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Chemistry

POLYVINYL PLASTISOL QUALITY STUDY

C. R. Schmitt

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**UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT**

operated for the **ATOMIC ENERGY COMMISSION** *under* **U. S. GOVERNMENT** **Contract W-7405 eng 26**

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OAK RIDGE Y-12 PLANT
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Date Issued: March 9, 1966

Report Number Y-1508

Chemistry - TID-4500

UNION CARBIDE CORPORATION

Nuclear Division

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Contract W-7405-eng-26

With the US Atomic Energy Commission

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C. R. Schmitt

Oak Ridge, Tennessee

Report Number Y-1508

Chemistry - TID-4500

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ABSTRACT

Laboratory studies were made on samples of various production quantities of polyvinyl chloride plastisols to determine the cause of cracks, blisters, and other imperfections in flexible molds made from the material by heat curing.

Moisture was found to be the major troublesome contaminant of those batches that produced cured molds of inferior quality. Although the quality of the plastisol could be upgraded by vacuum treatment to remove moisture, degradation re-occurred after subsequent exposure to a moist atmosphere.

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INTRODUCTION

Polyvinyl plastisol is a colloidal dispersion of polyvinyl chloride resin in a plasticizer or a combination of plasticizers. In addition to these two major ingredients, commercial polyvinyl plastisols usually contain fillers and pigments, stabilizers, thinners, and surface acting agents to provide specific properties for the finished product or to modify flow characteristics.

To cure (solidify) a liquid polyvinyl plastisol compound, it is only necessary to heat it to the fusion temperature (approximately 180° C). By the application of heat, the viscosity of the plastisol increases until a gelled or solid state is reached and the desired physical properties such as tensile strength, tear strength, elongation, and Durometer hardness are developed by fusion.

Flexible molds of various sizes, shapes, and wall thickness are made at Y-12 by heat curing polyvinyl chloride plastisols which are purchased from various commercial sources. These flexible molds are then filled with powders and pressed isostatically in pressure vessels containing mineral oil as the pressure-transmitting fluid.

Recently, cured polyvinyl chloride made from plastisols has exhibited cracks, blisters, holes, and other defects which seriously impair its use for isostatic pressing. A laboratory test program was therefore initiated to determine and eliminate the cause of these imperfections, which were suspected to originate from some volatile constituent in the raw plastisol compound.

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SUMMARY

Laboratory studies made on various production quantities of polyvinyl chloride plastisol indicated that moisture was the principal troublesome cause for cracks, blisters, and other imperfections that develop in the flexible molds made from the material by heat curing. Moisture is picked up by the plastisol because it is highly hygroscopic.

As a result of this study it is recommended that proper storage conditions be provided to prevent moisture pickup. It is also recommended that cured polyvinyl plastisol be used as a coating for the drums used to store the raw plastisol. This would eliminate the presence of shredded fragments of polyethylene liner material which has been found in some of the plastisol.

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EXPERIMENTAL WORK

SAMPLING

Polyvinyl chloride plastisol at the Y-12 Plant is currently obtained from three commercial vendors and is shipped and stored in 55-gallon drums fitted with a polyethylene liner. Samples of various plastisol batches as currently stored in unopened drums at the warehouse were obtained for laboratory analyses and evaluation.

For those batches chosen, a sample was taken from the top of each drum by scooping the plastisol into a cardboard cup; a sample was withdrawn from the bottom of each drum by a modified sampling device and procedure described in ASTM-D-1045-49T.⁽¹⁾ This device consisted of a one-quart wide-mouth glass bottle fitted with a removable mushroom-type stopper and attached to a steel rod. After the sample bottle and rod had been submerged to the bottom of the drum, the stopper was pulled out by means of an attached wire and the jar was allowed to fill with plastisol.

TEST CURINGS

Effect of Sample Point

Test curings of the top and bottom fractions of the various plastisols were then made by pouring approximately 80-gram portions of the sample into Petri dishes (11.0 cm D) and heating in an oven provided with hot-air circulation for 1 hour at 160° C. For each batch, the cured specimen obtained from the drum-bottom plastisol sample had a distinctly different appearance from the corresponding drum-top specimen. The drum-top curings exhibited severe cracks, blisters, and similar bubble-like defects, whereas the drum-bottom curings were practically free of defects. Photographs showing the various degrees of blistering and other defects in the cured top and bottom fractions of batches from Vendor A plastisol are shown in Figure 1.

Similar test curings made on four drums of Vendor C plastisol exhibited severe bubble-like defects for the samples taken from both the top and bottom of the drums.

Effect of Air and Water Additions

The difference in quality between the top and bottom fractions of plastisol from a single drum indicated that some volatile or low-density constituent, such as entrapped air or moisture, may have been responsible for these imperfections. The deleterious effects of trapped air and water on the quality of polyvinyl plastisol test curings are shown in Figure 2. Air agitation was provided by rapidly stirring the plastisol in a Petri dish with a spatula for approximately two minutes. Distilled

Figure 1. COMPARISON OF HEAT-CURED VENDOR A POLYVINYL PLASTISOL FRACTIONS FROM THE TOP AND BOTTOM OF THE DRUMS. (1.5X)

water was added by injecting it into the liquid plastisol with a fine (Size 26) hypodermic needle.

CHEMICAL AND PHYSICAL CHARACTERIZATION

An attempt was made to correlate the hydroxyl number, acid content, and water content of the raw plastisol samples with the quality of the resultant test curings. The hydroxyl number measures the amount of hydroxyl attached to a carbon atom which, in turn, is attached to a carbon or hydrogen atom. These data, summarized

Figure 2. EFFECTS OF AIR AGITATION AND WATER INJECTION ON THE QUALITY OF VENDOR A POLYVINYL PLASTISOL TEST CURINGS. (1.5X)

in Table 1, indicate that none of these analyses can be used to distinguish between "good" and "bad" raw plastisol batches.

The wide divergence in results obtained by two independent laboratories with the Karl Fischer method of water analysis also indicates that this method is not well suited for these high-viscosity plastisols. According to Meeker,⁽²⁾ determination of water in some organic systems is difficult or impossible by the Karl Fischer method.

Infrared analyses of top and bottom fractions of a drum of Vendor A, Batch 1 plastisol showed the presence of hydroxyl and vinyl groups in the top fraction, whereas in the bottom fraction the hydroxyl groups were in much smaller quantity and the vinyl groups did not appear.

Some of the volatile ingredients of a 200-milliliter sample of a "bad" batch of plastisol were vacuum treated at room temperature and collected in a trap cooled by liquid nitrogen. After two hours of pumping, approximately 2 cubic centimeters of a two-layered condensate had been collected in the cold trap. Infrared analyses

Table 1
CHEMICAL ANALYSES OF RAW POLYVINYL PLASTISOLS

Vendor	Batch Number	Sample Point	H ₂ O ⁽¹⁾ (ppm)		Hydroxyl Number ⁽²⁾ (equivalent OH/100 gms)	Acidity (equivalent H ₂ /100 gms)	Quality of the Test Curing
			Lab A	Lab B			
A	1	Top of Drum	0.25	0.10	0.021	0.003	Bad
A	1	Bottom of Drum	0.44	0.07	0.014	0.003	Good
A	2	Top of Drum	0.31	0.31	-	-	Bad
A	2	Bottom of Drum	0.27	0.27	-	-	Good
A	3	Top of Drum	0.30	0.12	0.011	0.003	Bad
A	3	Bottom of Drum	0.28	0.10	0.013	0.003	Good
A	4	Top of Drum	0.30	0.09	0.022	0.003	Bad
A	4	Bottom of Drum	0.29	0.11	0.021	0.003	Good
A	5	(3)	0.25	-	0.033	0.004	Bad
B	1	Top of Ccn ⁽⁴⁾	0.21	-	-	-	Bad
B	2	Top of Drum	0.23	-	-	-	Bad
B	3	Top of Drum	0.23	-	-	-	Bad
B	3	Bottom of Drum	0.23	-	-	-	Good
B	3 ⁽⁵⁾	Top of Drum	0.09	-	-	-	Bad
B	3 ⁽⁵⁾	Bottom of Drum	0.20	-	-	-	Good
C	1	Top of Drum	0.24	-	-	-	Bad
C	1	Bottom of Drum	0.24	-	-	-	Bad
C	1 ⁽⁶⁾	Top of Drum	0.25	-	-	-	Bad
C	1 ⁽⁶⁾	Bottom of Drum	0.24	-	-	-	Bad

(1) By Karl Fischer titration.

(2) By tetrahydrofuran-lithium aluminum hydride method.

(3) Shop sample stored in a 5-gallon can for several weeks prior to sampling.

(4) Experimental 5-gallon sample supplied by vendor for acceptance tests.

(5) Second drum of Batch 3.

(6) Second drum of Batch 1.

showed that the bottom layer was primarily water. The top layer was mostly aliphatic compounds with some $-C-CH_3$ groups present. Some chains contained 5 - 6 CH_2 groups. Some hydroxyl, aromatic, and carbonyl groups were also found to be present. The aromatic group was believed to be conjugated with double bonds as in styrene, and the carbonyl was believed to be a ketone.⁽³⁾

An attempt was made to further identify the organic layer by using a Perkin-Elmer gas chromatograph. Although twenty distinct peaks were found, only two were identified; namely, hexane and heptane. A typical gas chromatographic analysis⁽⁴⁾ of the bottom layer of condensate was also made. Results, as shown in Table 2, confirmed the previous infrared results which had established the bulk of the condensate sample to be water.

Table 2
GAS CHROMATOGRAPHIC ANALYSES OF THE BOTTOM LAYER OF
THE CONDENSATE OBTAINED FROM RAW
POLYVINYL PLASTISOL
(Vendor A Plastisol, Batch 2, Top of Drum)

Peak Number	Peak Identification	Percent of Sample
1	Heptane	0.07
2	-	0.04
3	-	0.07
4	-	0.04
5	-	0.01
6	Decane	0.28
7	Water	99.49

Test curings of the "bad" plastisol were bubble-defect free after this vacuum treatment indicating that the troublesome contaminant in the raw plastisol had been removed.

Data on viscosity, dissipation factor, capacitance, and weight loss upon curing at 160° C were also obtained for various batches of raw polyvinyl plastisol and related to the quality of Petri dish test curings made at 160° C. Results are summarized in Table 3.

Measurements of the dissipation factor and capacitance were obtained using the apparatus shown in Figure 3, which consisted of a capacitance probe and impedance bridge (General Radio Company, Type 1650A). The capacitance probe was constructed from a variable, 43-plate, 13.5 - 320 $\mu\mu F$ capacitor fixed at its maximum range. The data of Table 3 do not show a positive correlation of the dissipation factor or capacitance with the quality of polyvinyl plastisol, and it is believed that the use of different plasticizers, and concentrations, by the various vendors probably accounts for the wide variation in results.

Table 3
CHARACTERIZATION OF VARIOUS BATCHES OF POLYVINYL PLASTISOL

Batch Identification	Sample Point	Viscosity ⁽¹⁾ (centipoise)	Dissipation Factor ⁽²⁾	Capacitance (μF)	Weight Loss on Curing (%)	Appearance of Test Curing
Vendor B, Batch 1 ⁽³⁾	Top of Can	1,638	0.93	1,660	1.34	No bubbles; perfect.
Vendor B, Batch 3	Top of Drum	5,277	3.50	1,625	1.94	No large bubbles; small number of small bubbles.
Vendor B, Batch 3	Bottom of Drum	4,970	2.90	1,621	1.76	No large bubbles; small number of small bubbles.
Vendor A, Batch 2	Top of Drum	3,621	1.29	1,575	0.56	Many medium bubbles.
Vendor A, Batch 2	Bottom of Drum	3,844	1.32	1,578	0.56	Many small bubbles.
Vendor A, Batch 3	Top of Drum	-	1.32	1,570	0.49	Very many medium bubbles.
Vendor A, Batch 3	Bottom of Drum	5,013	1.28	1,560	0.64	Few small bubbles.
Vendor A, Batch 4	Top of Drum	4,080	1.72	1,622	0.76	Very many large bubbles.
Vendor A, Batch 4 ⁽⁴⁾	-	-	1.67	1,620	0.71	Very many large bubbles.
Vendor C, Batch 1	Top of Drum	9,350	2.55	3,550	0.91	Many large bubbles.
Vendor C, Batch 1	Bottom of Drum	16,880	1.17	1,600	1.20	Many large bubbles.
Vendor C, Batch 1	Bottom of Drum	15,940	1.13	1,540	0.93	Many large bubbles.

(1) Each value represents the average of three determinations at 25° C using a Brookfield viscometer.

(2) At 1.0 kilocycle.

(3) Experimental 5-gallon sample supplied by the vendor for acceptance tests.

(4) After vacuum pumping and exposure to room air for one month.

Figure 3. CAPACITANCE PROBE AND IMPEDANCE BRIDGE FOR RAW PLASTISOL QUALITY STUDIES.

MOISTURE MONITORING

Capacitance Probe Tests

Instruments based on capacitance and radio-frequency power absorption are very adaptable for the detection and monitoring of small amounts of moisture in a variety of solid and liquid materials. To determine the effectiveness of the capacitance probe for moisture detection, measurements of capacitance and dissipation factor were made on a single batch of Vendor A plastisol to which had been added measured amounts of distilled water. Results, as summarized in Table 4, indicate a general correlation between the dissipation factor and the amount of water present in the plastisol.

After adding water to the plastisol, the dissipation factor and capacitance values were found to be continually changing. With continued gentle stirring using the probe, the increasing rate of change of readings became slow enough to provide a reasonable measurement, but never did attain equilibrium. A batch of plastisol to which 0.1% H_2O had been added still had not come to a constant value after sixteen hours, suggesting that slow absorption of water by the plastisol was occurring.

Table 4
EFFECT OF THE MOISTURE CONTENT OF RAW PLASTISOL ON THE
DISSIPATION FACTOR AND CAPACITANCE
(Vendor A Plastisol, Batch 1)

Water Added (wt %)	Dissipation Factor ⁽¹⁾	Capacitance (μF)
None	1.40	1570
None ⁽²⁾	1.30 ⁽²⁾	1560 ⁽²⁾
0.05 ⁽²⁾	1.40 ⁽²⁾	1575 ⁽²⁾
0.10	1.48	1595
0.10 ⁽²⁾	1.50 ⁽²⁾	1590 ⁽²⁾
0.20	1.69	1682
0.20 ⁽²⁾	2.33 ⁽²⁾	2640 ⁽²⁾
0.30 ⁽²⁾	2.57 ⁽²⁾	3140 ⁽²⁾
0.50	2.62	3350
None ⁽³⁾	1.40	1545

(1) At 1.0 kilocycle.

(2) Denotes values obtained in an alternate series of measurements.

(3) After vacuum treatment to remove volatiles.

Calcium Carbide Tests

An apparatus was constructed to measure the moisture content of polyvinyl plastisol quantitatively, based on the reaction of calcium carbide and water to form acetylene gas. The unit consisted of a five-inch-diameter stainless steel vessel equipped with a flanged lid and Teflon gasket. By using a length of 1/4-inch tubing extending from the top of the vessel, any gas formed was collected by displacement using an inverted graduated cylinder filled with silicone oil. The vessel was charged with 30 grams of calcium carbide pellets and 500 milliliters of plastisol to which water had been added by rapid spatulation.

The variation in the amount of acetylene gas collected after 24 hours contact and the water content of polyvinyl plastisol is shown by the graph in Figure 4.

HEAT-CURED PLASTISOL EVALUATIONS

Numerous Petri-dish test curings were made in an oven at 160°C to study the effects of viscosity, water content, and vacuum treatment on the quality of heat-cured plastisol. Photomicrographs and data showing the effects of some of these treatment conditions are presented in Figure 5 and Table 5.

In general, there was little correlation between the viscosity of the raw plastisol and the quality after heat curing. As shown in Figure 5 and Table 5, vacuum-treated plastisol having a viscosity as high as 16,000 centipoise gave a defect-free cure. For relatively high-viscosity plastisol (≥ 6000 centipoise), however, it is expected that any entrapped air-bubble defects in the cured plastisol would probably result.

Figure 4. VARIATION IN THE VOLUME OF ACETYLENE GAS PRODUCED BY THE REACTION BETWEEN CALCIUM CARBIDE AND WATER IN POLYVINYL PLASTISOL.

Table 5
DATA FOR PHOTOMICROGRAPHS SHOWN IN FIGURE 5

Plastisol Identification	Photomicrograph Number in Figure 5	Approximate Viscosity (centipoise)	Treatment	Appearance
Vendor A, Batch 4	A	4,000	None	Numerous bubble defects.
Vendor A, Batch 4	B	4,000	After vacuum pumping.	No bubble defects.
Vendor A, Batch 4	C	4,000	Exposed to room air 4 days after vacuum pumping.	Numerous bubble defects.
Vendor C, Batch 1	D	16,000	None	Numerous bubble defects; visible flow pattern lines on surface.
Vendor C, Batch 1	E	16,000	After vacuum pumping.	No bubble defects; visible flow pattern lines on surface.
Vendor A, Batch 1	F	4,000	0.1% H ₂ O added by injection.	Numerous bubble defects and cracks.
Vendor C, Batch 1	G	16,000	0.5% H ₂ O added by injection prior to vacuum pumping.	No bubble defects; visible areas of discoloration and flow pattern lines on surface.

Figure 5. PHOTOMICROGRAPHS OF HEAT-CURED POLYVINYL PLASTISOL AFTER VARIOUS TREATMENTS. (2X)

Addition of 0.1% water to various raw plastisols caused severe interior defects to occur in every test curing even though the original plastisol had shown high-quality, bubble-free test curings. It is significant that only a relatively small amount of water (0.1%) can cause severe imperfections in cured plastisol. The addition of relatively larger amounts of water (0.5% H_2O) caused large cracks and severe surface disruptions in addition to interior bubble defects.

All batches of the various plastisol samples gave high-quality, defect-free test curings after vacuum pumping. One sample was injected with 1.2 cubic centimeters of water (0.6%), then vacuum treated for two hours in the apparatus shown in Figure 6. The entire amount of water was recovered quantitatively in the condensate receiver. As shown in Figure 5(g), vacuum treatment of a high-viscosity plastisol resulted in a bubble-free cure even after it had been doped with as much as 0.5% water. The dark areas shown in the photomicrographs of Figure 5 are believed to be differences in coloration which may have resulted from localized concentrations of acid which had been extracted by water and then left behind when the water was volatilized. Some of the flow lines resulting from pouring the high-viscosity plastisol into the Petri dish are also visible.

Figure 6. APPARATUS FOR VACUUM TREATING RAW POLYVINYL PLASTISOL.

It may be concluded from an inspection of Photomicrographs A, B, and C (Figure 5) that a bad-quality plastisol can be upgraded to a satisfactory material by vacuum treatment to remove volatiles, but it degrades upon subsequent exposure to atmospheric moisture, indicating that the plastisol has a high hygroscopicity.

HYGROSCOPICITY STUDIES

Using the apparatus shown in Figure 6, a sample of polyvinyl plastisol was vacuum treated for several hours to remove air and moisture. Portions of this sample were

then placed in weighed Petri dishes and exposed to dry and moist air environments. Results are shown in Figure 7.

Figure 7. HYGROSCOPICITY OF POLYVINYL PLASTISOL.

The sample of plastisol that was exposed to room air lost weight at a constant rate, which indicates the loss of volatile plasticizer by evaporation. After 94 hours, the plastisol exposed to 100% relative humidity had gained more than 0.5 wt % moisture. When this sample was put in a desiccator containing Drierite granules, a loss in weight was observed (Curve D). No attempt was made to determine if this weight loss was entirely due to moisture absorption; some of the loss could have been due to plasticizer absorption. The dips shown in Curves B and C after an exposure of twenty-two hours are attributed to changes in the sequence of weighings which caused changes in relative humidity conditions inside the desiccators. Test curings made on these humidity-exposed plastisols by heating in an oven at 160° C for one hour showed blister defects. Test curings of the vacuum-treated plastisol prior to exposure were free of defects.

CONCLUSIONS AND RECOMMENDATIONS

It has been demonstrated that polyvinyl plastisols are extremely hygroscopic and can be degraded by absorption of moisture from the atmosphere. Thus, proper storage conditions for this material prior to use are of primary importance to prevent moisture pickup and excessive evaporation of the plasticizer.

Another important condition affecting the quality of the plasticizer is the containing vessel used for storage. According to the Society of the Plastics Industry,⁽⁶⁾ "in choosing the container for the plastisol, the physical properties of the latter should be considered since softening or solution of the container lining or chemical attack of the metal by the liquid components of the plastisol may introduce extraneous foreign matter or discolor the product". In this regard, current Y-12 Plant practices employing polyethylene liners in 55-gallon steel drums for plastisol is not ideal inasmuch as one batch of plastisol was found where chemical attack of the polyethylene had occurred.

As shown in Table 1, both the top and bottom fractions of two drums of Vendor C plastisol exhibited a high degree of bubble-like defects after test curing. It was observed that this plastisol was considerably more viscous than any of the other plastisol samples. In one of the two drums of this material sampled, the polyethylene drum liner had deteriorated and small shredded fragments of polyethylene were interspersed throughout the top and bottom fractions. Such liners should not be used in contact with polyvinyl chloride plastisols because polyethylene is appreciably soluble in some of the organic plasticizers which are present in these plastisols.

Since cured polyvinyl plastisol has outstanding chemical resistance and good flexibility and toughness,⁽⁷⁾ it is believed that this material could be used advantageously as a drum coating for raw plastisol shipment and storage. The plastisol-container coating could be applied by dipping or spraying.⁽⁷⁾

After storing a batch of plastisol from Vendor B in the laboratory for three months it was observed that phase separation had occurred, indicating that this batch was of inferior quality. Although the aging characteristics of plastisol are variable, "properly formulated and compounded plastisols have been aged for years without any detectable deterioration in the form of phase separation, discoloration, etc."⁽⁶⁾

It was found that the quality of a batch of polyvinyl plastisol could best be evaluated rapidly and expediently by pouring approximately 80-gram samples of the plastisol into Petri dishes and oven curing for one hour at 160° C to provide a cured plastisol wafer having a thickness of approximately 1/2 inch. This test afforded a direct visual estimation of any cracks, bubbles, and other defects that would be experienced in production-scale heat-curing operations.

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