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DEPARTMENT OF CHEMICAL ENGINEERING
THE UNIVERSITY OF TEXAS AT AUSTIN
CHE 395E

Could we use the Micro 5 Gn Phil study?

Phenoxy/Polyester Blends

2 rewrds?

I. OBJECT

Phenoxy resins are thermoplastic condensation products of bisphenol-A and epichlorohydrin which contain secondary hydroxyl functionality. This functionality is probably responsible for their reported miscibility with a variety of aliphatic polyesters, including poly(caprolactone), PCL, and their reported compatibility with aromatic polyesters, including poly(butylene terephthalate), PBT, and poly(ethylene terephthalate), PET. The mechanism for miscible blend formation is somewhat unclear. PCL/Phenoxy blends seem to be miscible because of hydrogen bond formation between the secondary hydroxls and the carbonyl ester functionality on PCL. Miscible PET/Phenoxy blends can be formed during melt processing, but these materials are not normally miscible and the appearance of a single T_g seems to be related to cross-linking. The problem is complicated by the catalysts used to form the polyesters. PCL is formed with a tin catalyst, while PET is usually formed with a titanium isobutoxide catalyst which is known to promote transesterification. The possibility exists for chemical transesterification to be the driving force for miscibility of Phenoxy and polyesters. The purpose of this experiment is to explore this possibility.

II. REFERENCES

1. L. M. Robeson and A. B. Furtek, J. Appl. Polym. Sci., 23, 645 (1979).
2. F. M. Berardinelli, U. S. Patent No. 3,962,174, assigned to Celanese Corp., Dec. 9, 1975.
3. J. S. Gall, U. S. Patent No. 4,008,199, assigned to Celanese Corp., Feb. 15, 1977.
4. J. E. Harris, S. H. Goh, D. R. Paul, and J. W. Barlow, J. Appl. Polym. Sci., 27, 839 (1982).

III. EQUIPMENT AND SUPPLIES

1. Brabender Plasticorder with 50 cc batch mixing head and motor torque readout.
2. Union Carbide PCL-700 poly(caprolactone)
3. Celanese PETPAK poly(ethylene terephthalate)
4. Eastman KODAR poly(1,4 cyclohexane-dimethanol iso/terephthalate)
5. Titanium isobutoxide
6. Differential Scanning Calorimeter

IV. PROCEDURES

A. All materials should be thoroughly dried befor melt processing to prevent loss of polyester molecular weight by hydrolysis reactions. Your materials will be found in an air oven at 100 °C, where they have been placed to dry at least 4 hours prior to the beginning of class. PCL melts at 60 °C. This material will be found in a vacuum oven where it has been placed to dry. Take only as much material as you need to prepare a blend and return the remainder to the drying oven. Quickly weigh out the required materials and add them to the mixing bowl before they have cooled sufficiently to regain water. The mixing bowl has a capacity of 50 cc. All blends should fill the mixing bowl for consistent results.

B. Thermal Stability Studies. The Brabender Plasticorder is used to affect melt mixing of the Phenoxy and polyester and to monitor the melt viscosity of the blend with time a processing conditions. Your instructor will demonstrate its use. Rotor speed should be set to 60 RPM for all studies. The Bowl temperature should initially be set at 290 °C for all compositions.

1. The following compositions should be initially examined:

   (1) 50PETPAK/50PHENOXY
   (2) 50 KODAR/50PHENOXY
   (3) 50 PCL/50PHENOXY
   (4) PCL
   (5) PHENOXY
   (6) PETPAK

   Measure the torque vs time profiles for each system to about 20 min. Draw a small sample suitable for obtaining T_g information by DSC about every two minutes, or sooner, depending on the rate of change of torque with time.

2. Repeat the above experiments but add 0.5% by weight titanium isobutoxide catalyst to the mixture.

3. Repeat the above experiments at lower temperatures if possible. PCL/PHENOXY blends can probably be processed as low as 140 °C.

4. If available, add a phosphite stabilizer to the mixtures and repeat the experiments in item 1.

C. DSC MEASUREMENTS

1. Determine the T_{gs} of the samples obtained at various times. Use this information to judge miscibility and/or the influence of reaction on apparent miscibility of the blends.

V. ANALYSIS

A. What evidence exists for transesterification being responsible for formation of miscible blends of Phenoxy with Polyesters? What evidence exists to the contrary?

B. Explain the roles played by the additives used in determining blend miscibility and stability.

C. Suggest other ways to promote stability in Phenoxy/ Aromatic Polyester blends, if you can. Is 20 minutes stability sufficient for melt processing?

D. Suggest uses for the present systems.

E. The presence of a single Tg is usually considered to be a reasonable criterion for miscibility and usually also signifies a single phase , optically transparent, melt. Do these generalizations hold for these systems?