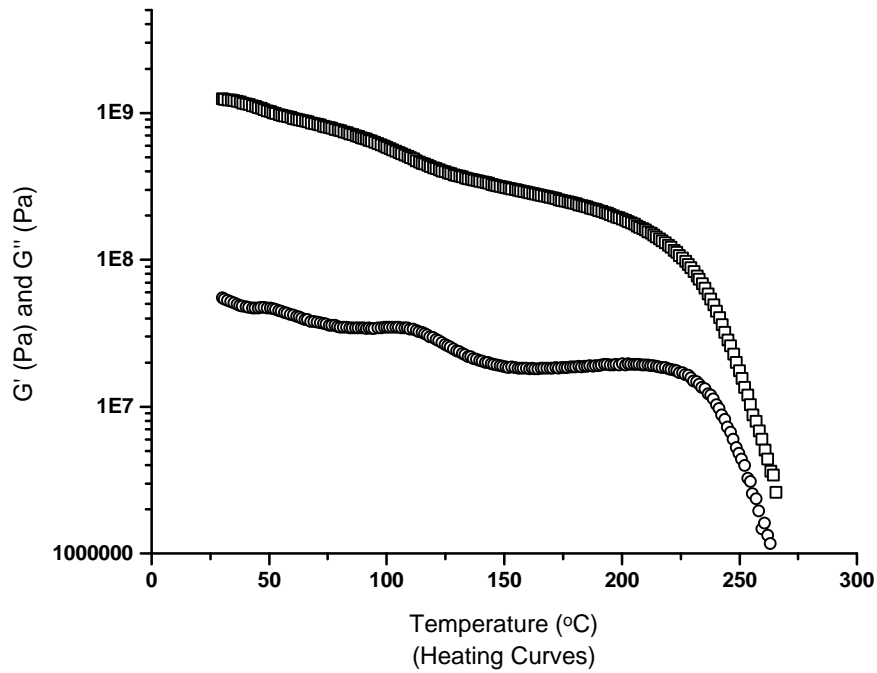
**APPENDIX C:**  
**DMTA DATA**



**Figure C.1** DMTA Data of Storage Modulus ( $G'$ ) and Loss Modulus ( $G''$ ) for Two Separate Runs of Pure HX8000 Injection Molded Plaques. Run from 30°C to 300°C at a Rate of 2.5°C/min, at a Angular Frequency ( $\omega$ ) of 10 rad/sec, and at a Strain of 0.1%. Run #1 Storage Modulus = ( $\square$ ), Run #1 Loss Modulus = ( $\circ$ ), Run #2 Storage Modulus = ( $\Delta$ ), and Run #2 Loss Modulus = ( $\nabla$ ).



**Figure C.2** DMTA Data of Storage Modulus ( $G'$ ) and Loss Modulus ( $G''$ ) for 20 wt% Reclaimed HX8000 / 80 wt% Pure HX8000 Injection Molded Plaque. Run from 30°C to 300°C at a Rate of 2.5°C/min, at a Angular Frequency ( $\omega$ ) of 10 rad/sec, and at a Strain of 0.1%. Storage Modulus = ( $\square$ ) and Loss Modulus = ( $\circ$ ).



**Figure C.3** DMTA Data of Storage Modulus ( $G'$ ) and Loss Modulus ( $G''$ ) for the 40 wt% Reclaimed HX8000 / 60 wt% Pure HX8000 Injection Molded Plaque. Run from 30°C to 300°C at a Rate of 2.5°C/min, at a Angular Frequency ( $\omega$ ) of 10 rad/sec, and at a Strain of 0.1%. Storage Modulus = ( $\square$ ) and Loss Modulus = ( $\circ$ ).

# Vita

## Montgomery C. Collier

The author was raised in the suburbs of Chesapeake, Virginia. Upon the completion of his secondary education, the author attended Virginia Polytechnic Institute and State University, where he was awarded a Bachelor of Science in Chemistry in 1993. The author went on to work for two years as a chemist with three different companies: 1) Environmental Testing Services, Inc. in Norfolk, VA, 2) Gage Specialty Products, Co. at the Ford Motor Company Assembly Plant in Norfolk, VA, and 3) Procter & Gamble Co. in Cincinnati, OH. In August of 1995, the author enrolled in Graduate School at Virginia Tech in the Chemical Engineering Department. He then received a Master's of Science under the advisement of Dr. Donald G. Baird at Virginia Tech in 1998. He was offered and accepted a position at National Starch and Chemical Company.