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MOISTURE EFFECTS ON THERMOSET
MOLDING COMPOUNDS

PDO 6984548, Interim Report

J. M. Conway, Project Leader

Project Team:
C. B. Swearngin
J. B. Watkins

March 1973

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**Project Leader:
J. M. Conway
Department 814**

**Project Team:
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Interim Report**

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MOISTURE EFFECTS ON THERMOSET MOLDING COMPOUNDS

BDX-613-821 (Rev.), UNCLASSIFIED Interim Report, March 1973

Prepared by J. M. Conway, D/814, under PDO 6984548

The effects of moisture on the physical properties of premolded Fiberite E-9405, Fiberite FM-1132, and Allied Chemical diallyl phthalate 52-20-30 were determined. Test results confirmed that the mechanical and electrical properties of molded specimens of E-9405 and FM-1132 deteriorated substantially when the raw materials were exposed to humidity at 25°C. Specimens molded from diallyl phthalate experienced limited degradation of physical properties and then only when the raw material was exposed to high relative humidity.

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SUMMARY

Moisture absorption in bulk thermosetting molding compounds often causes large increases in the spiral flow length¹ of these compounds and prior to this study was suspected to have caused significant degradation in the physical properties of molded parts. In this study the effects of moisture absorption were determined for three types of molding compounds: a mineral filled epoxy, a cotton filled phenolic, and a long glass fiber filled diallyl phthalate resin compound. Of particular interest were the changes in the mechanical, electrical, and flow properties when varying amounts of moisture were absorbed into the premolded forms of the materials. Test results supported the following conclusions.

- Prolonged exposure to humid conditions results in a significant reduction in the mechanical and electrical strengths of Fiberite E-9405 and especially FM-1132 because of the tendency of these compounds to absorb moisture.
- Diallyl phthalate 52-20-30, because of its lower tendency toward moisture absorption, retains its mechanical and electrical strengths in humidity environments up to approximately 90 percent humidity before significant deterioration occurs.
- Molding compounds received from a vendor contain significant amounts of residual moisture. Generally the physical properties, spiral flow, shrinkage, and moisture content of these compounds closely parallel material that has reached equilibrium in 50 percent humidity.
- Inconsistencies in lot acceptance testing and deteriorations in physical properties caused by moisture require that better packaging and handling techniques be evaluated.

¹J. E. Hess, "Effect of Moisture on Flow of Thermoset Molding Compounds", Modern Plastics, Nov. 1971.

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DISCUSSION

SCOPE AND PURPOSE

The effects of moisture in stored and incoming thermoset molding compounds are known to be related to many problems associated with processing variations. Inconsistent shrinkage, and B-staging* are examples. Moisture is suspected of being a factor in the deterioration of the mechanical and electrical properties of molding compounds. Recent examples of such problems were encountered with lots of diallyl phthalate and epoxy molding compounds (Lots L9806 and W-6924) which had B-staged to such an extent during in-house storage that these lots could not meet the minimum spiral flow length requirements called out in the applicable material specifications.

One lot of Fiberite E-9405 (Lot W-3444) that had originally passed lot acceptance testing failed a shelf life extension test when the flexural strength could not meet the minimum requirement. Past experience has shown many variations in acceptance test results for spiral flow in materials that were supposedly alike. It is quite common for a material that had originally passed acceptance testing to be deviated at a later date due to spiral flow increasing out of tolerance. A recent example was Lot W-3579 of Fiberite E-9405 which before storage had a rated spiral flow under the 44 inch (112 cm) material specification high limit that, after successive retests, eventually had deviated to 80 inches (203 cm). Such occurrences, coupled with the fact that many thermoset molding compounds fail to meet acceptance standards that previous lots identically processed had met, strongly suggest that moisture is a definite factor.

It was the objective of this study to determine if variations in moisture content of the three premolded thermoset compounds studied are responsible for the changes that resulted in a number of critical physical properties. From this data it will be determined what moisture content levels can be tolerated in thermoset molding compounds to optimize material use, and to determine what packaging and storage conditions are necessary, if any, to insure that only material of suitable quality will be used in making production parts.

*Partial cure.

ACTIVITY

Fabrication of Preforms

One lot each of E-9405 (Lot W-6924), diallyl phthalate (Lot L9806), and FM-1132 (Lot C-2446) was used to evaluate the effects of moisture in the uncured forms of a mineral filled epoxy, a long glass fiber reinforced diallyl phthalate, and a cotton filled phenolic compound, respectively. The initial preparations involved the pressing of these compounds into 376 preforms having 2 inch (5.08 cm) diameters to make the required amount of test specimens. The diallyl phthalate 52-20-30 and FM-1132 were room temperature pressed at 6800 psi (46.88 MN/m²) while the E-9405 was compacted using 9000 psi (62.05 MN/m²) at room temperature. The diallyl phthalate 52-20-30 and FM-1132 preforms were of varying thicknesses to achieve the right charge weight depending upon the type of test specimen made. This was necessary because these two materials have rather limited flow properties and it was necessary to compression mold the test specimens. All E-9405 preforms were able to be made the same size for convenience since the excellent flowing E-9405 could be transfer molded and any excess material would flash out.

Humidity Conditioning of Preforms

One fourth of the preforms pressed for a given material were exposed to each of the four humidity conditions at room temperature until equilibrium was established. This amounted to exposing 34 preforms of the E-9405 and 30 preforms of FM-1132 and 52-20-30 to each of the following conditions.

Zero Percent Relative Humidity

An air tight glove box equipped with dry nitrogen flow was used to approximate a zero percent relative humidity environment. Although there were trace amounts of moisture present (120 ppm) the humidity was maintained below 1 percent.

As Received

This pertains to the condition of the molding compound samples pressed from material taken directly from the manufacturer's shipping container. This material was double packaged in a fiber barrel and polyethylene bag and stored at 2° to 7°C until pressed.

50 ±5 Percent Relative Humidity

This environment was maintained at 25° ±3°C. All preforms were exposed to

this condition until saturated and no further weight gain was recorded.

90 ±5 Percent Relative Humidity

This condition was maintained in a Blue M circulating humidity chamber at $27^{\circ} \pm 3^{\circ}\text{C}$. All preforms were exposed to this condition until no further weight gain was recorded.

Immediately after the preforms were saturated at one of the previously mentioned conditions the preforms were individually heat sealed into vapor barrier bags which were conditioned with the preforms to insure that no moisture loss or gain would occur prior to molding.

Molding Test Specimens

Preforms of a given compound were molded the same regardless of conditioning; however, the poorly flowing compounds, FM-1132 and 52-20-30, were necessarily molded differently than the soft flowing E-9405. A summary of the molding conditions is given in Table 1.

Table 1. Molding Conditions at a Cure Temperature of $300^{\circ} \pm 5^{\circ}\text{F}$
($149^{\circ} \pm 2.78^{\circ}\text{C}$)

Material	Molding Technique	Cure Time (min)	Clamp Pressure*	
			Tons	MN
E-9405	Transfer molded	10	38	0.341
FM-1132	Compression molded	20	20	0.177
52-20-30	Compression molded	10	20	0.177
*6.89 MPa				

There are two exceptions to the data in Table 1. Dielectric and tensile specimens of E-9405 required 2230 and 1630 psi (15.37 and 11.24 MN/m²), respectively, at slightly reduced temperatures to fill the mold cavities of these specimens. Otherwise, all other specimens of E-9405 were molded at the conditions previously designated. Note that the clamp pressure for the soft flowing E-9405 was higher than that of the compression molded compounds. This pressure is not necessarily indicative of the pressure on the material, but was applied to keep the mold plates together when 1000 psi (6.89 MN/m²) was applied to the material.

After each preform was molded into the respective test specimen, the specimen was heat sealed in a vapor barrier bag within 5 minutes after its removal from the mold. This was done primarily to protect the molded test specimens from large humidity fluctuations that could occur before testing.

Test Procedures and Results

The bagged specimens were tested for the following properties: tensile, flexural, impact, and dielectric strength; moisture content, heat distortion and specific gravity, spiral flow, and post mold shrinkage. The impact, flexural and dielectric strength, heat distortion and specific gravity tests were performed on all the compounds in accordance with ASTM D-256, D-790, D-149, D-648-56, and D-792 respectively. Tensile testing of the E-9405 was done in accordance with ASTM D-638 while the hard flowing compounds were tested per D-651. Post mold shrinkage data was obtained from the percentage difference between a specimen dimension and the corresponding mold dimension both at room temperature. The specific dimension used was the length of a 0.5 by 0.5 by 5 inch (1.27 by 1.27 by 12.7 cm) test specimen. Spiral flow test procedures were divided into standard EMMI* testing (149°C and 6.89 MN/m²) of the soft flowing E-9405 and the Mesa** method (149°C and 20.2 MN/m²) used in testing the hard flowing FM-1132 and 52-20-30. Moisture content was determined with a Consolidated Electrodynamics Corporation analyzer, an instrument having an accuracy on the order of 0.001 percent. The instrument is equipped with an electrical circuit in which one segment of wire is coated with a compound having an extremely high affinity to moisture. A dry inert gas is passed over a small sample carrying the moisture across the coated wire. As moisture is absorbed by the coating, the current in the circuit changes and is measured and converted to percent moisture content.

Testing Fiberite E-9405

The test data shows a general decrease in the strength properties of uncured E-9405 for increasing moisture contents. An exception to this is the impact strength which fluctuated for the moisture contents investigated. The impact strength also behaved somewhat unexpectedly for the 52-20-30 and FM-1132 in that it increased with increasing moisture contents. Most of the strength properties of the E-9405 (Table 2 and in Figure 1) experienced a 15 to 30 percent decrease from the maximum value when the E-9405 was saturated at 90 percent relative humidity. Dielectric strength dropped 50 percent for a moisture pickup of 1.0 percent pbw while spiral flow length

*Epoxy Molding Materials Institute

**Archimedes Spiral Flow Test of Allied Chemical Corporation

Text continued on page 18.

Table 2. Physical Properties and Test Data for Fiberite E-9405

Physical Properties	Vendor Data	Specification Requirements	Dry Compound	As Received Compound	Compound Saturated at 50 Percent RH	Compound Saturated at 90 Percent RH
Tensile Strength, psi (MN/m ²)	10000 (68.95)	8000 (55.16)	9722 (67.03)	8770 (60.47)	8770 (60.47)	7127 (49.14)
Flexural Strength, psi (MN/m ²)	19000 (131.0)	12000 (82.74)	15350 (105.9)	14507 (100.0)	13732 (94.68)	12361 (85.23)
Impact Strength, ft-lbs/in. (Nm/m)	0.400 (21.4)	0.350 (18.7)	0.280 (15.0)	0.309 (16.5)	0.252 (13.4)	0.269 (14.4)
Moisture Content, Percent (pbw)	0.20	No requirement	0.09	0.32	0.37	1.10
Heat Distortion Temperature, °F (°C)	295 (146)	No requirement	353 (178.7)	331 (166.6)	323 (161.7)	298 (147.9)
Molding Shrinkage, Percent	0.50 to 0.70	No requirement	0.55	0.59	0.58	0.72
Specific Gravity	1.62 to 1.65	No requirement	1.62	1.63	1.63	1.63
Spiral Flow, Inches (cm)	24.5 (62.3)	23 + 3 - 2 (60 ±6)	18.3	24.5	29.8	30.8
Dielectric Strength S/T*, volts/mm	15700	13300	24100	24055	Bad test	14750
Dielectric Strength S/S**, volts/mm	13300	11810	22680	20710	19570	11020
*Short time **Step-by-step

Figure 1. Physical Properties Versus Percent Relative Humidity for Three Compounds

Figure 1 Continued. Physical Properties Versus Percent Relative Humidity for Three Compounds

nearly doubled for the same pickup. Post mold shrinkage was not significantly affected until high moisture contents were realized. It is interesting to note that specimens of E-9405 that were saturated at the 90 percent relative humidity condition (moisture percent 1.1) failed five of the six specification requirements while the as received material passed all but the impact strength requirements which was low regardless of moisture content.

Another point of interest is the differences between the vendor data and the experimental data. Some of these differences were evidently because the vendor was unaware of the effects that moisture in the raw material had upon some of the physical properties. Other differences occurred because the same test did not apply to the same test conditions. For example, the experimental data of this study indicates that the dielectric strength of Fiberite E-9405 degrades significantly with exposure to high humidities, yet the Fiberite vendor states that one of the outstanding features of E-9405 is its excellent retention of electrical properties under humid conditions. However, the data of this experiment refers to the changes in the physical properties of molded E-9405 when moisture is absorbed in the premolded form of this compound, whereas, the vendor is referring to the changes in dielectric strength that occur when cured parts are exposed to high humidities. The same is true regarding the dielectric properties of the other compounds investigated. Thus, the vendor data may not always be directly compared with the experimental data but should merely serve as a guideline so that the changes in the physical properties of the compounds with increasing moisture contents in the raw forms can be placed in the proper perspective.

The data in Table 2 indicates that care must be exercised in the storage of Fiberite E-9405 if this material is used in applications where tensile strength is critical. This is because incoming E-9405 having normal moisture content levels will most likely have a tensile strength that is marginal and additional moisture pickup in storage would result in a tensile strength below that specified in the applicable material specification. The need for special storage or packaging will be required for E-9405 where consistent flow properties are required since spiral flow is especially sensitive to moisture content.

From the data it appears that E-9405 that is known to have high moisture content would still be suitable for use in applications in which flexural, dielectric and impact strengths are needed. Although, the dielectric strength of parts molded for E-9405 is reduced greatly when wet molding compound is used, the dielectric strength of these parts will not fall below the minimum requirement unless the compound is saturated with moisture in humidities over 90 percent. Despite the fact that the impact strength was below the specification requirement, regardless of moisture content for the

lot of material evaluated, this property does not appear to degrade to any great extent with increasing moisture contents in the new material.

Testing Diallyl Phthalate 52-20-30

Of the compounds tested, raw compounds of phthalate 52-20-30, due to its low tendency towards moisture absorption, displayed the best retention of physical properties upon exposure to high humidities. This was because the filler in diallyl ortho-phthalate resin compound contains a long glass filler that has little or no affinity towards moisture absorption and has a relatively small surface area. Evidently, for low moisture absorption, both of these conditions must be met because the resin and mineral filler in E-9405, also have little affinity towards moisture yet E-9405 absorbs 1.10 pbw moisture when saturated at 90 percent relative humidity, by virtue of its large filler surface area, whereas 52-20-30 absorbs less than half as much.

Table 3 and Figure 1 show that the dry diallyl phthalate was below specification requirements in flexural strength, impact strength, and spiral flow; and over the high limit for molding shrinkage. It should be noted, however, that the same is true, with the exception of flexural strength, for all the conditions of the diallyl phthalate studied which indicates that the lot evaluated was below standard. For the purposes of this study the primary concern is the manner in which the physical properties of diallyl phthalate behave for various moisture contents in the raw material. If the physical properties of the dry compound are compared to those of the compound conditioned at 90 percent relative humidity it can be seen that most losses are on the order of 10 percent. This is considerably less than the percentage changes experienced with the E-9405 or FM-1132 for the same conditions.

Considering only the percent change, dielectric strength and spiral flow are not affected to any great extent. Despite the deterioration of the dielectric strength of diallyl phthalate 52-20-30 when saturated at 90 percent relative humidity this material still remains well above the material specification requirement.

Spiral flow inconsistencies caused by excessive moisture absorption had led to many processing problems associated with diallyl phthalate compounds. Parts molded from diallyl phthalate having an excessive spiral flow length have in some cases resulted in incomplete parts or parts that had surface defects due to excessive flash during molding. The presence of excessive moisture for long periods of time in diallyl phthalate compounds will cause the compound to B-stage. This invariably results in a decrease in spiral flow and if the degree of B-staging is sufficient the spiral flow will fall below the minimum specification tolerance. The use of such material has resulted in parts having knit lines or parts that are incomplete.

Table 3. Physical Properties and Test Data for Allied Chemical Diallyl Phthalate 52-20-30

Physical Properties	Vendor Data	Specification Requirements	Dry Compound	As Received Compound	Compound Saturated at 50 Percent RH	Compound Saturated at 90 Percent RH
Tensile Strength, psi (MN/m ²)	7000 (48.27)	6000 (41.37)	6850 (47.25)	6237 (47.00)	6472 (44.62)	6376 (43.96)
Flexural Strength, psi (MN/m ²)	18000 (124.0)	10000 (68.9)	9675 (66.70)	11500 (69.28)	11694 (80.63)	10145 (69.95)
Impact Strength, ft-lbs/in. (NM/m)	4 (214)	2.75 (146.8)	1.65 (88.0)	1.81 (96.6)	1.78 (95.0)	1.630 (87.0)
Moisture Content, percent	0.30*	0.50	0.07	0.19	0.21	0.45
Heat Distortion Temperature, °F (°C)	544 (284)	356 (180)	550 (288)	570 (300)	512 (266)	465 (241)
Molding Shrinkage, Percent	0.1 to 0.3	0.1	0.39	0.44	0.41	0.36
Specific Gravity	1.70	No requirement	1.64	1.64	1.64	1.64
Spiral Flow, Inches (cm)	No data	8 ±2 (20 ±5.1)	2.5 (6.35)	3.88 (9.80)	4.10 (10.3)	5.31 (13.5)
Dielectric Strength S/T***, volts/mm	No data	12795	26456	27520	28425	17480
Dielectric Strength S/St, volts/mm	15350 to 17320**	11810	24646	23980	25944	17125

*48 hours at 122°F (50°C)
 **48 hours immersed at 175°F (80°C)
 ***Short time
 †Step-by-step

Knit lines are areas where two or more fronts of material have joined together and have bonded improperly. Incomplete parts, which are simply due to incomplete fills, occur when the pressure exerted on the molten compound is insufficient to cause the material to fill the mold cavity.

Though moisture absorption has little effect upon most of the physical properties of diallyl phthalate it can have significant effects upon spiral flow. In applications involving complex flow patterns in which precise and consistent spiral flow is required moisture control will be needed. In applications that have less stringent flow requirements diallyl phthalate having moisture contents approaching saturation would still be usable in view of the fact that the compound exhibits excellent retention of physical properties.

Testing Fiberite FM-1132

FM-1132 is a two stage phenolic molding compound containing macerated cotton rag as a reinforcement. The cotton filler in FM-1132 exhibits an affinity towards moisture which causes the uncured compound to absorb large amounts of moisture when exposed to moderate humidities of more than 50 percent. Significant degradation of tensile, flexural, and especially dielectric strength values were experienced in specimens molded from uncured FM-1132 that had been allowed to saturate in a 50 percent relative humidity environment. Post molding shrinkage was also affected adversely in that specimens molded from the dry material shrank 0.39 percent while specimens molded from FM-1132 which had reached equilibrium in 50 percent relative humidity shrank 0.50 percent or 0.005 inches/per inch. Dielectric strength, the property most affected, dropped from 10355 volts/mm for the dry compound to only 3425 volts/mm. The saturation moisture content for uncured FM-1132 in a 50 percent relative humidity atmosphere at room temperature is 2.57 percent pbw, more than twice that of the other compounds investigated.

The data in Table 4 and Figure 1 indicate that when FM-1132 is allowed to saturate in environments of 90 percent relative humidity, severe reductions in the physical properties result. Tensile and flexural strength experience reductions on the order of 25 to 30 percent while dielectric strength becomes nearly nonexistent. It was found that the saturation level of FM-1132 in such environments is approximately 7.0 pbw moisture, a value high enough to cause the specific gravity to fall below the minimum specification requirement of 1.36.

The reduction of the strength properties in FM-1132 having high moisture levels is most likely the result of an insufficient degree of cure as evidenced by a 30°F drop in heat distortion temperature from the dry to extremely wet FM-1132. The decreases in the strength properties of the other compounds

Table 4. Physical Properties and Test Data for Fiberite FM-1132

Physical Properties	Vendor Data	Specification Requirements	Dry Compound	As Received Compound	Compound Saturated at 50 Percent RH	Compound Saturated at 90 Percent RH
Tensile Strength, psi (MN/m ²)	7000 (48.26)	5000 (34.47)	5560 (38.34)	5230 (36.06)	4950 (34.13)	3760 (25.93)
Flexural Strength, psi (MN/m ²)	12500 (86.19)	10000 (68.95)	9500 (65.50)	9200 (63.43)	8600 (59.30)	7400 (51.02)
Impact Strength, ft-lbs/in. (Nm/m)	60 (85.4)	1.20 (64.1)	1.44 (76.9)	1.72 (91.8)	1.66 (88.6)	2.24 (119.6)
Moisture Content, Percent	1.5	No requirement	0.13	1.58	2.57	7.19
Heat Distortion Temperature, °F (°C)	325 (163)	No requirement	330 (166)	325 (163)	333 (168)	299 (148)
Molding Shrinkage, Percent	0.3 to .5	No requirement	0.39	0.43	0.50	0.55
Specific Gravity	1.36 to 1.38	1.38 ±0.02	1.36	1.36	1.36	1.32
Spiral Flow, Inches (cm)	No data	No requirement	0.36 (0.91)	1.58 (4.01)	2.33 (5.92)	4.75 (12.06)
Dielectric Strength S/T*, volts/mm	11810	No requirement	10355	10670	3425	Failed for all voltages
Dielectric Strength S/S**, volts/mm	11023	No requirement	13583	7283	3070	Failed for all voltages

*Short time
**Step-by-step

studied E-9405 and diallyl phthalate 50-20-30 also were due to moisture inhibiting the cure. Of the compounds investigated, FM-1132 was most affected by humid environments in that the compound was significantly affected by exposures to even moderate humidities. The experimental data in Table 4 indicates that if parts molded from FM-1132 are to meet specification requirements strict moisture control procedures in the handling and storage of FM-1132 will definitely be needed.

ACCOMPLISHMENTS

The presence of moisture in the three thermoset molding compounds investigated detrimentally affects the physical properties of parts molded from these compounds. The experimental data provides information concerning which physical properties are affected by moisture and the extent that each property is affected for a given molding compound. Material that has passed lot acceptance testing and has been in cold storage for some time often picks up significant amounts of moisture which usually causes at least one physical property to be out of specification tolerance. The knowledge gained in this study could be used to predict what physical properties would deviate for a given moisture content of the compound and thus determine a suitable application for the compound.

This study has also revealed a critical need for moisture control for incoming E-9405, FM-1132, and possibly diallyl phthalate 52-20-30. As a result, changes requiring improved packaging and handling techniques will be incorporated into the material specifications of the compounds studied. The requirement will provide that the manufacturer double bag and heat seal the material in polyethylene bags and will also require that after each use the bags be resealed.

FUTURE WORK

Future work will consist of the determination of how the flow properties vary with time for various moisture contents in E-9405, FM-1132 and 52-20-30. Since moisture is known to affect the degree of cure in the compounds studied it is also expected to affect cure times.

In addition, future work will involve an evaluation to determine if the presence of moisture in thermoset compounds is responsible for a number of surface defects that occur in molded parts.