

PERFLUOROPOLYETHER PRODUCTION

Lugão, A.B.*; Andrade e Silva, I.G.*; Castro, A.R.M.**; Ghilardi, F.**; Lopezgolo, I.D.**

(*) Instituto de Pesquisas Energéticas e Nucleares (IPEN/CNEN-SP)

(**) Coordenadoria de Projetos Especiais (COPESP)

Caixa Postal, 11.049 - Pinheiros - CEP 05499

SÃO PAULO-SP - BRASIL

Introduction

Perfluoropolyether (PFPE) have exceptional inertness towards a range of chemical and temperature (-100 to 300°C). Due to their high price, they are only used in demanding applications; for instance, compressor and vacuum pump fluids for liquid oxygen, UF_6 and other reactive chemicals.

Adjusting of polymerization parameters in order to attain molecular weight distribution matching the most useful cuts is important for process feasibility.

PFPE process development was studied in a previous paper⁽¹⁾. Sianesi et al^(2,3), studied the polymerization kinetics and hold the initial process patents. This paper shows preliminary results on fluorination effects on molecular weight distribution.

Experimental

PFPE's were obtained by the following processes: hexafluoro propene oxidation and polymerization; polymer fluorination and PFPE destillation in various cuts.

The fluorination apparatus consisted of a fluorine cell, bubbling in a glass reactor connected to a reflux condenser. The glass reactor could be heated at 300°C by means of a common magnet mixer/heater. The destillation apparatus followed ASTM D-1160-87 operating at 0.1 Torr and temperatures up to 400°C. Fluorination was followed by IR spectroscopy and viscosimetry. Des

tilation was followed by viscosimetry.

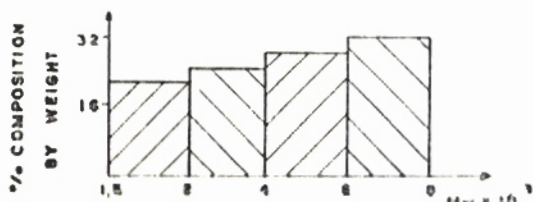
Results and Discussion

The polymer had an average kinematic viscosity from 20 to 4000 cSt. The IR spectra showed absorption due to COOH and COI groups. The COOH should come from COI hydrolysis.

TABLE 1
FLUORINATION OF RAW POLYMER

Run	Sample	Molecular Weight before	Molecular Weight after
1	A	8222	6439
2	B	7142	5358
3	B	5358	5292

FIGURE 1
PFPE DESTILLATION PROFILE



Runs 1 and 2 in table 1 showed large polymer degradation, probably due to very exothermal reactions involved in the process. Runs 2 and 3 were made with the same raw material. The second fluorination showed negligible degradation, showing high stability to reaction conditions. Weight loss was observed in all runs. Surprisingly, in Figure 1, no low molecular weight product (below 1500 u.m.a.) was shown. The absence of very low Mw indicated that the high viscosity drop during fluorination is even greater than showed in table 1. Weight loss must occur mainly in low molecular weight fractions. Comparison with Fomblin cuts showed large production of non useful fractions. Further studies should be performed on milder fluorination conditions and also to fit polymerization conditions in order to offset fluorination degradation.

References

1. Luqão, A.R.; Andrade e Silva, L.G. & Oliveira H. Proceedings of 4th Macromolecular Colloquium, Brasil, Oct. 14-18, 1990, p.91.
2. Patent U.S. 3,442,992. Sianesi, D. et al. May 6, 1969.
3. Sianesi, D. La Chimica e L'Industria, 50 (2): 206-14, 1968.