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SHELL DEVELOPMENT COMPANY

EMERYVILLE, CALIFORNIA

TELEPHONE OLYMPIC 3-2100

June 22, 1956

PLAINTIFF'S EXHIBIT

SH-1030

Shell Chemical Corporation
50 West 50th Street
New York 20, New York

Attention: Manufacturing Department

Gentlemen:

In answer to your letter of April 9 and your wire of May 8, 1956 we are transmitting our most recent information on the manufacture and testing of EPON 562 and EPON Adhesive IX. Included is a brief description of the laboratory test procedures and equipment for evaluating EPON 562 in textile applications. We have also included copies of EMS procedures used in analyzing EPON 562. EMS 5M1/51 "Molecular Weight--Ebullioscopic Method" is currently being revised and we will forward a copy of the revised procedure as soon as it is available. If additional copies of these procedures are needed, they can be obtained from the Analytical Standardization Committee.

Manufacturing and testing of EPON Adhesive IX is quite similar to that of Adhesive VIII, with which you are familiar.

In your wire of May 8 you also requested information on poly(allyl glycidyl ether). Manufacture of this material is in the very early stages of development and we have made only one small pilot batch. A brief description of the procedure used, and the properties of the product, is attached for your information.

We are currently reviewing the potential hazards of this process in view of the possibility of further manufacture of PAGE here. Presumably, we will have more experience with this process before you will be required to start manufacture.

A description of the glycidyl ether manufacture has been delayed to include the latest information on allyl glycidyl ether. We are currently using a different process which avoids the difficult distillation of allylmonochlorohydrin ether. This work should be completed soon and we will send you a description of the process.

LAM 029005

ABS-018211

We trust that the information on EPON 562 and Adhesive IX will be adequate for your needs in manufacturing these materials in your PDU facilities. If we have omitted any pertinent data, please let us know.

Very truly yours,

R M Cole

R. M. Cole

JCR/au
Attachments

cc without EMS attachment:
Shell Chemical, New York: Manufacturing Operations
Shell Chemical, New York: Resins and Plastics
Shell Chemical, Houston
Shell Chemical Union Technical Service Laboratory, New Jersey

bc: Messrs. C. L. Raymond
S. A. Ballard
C. R. Nelson
M. D. Taylor
S. Z. Perry
T. F. Bradley
W. L. Everson
H. W. deJong
S. N. Wik
J. C. Papean

EPON ADHESIVE IX

Semi-Scale Manufacturing Practice

EPON Adhesive IX is essentially the same formulation as Adhesive VIII except that it contains a dispersed solid catalyst, dicyandiamide. It is particularly suited for long, open-assembly time applications, since it will not readily cure at 300°F and temperatures of 345-350°F are normally required.

Formulation of EPON Adhesive IX is certified under MIL-A-8331 (USAF) and no modification of the amounts of ingredients are permitted.

Process Equipment

Blending of the adhesives is carried out in a tilting Read-Standard kneader which is equipped with double sigma blades operating at 21 rpm, external heating coils, and a 7.5 hp motor. The unit has a total capacity of 150 gallons and a working capacity of 70 gallons if loaded to 0.9 times the 10-inch blade radius above the blade centerline. This volume permits the processing of a full 500-pound drum of EPON resin and allows the kneader blades to break the surface during the entire addition and kneading time. A drum hoist is provided to charge the mixer, which is located at ground level. Finished material is discharged into suitable containers by tilting the mixer.

In addition to the above-described mixer, two other pieces of equipment are utilized in the operation. The first of these is a 100-gallon stainless steel tank equpped with heating coil and air-driven propeller agitator. This tank is used for pre-dissolving solid resins (i.e., polyvinyl acetate) in the liquid EPON.

The second piece of auxiliary equipment is a Morehouse Hy-R-Speed mill equipped with replaceable carborundum stones with variable clearance. This mill is required for premixing of pigment or catalyst with the resin.

Processing

The procedure for typical batch runs as carried out in plant equipment is as follows:

Formulation

EPON 828	100 parts by weight
Vinylite AYAF	6
Allyl Glycidyl Ether	2
Asbestos	30
Dicyandiamide	8

Vinylite AYAF is dissolved in EPON resin by stirring 30 pounds with 380 pounds of EPON 828 for one hour at 160°C (320°F). The solution is transferred to the Read mixer and blended for two hours at about 70°C (158°F) with 150 pounds of asbestos.

In a separate vessel 120 pounds of EPON 828 is heated and mixed at 65-70°C (149-158°F) with 40 pounds of dicyandiamide (Dicy). The mixture is then passed through the Morehouse mill three times using 60-grit carborundum stones with sufficient clearance to keep the temperature below 70°C. The EPON-Dicy concentrate is then added to the EPON-Vinylite-asbestos mixture in the Read mixer and the charge completed by the addition of 10 pounds of allyl glycidyl ether. Blending at 70°C is continued for one hour to give the finished adhesive.

No study has been carried out as to the maximum allowable mixing temperature and all runs to date have been made at temperatures not exceeding 70°C.

Process Materials

EPON 828 and allyl glycidyl ether are supplied by Shell Chemical Corporation.

Asbestos 7TF is a product of the Johns-Manville Company and is classified under the designation of "shorts" and "floats".

Vinylite AYAF is obtained from Union Carbide and Carbon Corp. The designation AYAF determines the grade and viscosity of this polyvinyl acetate.

Dicyandiamide is a micro-pulverized grade supplied by American Cyanamid Company.

Titanium Dioxide is marketed under the trade name of Titanox T i-Pure-R and is supplied by E. I. duPont de Nemours and Co., Ltd.

Acetamine Orange is also obtained from duPont under the designation of GR Conc 175%.

Testing

Each batch of finished adhesive is tested for tensile shear strength and viscosity.

Test specimens are cleaned, assembled, and cured as outlined in Shell Chemical Corporation Technical Bulletins SC:52-46, SC:55-29, SC:55-30, and SC:55-47. They are then tested according to MIL-A-8331, which requires certain minimum performance standards.

Viscosities are run after constant temperature (77°F) storage for 48 hours, using a Brookfield Synchro-lectric viscometer with No. 7 Spindle. A maximum viscosity specification of 1 million cp measured at 2 rpm has been imposed upon Adhesive VI. No maximum has been set for Adhesive VIII and IX.

PREPARATION OF POLY(ALLYL GLYCIDYL ETHER)

Following is a description of a single pilot batch preparation of poly(allyl glycidyl ether), PAGE.

PAGE is made by polymerization of allyl glycidyl ether (AGE) principally through the double bonds. Although this polymerization can be initiated by oxygen and peroxides, di-tert-butyl peroxide is preferred because it allows better control over the reaction and results in a more uniform and reproducible product.

Operating Procedures

The procedure used in the plant preparation followed very closely that used in the laboratory on 5-gallon scale.

The reaction was carried out in a 100-gallon stainless steel Dopp kettle. This kettle was equipped with an overhead condenser and a 12-gallon accumulator. Mixing is provided by dual, counter-agitators, one set of which acts as a wallscraper. The vessel is heated by circulating hot oil through the jacket and temperature is automatically controlled by bypassing a part of the oil flow through a cooler. Nitrogen is supplied to the autoclave under pressure and the vessel is equipped with a back pressure controller.

Following are the steps used in making a batch of PAGE:

The autoclave was carefully cleaned free of any materials (acids, bases, heavy metal salts) which might catalyse the condensation of AGE or PAGE. Allyl glycidyl ether from normal plant production was charged (620 lbs.) and the vessel was flushed with nitrogen and pressured to 130 psig. The charge was heated to 180°C, and after checking to be sure that the heat transfer system was working properly, the flow of initiator was started at a rate of 0.1 lb. per minute per 100 lbs. of AGE. Almost immediately the temperature of the oil dropped about 10°C indicating that the exothermic reaction had started. Peroxide addition rates were maintained and the temperature was held at 180°C until the desired conversion was reached (22%) as indicated by the required increase in RI.

When the last of the initiator had been added, the charge was held at 180°C for 30 minutes to destroy the catalyst and then cooled. Nitrogen pressure was released and unreacted AGE was distilled to a final kettle temperature of 165°C at 5 mm Hg. abs. pressure. A nitrogen purge was used to aid in removing traces of AGE. The product was cooled to 30°C and drained to drums.

Yield

Charge:	AGE	619.6 Lbs.
	DTBP	14.9
Recovered:	AGE	472.3[a)]
	Product	128.3
Conversion AGE to Product		20.7%w
Raw Materials:	AGE	115 Lbs/100 Lbs Resin
	DTBP	11.6

Product Analysis

	Plant Batch	Lab Batch (Typical)
Epoxide, Eq/100g	0.724	0.732
Ester, Eq/100g	0.026	0.020
Hydroxyl, Eq/100g	0.12	0.13
Acidity, Eq/100g	< 0.001	< 0.001
Density 20/4	1.1223	1.1244
Molecular Weight	424	470
Viscosity, Brookfield, poise	5.5	6.3
Color, Gardner-Holt	9	7

Safety Precautions

Potential hazards of this preparation are associated with the fact that AGE can polymerize either through the double bond or through the epoxide group with the liberation of a large amount of energy. Furthermore, AGE and probably PAGE decompose exothermally at high temperatures (250°C) giving low molecular weight compounds and causing a rapid increase in pressure under confined conditions.

In this pilot batch, provision was made to drop the pressure on the autoclave quickly at the first indication of a run-away reaction. The vessel was also protected by a bursting disc.

a) Some losses were incurred during stripping, which could be avoided.