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MONTHLY REPORT

MAY 1973

MEMORANDUM

TO: Dr. H. E. Fritz
FROM: L. G. Krauskopf
DATE: June 1, 1973
SUBJECT: Vinyl Resins and Plasticizer Summary - Tarrytown Technical Center

HIGHLIGHTS

Non-Asbestos Tile

GAF has conducted an experimental plant trial of NAT on the "mottle line". Processability of NAT was judged very favorably; mechanical properties of the test tiles were not on target, but believed within range of a plasticizer adjustment. A larger-scale plant trial of NAT is planned by GAF for the near future.

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PLASTICIZERS FOR MOLDING AND EXTRUSION (211V50)

Franklin Plastics Corporation, Bound Brook, N.J.

Four different formulated PVC compounds representing potential candidates for approval by Ford Motor Co. were tested against the critical requirements in the supplied specifications.

Two compounds were unable to meet the specifications due to low percent elongation and a high specific gravity. Other critical requirements examined (hardness, flammability, etc.) were satisfactory. Further testing according to the specifications was not done due to the availability of test apparatus and/or materials. The data were presented to Mr. Robert Holzapfel of the subject company.

Franklin purchases about 2 MM lbs. of BAKELITE vinyl resins and 3 MM lbs. of FLEXOL plasticizers per year.

W. B. Drozdowski/D. B. Braun

VINYL RESINS FOR CALENDERING AND FLOORING (211V40)

Kentile Floors, Inc., Brooklyn, N.Y.

For comparative testing, Kentile submitted 9 vinyl chloride-vinyl acetate copolymers from various suppliers, and tile made from each. The objective of this study was to determine if copolymer resins from different suppliers controlled within specification limits for inherent viscosity and PVC content would exhibit measurable molten flow differences when each is formulated into vinyl asbestos tile.

This study has been completed resulting in the expected finding that copolymers exhibiting compositional factors (inherent viscosity x % PVC) within Kentile's specification limits of 34 to 37 show only minor differences in tile molten flow properties. Measurable differences in Brabender molten flow properties (tile) were determined for those copolymers found to be outside these specification limits. These differences were considered to be of sufficient magnitude to cause processing problems on tile production lines. A formal Technical Service Report documenting this information has been prepared and will be submitted to the customer.

R. P. Braddicks/R. F. Horvath

MECA, GO, CAPROLACTONE DERIVATIVES (211S93)

1. Crosslinked TDI/D-737

Two additional crosslinked TDI/D-737 (excess TDI) plasticizers are undergoing initial evaluation in PVC as a possible second generation solid type plasticizer with improved performance over PCL-780. One of the TDI/D-737 plasticizers prepared with 4% excess TDI appears to be similar to the first lot of a crosslinked plasticizer with respect to mechanical properties.

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The other crosslinked plasticizer (10% excess TDI) in PVC reveals slightly higher tensile and modulus values which is not surprising since it should contain more crosslinks. All of the crosslinked TDI/D-737 plasticizers contribute higher tensile values as the concentration of each plasticizer is increased. Even more surprising has been an increase in tensile values at constant levels of TDI/D-737 in PVC when the EPO is incorporated in increasing levels up to 10 PHR. This is in contrast to "Santicizer" 334F in PVC, which is being examined as a control along with the crosslinked plasticizers.

One of the objectives for a second generation solid plasticizer is a reduction in Durometer values compared to PCL-780/PVC. The crosslinked TDI/D-737 plasticizers reveal a slight reduction in Durometer values when compared to PCL-780 but are inferior to "Santicizer" 334F in its ability to produce relatively low Durometer values. A reduction in the molecular weight of the crosslinked plasticizers may be in order to reduce the Durometer values. The elongation values exceed that of 334F.

Further work will explore the moisture and solvent resistance of the crosslinked TDI/D-737 plasticizers in comparison to PCL-780 and "Santicizer" 334F.

R. E. Godlewski

2. PCL-780

A comparison of an older lot of PCL-780 pelletized (lot B-1040) with a more recent lot of PCL-780 (lot 9533-59) which was in shredded form and devolatilized of styrene was undertaken to determine whether the former had changed in plasticizing efficiency due to a reported decrease in reduced viscosity caused by moisture depolymerization of the polycaprolactone segments with time. Both were incorporated into QSAH-7 with and without EPO. The mechanical properties including low temperature performance were similar. One difference noted visually was the more nervy character of the more recent lot in PVC during processing. This is probably due to the higher reduced viscosity value for the recent lot. This study alleviates the fear that the sampling of the older lot PCL-780 would not be typical of what could be produced currently.

R. E. Godlewski

3. Compatibility of PCL-780 and Polyurethanes in PVC

It has been noticed that a powdery bloom does appear on the surface of molded plaques of PCL-780/PVC and the TDI/D-560 plasticized PVC with time. It has been theorized that too high a level of metallic stabilizer in the formulation will upset the solubility of the solid plasticizers causing bloom. However, preliminary studies have indicated that perhaps too little of the metallic stabilizer causes the formation of bloom. This in combination with increasing levels of EPO also encourages bloom formation. It is too early to speculate on what is really occurring but evidence is being accumulated to make some sensible judgments as to factors influencing bloom and ways to avoid it.

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This study has also pointed out that environmental conditions (temperature and humidity) play an important role in bloom formation.

R. E. Godlewski

4. Polyurethane Elastomers in PVC

I have been recently assigned to participate in a portion of the work connected with this project. Assimilation of the necessary background information is in progress. Plans are being formulated for the necessary laboratory work. At present, polyurethane solid plasticizers identified as 10184-80-1, 2, 3, 4, 5 representing D-560 and D-737 polyols reacted with various stoichiometric quantities of TDI are being scheduled for evaluation as permanent PVC plasticizers. A review of previous test procedures is being made in an effort to select those tests which provide data most beneficial in effecting a reliable evaluation of these new candidates.

R. P. Braddicks

PCL IN NAT (211S94)

1. GAF Corporation, Vailsgate, N.Y.

An experimental plant run of the NAT disclosure formulation has been made at the GAF, Vailsgate factory. This test run demonstrated that the Banbury and milling properties of this composition are within acceptable limits for GAF's tile operations. Although this evaluation was conducted on the mottle line and not the tile production unit, it is felt that the results are sufficiently encouraging to now schedule a test run on the tile production line. The tile produced during this evaluation proved to be too hard resulting in poor impact resistance. It is believed that tile reformulated to proper hardness will exhibit improved impact resistance. Dimensional stability of this tile proved to be well within the 24 mils per 12 inch limit.

R. P. Braddicks

2. NAT PATENT APPLICATION STATUS

The original filing of the NAT patent application was rejected by the examiner on the grounds that the claims embodied in this document were obvious to one skilled in the art. As a result of an appeal, it became apparent that additional data would have to be generated before this examiner would consent to a reversal of his decision. G. Skoler of UCC's Legal Department has requested that additional work be done to establish that PCL-700 does in fact improve the "hot tensile strength" of non-asbestos containing stocks and that our claims do not conflict with compositions referenced in the Henriques patent # 3,091,597. This work has been completed and the results obtained provide the needed support. A Project Report summarizing this work will issue shortly.

R. P. Braddicks/R. F. Horvath

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VINYL RESINS FOR WIRE AND CABLE APPLICATIONS (223T90)

Phelps-Dodge Industries, Yonkers, N.Y.

This company has periodically experienced problems with rapid clogging of extruder screen packs. It is their belief that these problems occur most frequently when they are using our QSAN-7 vinyl resin. In late April, they sent us the screen packs which have caused the problem along with the dryblend they were using. Chemical and screen analyses indicated the clogging was caused by agglomerates of calcined clay filler.

In early May we visited their plant to try to learn more about the problem. Observation of the mixing operation and examination of amperage and temperature charts for their Beacon Mixer strongly suggested that their mixing cycle was too short. It appeared to us that clay filler was being charged to the mixer before a dryblend of resin and plasticizer had been achieved. This suggests that plasticizer still present on the surface of the resin particles was being adsorbed by the filler causing filler agglomerates.

Based on this theory, several suggestions for changes in mixing cycles were offered. We also recommended an inspection of the inside of the mixer, which reportedly was last done several years ago. They will report their findings to us some time in June.

D. B. Braun

PCL-780 IN WIRE AND CABLE APPLICATIONS (223T60)

1. Times Wire and Cable Corp., Wallingford, Conn.

PCL-780 plasticizer was introduced to personnel of Times Wire and Cable Corp. during an omnibus technical meeting at Bound Brook in mid-May.

They immediately expressed interest in its potential use in a non-migrating PVC jacket for polyethylene insulated coaxial cables. Since they have no facilities for laboratory or plant scale mixing of PVC, they requested we supply them with 25 lbs. of a candidate formulation to try in a small plant scale trial. We agreed to supply the material. They also indicated a willingness to permit us to observe the trial.

D. B. Braun

2. PCL-780/PVC Wire and Cable Insulation for Non-Moisture Applications

Efforts have centered around the development of a PCL-780/PVC insulation for U.L. 758 125°C rated appliance wire.

Six compounds were extruded as part of a factorially designed experiment which examines the effect of five formulating variables (resin molecular weight, lead stabilizer concentration, filler level, use of antioxidant and antimony trioxide). 1/64" wall insulation was produced on # 20 AWG tinned solid copper conductor. No degradation occurred at extrusion temperatures in excess of 400°F. However, a generally rough surface insulation was produced.

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The PCL-780/PVC system has already demonstrated the ability to meet oil resistance, cold bend and horizontal flame resistance requirements necessary for the 125°C rating. Thermal stability and deformation tests of this very rigorous application are being conducted on the extruded wire.

W. B. Drozdowski/D. B. Braun

3. PCL-780/PVC Insulation for Xerox Copy Machine Wire

Once again, the fine wire (# 22 AWG) thin wall (1/64") copy machine insulation extruded at Prestolite has failed the Xerox vertical flame test. The three compounds tested contained 5, 6 and 7 PHR of antimony trioxide respectively. Their Limited Oxygen Index was about 30%. The addition of color concentrates employed by Prestolite played no significant role in the flammability characteristics.

Samples of the extruded wire were retested at Tarrytown. Although they did not meet the Xerox vertical flame requirement (less than 6 sec. extinguishing time), the samples illustrated the great deviation found in this test method. The U.L. 44 vertical flame test was also run on the extruded wire. All samples easily passed this test.

The continued investigation of this application must determine if a higher LOI is needed (achieved by an increase in antimony trioxide) or whether the Xerox vertical flame test is a realistic test which can be passed at the necessary insulation construction.

W. B. Drozdowski/D. B. Braun

ASBESTOS (950S74)

1. Uvalde Rock Asphalt Co., Houston, Texas

This customer has complained that the use of UCC SG-100 asbestos in place of its standard "Stragari" (Yugoslavian) asbestos increases tile formulation costs. It is claimed that both increased asbestos and binder levels are required with SG-100 to match tile dimensional stability of "Stragari" containing tile.

Selected tile samples were provided by Uvalde for a comparative study. These samples represented production tiles made with UCC SG-100, Johns Manville "Ultrabestos" and "Stragari" asbestos and reportedly were formulation adjusted for equivalent dimensional stabilities. They were submitted to selected laboratory testing. This included determination of McBurney hardnesses, tensile strength and ultimate elongation at 180°F, tensile stress relaxation at 180°F, Brabender equilibrium torques at 130°, 140° and 150°C and tile oven shrinkage values at 220°F.

Based upon the results of this study of the rheological properties of test tiles submitted, it is concluded that the reported reinforcing deficiency of SG-100 has not been confirmed. Other means of improving tile dimensional stability have been provided in a Technical Service Report in which this work has been summarized.

R. P. Braddicks/R. F. Horvath 13

2. GAF Corporation, Vailsgate, N.Y.

Several months ago, aluminum tristearate was recommended to this customer as a potential additive capable of reducing the water sensitivity of tile made with SG-100 asbestos. This recommendation has been evaluated by GAF personnel and its value for this purpose has been confirmed. This information will be made available to other asbestos customers when needed.

R. P. Braddicks

OPS-83, PLASTICIZERS (219S10)

MCA Committee on Phthalates

Two publications appeared in April, 1973 which continue to sustain concern about phthalates:

1. R. J. Rubin and P. P. Nair - both having contributed toward the raising of concern over phthalates - wrote a letter to the editor of The New England Journal of Medicine in which they point out that phthalates were found to be present in 46 to 73% of a test group of people that never had received blood transfusions from plasticized PVC bags. This part of their work had not been previously published along with data showing high incidence of DOP in transfused patients. While Rubin and Nair express belief that the source of DOP in untransfused patients is environmental, they admit endogenous biosynthesis remains as a potential source and that the biologic or toxicologic role of phthalates has not been fully defined.

2. M. Schnitzer and S. U. Khan composed a textbook on Humic Substances in the Environment. This book identifies specific phthalate esters occurring in soil samples and conjectures that the sources could be biological. However, the major phthalates found are common in industry:

di(2-ethylhexyl) phthalate
butyl, benzyl phthalate
di(butyl) phthalate
di(octyl) phthalate

L. G. Krauskopf

FIELD CONTACTS

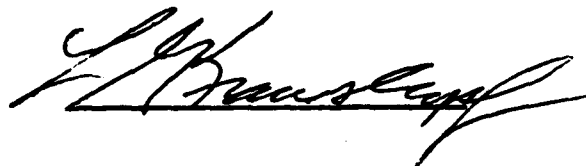
<u>Date</u>	<u>R&D Personnel</u>	<u>Company and Subject</u>
May 2, 1973	D. B. Braun	Phelps Dodge, Yonkers, N.Y., Screen plugging problem
May 5-10, 1973	D. B. Braun	ANTEC of the SPE, Montreal, Can.
May 16, 1973	D. B. Braun	Times Wire & Cable Corp., Meeting at Bound Brook R&D, Introduction of PCL-780

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REPORTS ISSUED

<u>Date</u>	<u>Authors</u>	<u>Report Type and Title</u>
April 24, 1973	R. E. Godlewski W. B. Drozdowski D. B. Braun	<u>Project Report</u> , "Technical Service Supporting Plasticizer Sales to the Compounders and Specialty Segment of M.A. 211, 1st Quart. 1973", File No. P-73-68.
May 15, 1973	R. P. Braddicks W. B. Drozdowski R. F. Horvath	<u>Project Report</u> , "Evaluation of the Phthalate Ester of Hydrogenated Croton Oil", File No. P-73-78.
May 16, 1973	R. P. Braddicks R. F. Horvath	<u>Technical Service Report</u> , "Interchange of Asbestos Fibers and Its Effect on the Rheological Properties of VA Tile", File No. TSR-73-78.
May 23, 1973	R. P. Braddicks R. F. Horvath	<u>Technical Service Report</u> , "Compositional Factor of Vinyl Chloride-Vinyl Acetate Copolymers and Its Effect on Tile Molten Flow Properties", File No. TSR-73-80.

L. G. Krauskopf/et



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