

STATUS REPORT

EXPLORATORY VINYL RESIN SCREENING EVALUATION
OF QXOM-7 RESIN (BLEND 3)

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Date: June 1, 1966

Project No.: 399G10

File No.: 5757

SUMMARY Comparative evaluation of the QXOM-7 resin (Blend 3), which contains approximately 1.0 per cent ethylene and was prepared in production facilities at Texas City autoclaves with a QYTQ-7 resin of comparable molecular weight reveals the following:

Processibility

Initial Fluxing

The copolymer fluxes more readily than the QYTQ-7 resin in rigid and semi-rigid formulations. No significant differences in fluxing behavior were observed at higher plasticizer concentrations.

Apparent Melt Viscosity

The torque requirements for compounds containing the ethylene copolymer were less through the entire plasticizer concentration range (10 thru 60 phr FLEXOL DOP). Surprisingly, no difference between the copolymer and homopolymer was observed in the rigid formulation.

Physical Properties

The inclusion of ethylene results in reduced tensile strength and room temperature stiffness as well as increased elongation in both rigid and plasticized compounds. The vinyl chloride-ethylene copolymer requires approximately 5 phr less plasticizer than the homopolymer to achieve the same level of Shore hardness.

Thus, the major advantage of the "O" resin are faster fluxing and lower melt viscosity in flexible compounds, internally plasticized nature which should permit use of 3-5 phr less plasticizer, and faster dry-blending character.

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INTRODUCTION This report discusses the physical properties and melt characteristics of a vinyl chloride-ethylene copolymer (QXOM-7 Blend 3) in plasticized and rigid formulations. The properties are compared to QYTQ-7 (Blend 130), a homopolymer of comparable inherent viscosity.

The resins evaluated were prepared in Texas City production autoclaves using Process-7. The copolymer contains approximately 1 per cent ethylene with an inherent viscosity of 0.98. The inherent viscosity of the homopolymer is 1.0.

DISCUSSION

Plasticized Compounds

Melt Characteristics

The evaluation of the processing characteristics and the physical properties of the plasticized compounds of the vinyl chloride-ethylene copolymer (QXOM-7) and a poly(vinyl chloride) homopolymer QYTQ-7 were studied at 10 through 50 phr plasticizer level (FLEXOL plasticizer DOP). The formulations also contained 3.0 phr Mark M, a liquid barium-cadmium phenate stabilizer. These data are listed in Table III.

The melt characteristics, shown graphically in Figures 1 through 4 may be summarized as follows.

1. The compounds containing the vinyl chloride-ethylene copolymer fluxes more readily at the 10 and 20 phr plasticizer level than the comparative homopolymer. No difference in time to initial fluxing could be detected in the 30 through 50 phr plasticizer level. However, the method used for this evaluation does not allow an establishment of fluxing times of less than one minute due to sample loading time (approximately one half minute) and the time for the plasti-corder to establish equilibrium (approximately one half minute after sample loading).

2. The "O" resins exhibited slightly lower apparent equilibrium melt viscosity than the homopolymer counterpart through the entire plasticizer concentration range indicating that, in the same formulation, compounds containing the vinyl chloride-ethylene copolymer would process with lower power requirements.

Thus, compounds containing vinyl chloride-ethylene copolymers require less dwell time in intensive mixers at the lower plasticizer concentration range and would process with less power requirements at all plasticizer levels.

Physical Properties of Plasticized Compounds

The effect of the inclusion of ethylene on several physical properties may be summarized as follows:

1. Tensile strength is lowered, the rate of lowering of tensile strength with increasing plasticizer concentration is approximately the same for both homopolymer and copolymer. This data is shown in Table III, Figure 5.

2. The tensile elongation, as was expected, is increased in the compound containing the ethylene copolymer. Again, the rate of increase with increasing plasticizer concentration is approximately the same for both the homopolymer and copolymer (see Figure 6).

3. The brittleness temperature, shown graphically in Figure 7, is decreased an average of 3°C over the plasticized concentration range by the inclusion of 1.0 per cent ethylene in the polymer.

4. Shore hardness, often used as a measure of plasticizer efficiency, is shown in Figure 8 to be lower in the QXOM-7 sample throughout the plasticizer concentration range. The data indicates that to produce a simple flexible compound with a Shore hardness reading of 85 would require 47 phr FLEXOL DOP for the QXOM-7 resin and 52 phr FLEXOL DOP for the QYTQ-7 resin. Thus, it would require approximately 5 phr less plasticizer for the ethylene copolymer than the homopolymer. Previous data supports this observation (1).

5. Room temperature stiffness is also lowered as expected. Significant differences may be observed, (see Figure 9) in the semi-rigid compounds (10 and 20 phr plasticizer). The effect of the presence of ethylene in the resin decreases with increasing amounts of plasticizers.

Rigid Compounds

Although it is realized that the inherent viscosity of the resins evaluated restrict their use chiefly to plasticized formulations, this evaluation in a simple rigid compound was conducted to provide additional information on the influence of ethylene in copolymers. The formulation used contained 3.0 phr Mark WS, a powdered barium-cadmium stabilizer available from Argus Chemical Company. The data are shown in Table II.

Physical Properties - Rigid Formulation

1. The tensile strength as was expected, is approximately 17% lower in the compound containing the ethylene copolymer than the one containing the homopolymer.

(1) Brezinski, J. J., Fields, J. W., Exploratory Vinyl Resin Screening: Evaluation of QXAM-11 Resin Samples QEX-1197 and QEX-1198, Research and Development Department, Status Report, File No. 4910, November 22, 1965.

2. The QXOM-7 resin is approximately 44% higher in tensile elongation than the QYTQ-7 resin.

3. Room temperature stiffness is lowered approximately 11 per cent by the inclusion of ethylene.

4. The addition of approximately 1% ethylene results in an approximate 17 per cent drop in heat distortion temperature at this molecular weight (64°C vs 77°C for QYTQ-7).

Melt Characteristics - Rigid Formulation

The study of the melt characteristics in a rigid formulation indicates a faster fluxing time (approximately 45 per cent) for the ethylene copolymer when compared to the QYTQ-7 homopolymer. No difference could be detected between the two resins in apparent melt viscosity.

CONCLUSIONS

Flexible Applications

The inclusion of approximately 1 per cent ethylene at the 1.0 inherent viscosity range results in the following advantages over the homopolymer counterpart.

1. Faster fluxing time at the lower plasticizer concentration levels which should decrease residence time in intensive mixing devices.

2. Lower apparent melt viscosity which will decrease power requirement for processing and fabricating equipment.

3. An approximate 3°C lower brittleness temperature over the plasticizer concentration range.

4. An approximate 5 phr better plasticizer efficiency rating.

5. Higher tensile elongation. This property indicates that film processed from ethylene containing copolymers would elongate a greater distance before film rupture or breakage occurs.

The compromised physical properties are:

1. Lower tensile strength. The differences observed in this evaluation are not considered to be significant enough to exclude consideration of resins of this composition and molecular weight from flexible end product use.

Miscellaneous Property Evaluation

Dry Blend Performance

The copolymer resin (QXOM-7) dried approximately 60 per cent faster than the homopolymer (QYTQ-7) using the standard formulation and procedure. This faster dry blending characteristic is considered an advantage for the "O" resin.

Hard Resin Particles

The hard resin particles, as observed in a 10 mil flexible dyed film using a special test procedure presently under development are not considered to be sufficient to effect this resin for end product use; the resin contains a relatively low level of hard resin particles.

Particle Geometry

The sieve analysis indicates a longer particle size for the copolymer and a desirable low fines content.

Plasticizer Sorption

The two resins tested are essentially equivalent in plasticizer sorption.

Initial Color Mill Test (ICMT)

The initial color of both resins are within the experimental error of the test as being the same. The QYTQ-7 sample exhibited better intermediate stability (color after 20 minutes processing) while the QXOM-7 exhibited better ultimate stability (less color after 30 minutes processing).

NOTEBOOK REFERENCE: 20BAP-7237

PERIOD: April 15 to May 4, 1966

Attachments

3 Tables

9 Figures



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TABLE I

EVALUATION OF VINYL CHLORIDE-ETHYLENE COPOLYMERS
CHEMICAL AND GENERAL PROPERTIES

	QXOM-7 Blend 3	QYTQ-7 Blend 130
Inherent Viscosity; WC-31-B-4	0.98	1.00
Ethylene Comonomer, %	0.9	-
Heating Loss, %; WC-160-H	99.8	99.8
Apparent Density, lbs/ft ³ ; WC-2-I	29	31
ASTM Flow Time, seconds/400cc; ASTM D 1895-61T	3.0	3.7
ASTM Plasticizer Sorption, %; ASTM D 1755-60T	97	95
Sieve Analysis; WC-7-F		
% Thru 40-mesh	100	100
60-mesh	98	100
80-mesh	46	97
100-mesh	18	80
140-mesh	3	8
200-mesh	2	5
Median particle size, microns	181	126
Dry Blend Test (Plasti-Corder)		
Time to Dryness, minutes	3.3	8.0
Temperature at Dryness, .C	79	84
Hard Resin Particles, 10 Mil Film; (1)		
	110	120
Initial Color Mill Test (ICMT); WC-163-C	121	122
20 Minute Holding Power, %	77	86
30 Minute Holding Power, %	72	62

(1) Test under development.

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TABLE II

EVALUATION OF VINYL CHLORIDE-ETHYLENE COPOLYMERS
PHYSICAL PROPERTIES (RIGID FORMULATIONS)*

	<u>QXOM-7</u> <u>Blend 3</u>	<u>QYTQ-7</u> <u>Blend 130</u>
Tensile Strength, psi; WC-66-K-1 (1)	6880	8270
Tensile Elongation, % (1)	58	102
Yield Strength, psi (1)	7000	7400
Room Temperature Stiffness, psi (1)	361×10^3	408×10^3
Izod Impact Strength, lbs/inch/notched; ASTM D 256 (1)	1.00	0.97
Heat Distortion Temperature, °C; ASTM D 648-56 (1)	77	64
Melt Characteristics (Plasti-Corder)		
Apparent Melt Viscosity,		
Torque, meter grams second ⁻¹	2670	2600
Temperature, °C	186	186
Initial Fluxing Time, seconds	330†	600
Initial Fluxing Temperature, °C	174	179

(1) Formulation

Resin 100 parts
Mark WS 3.0 phr

Procedure

6" x 13" Roll Mill
Mill Temperature 170°C
Mill Time 5 minutes
Press Platen
 Temperature 175°C
Press Time 5 minutes

Brabender Plasti-Corder

Processing Conditions and Formulation

Resin 100 parts
Mark WS* 3.0 phr
Jacket Temperature 165°C
Rotor Speed 40 rpm

* Mark WS = Barium-cadmium powdered stabilizer available from Argus Chemical Company.

TABLE III

EVALUATION OF VINYL CHLORIDE-ETHYLENE COPOLYMERS
PHYSICAL PROPERTIES (FLEXIBLE FORMULATIONS)*

	10 phr		20 phr		30 phr		40 phr		50 phr		50 phr	
	<u>QXOM-7</u>	<u>QYTQ-7</u>	<u>QXOM-7</u>	<u>QYTQ-7</u>	<u>QXOM-7</u>	<u>QYTQ-7</u>	<u>QXOM-7</u>	<u>QYTQ-7</u>	<u>QXOM-7</u>	<u>QYTQ-7</u>	<u>QXOM-7</u>	<u>QYTQ-7</u>
Tensile Strength, psi; WC-66-K-1	6640	6540	5130	5450	3730	4000	3180	3310	2640	2820	2280	2390
Tensile Elongation, %	159	148	229	217	275	255	312	302	354	340	404	385
Room Temperature Stiffness, °C	350×10^3	375×10^3	80×10^3	137×10^3	4.5×10^3	8.6×10^3	1.7×10^3	1.9×10^3	0.7×10^3	0.7×10^3	0.4×10^3	0.6×10^3
Brittleness Temperature, °C	+42	+42	+12	+18	-6	-2	-18	-16	-26	-24	-34	-32
Melt Characteristics (Plasti-Corder)												
Apparent Melt Viscosity,												
Torque, meter grams second ⁻¹	2280	2320	1860	1980	1570	1680	1280	1370	1020	1140	-	-
Temperature, °C	171	175	170	172	161	167	155	158	152	154	-	-
Initial Fluxing Time												
Time, seconds	315	1080	110	210	(1)	(1)	(1)	(1)	(1)	(1)	-	-
Temperature, °C	162	164	155	155	(1)	(1)	(1)	(1)	(1)	(1)	-	-
Shore Hardness A-2	-	-	-	-	97	98	90	91	83	86	78	80

Brabender Plasti-Corder Conditions

Jacket Temperature 155°C

Rotor Speed 40 rpm

* Formulations, Physical Properties and Melt Characteristics

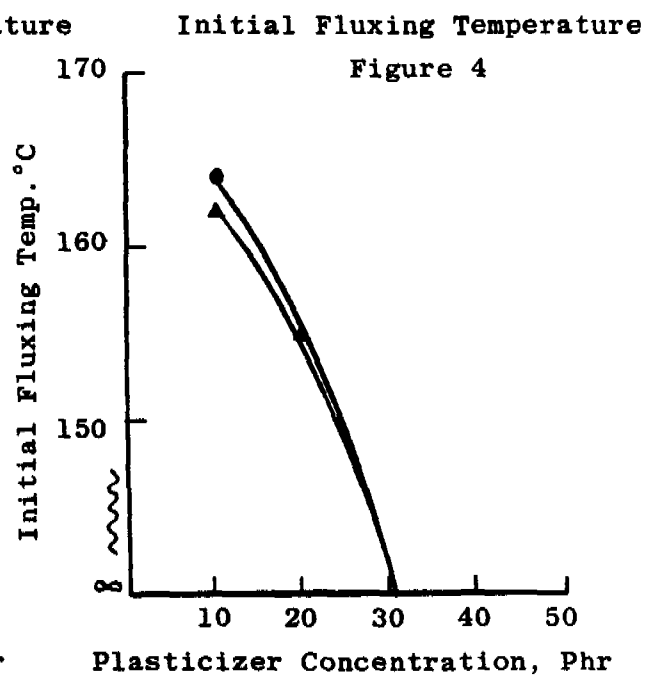
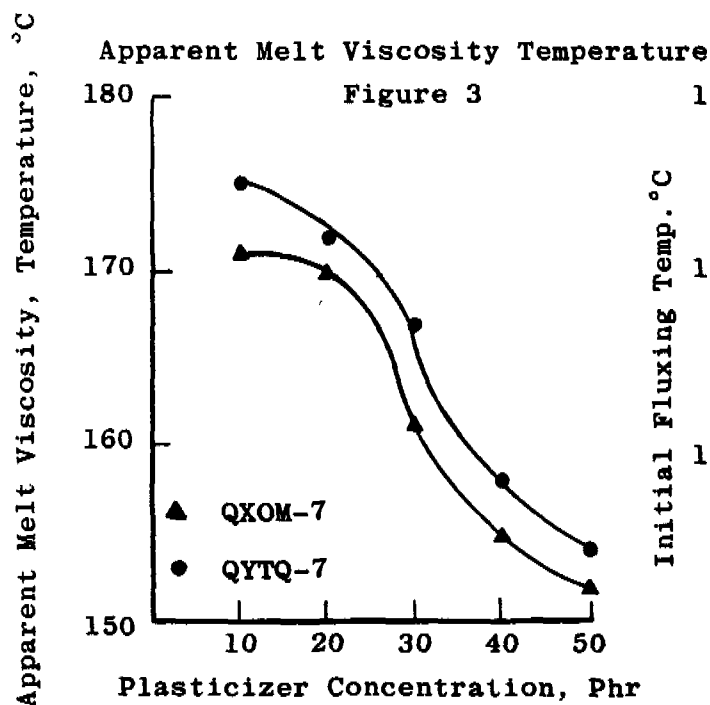
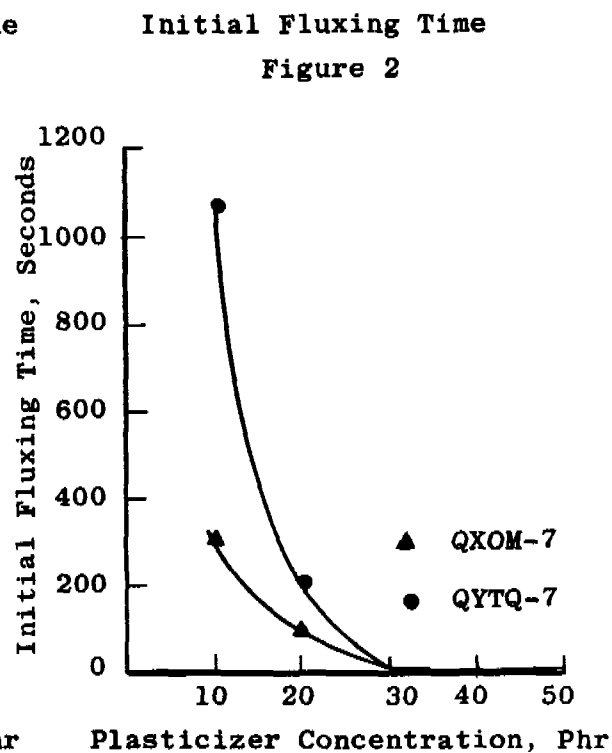
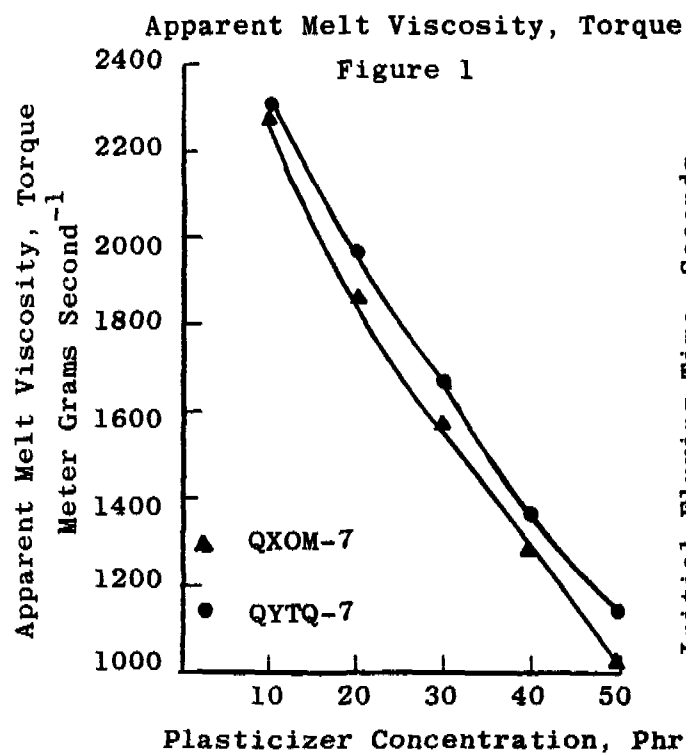
Resin 100 parts
FLEXOL DOP phr as shown
Mark M (2) 3.0 phr

(1) Immediate fluxing, less than 1 minute including loading time.

(2) Mark M = Liquid barium-cadmium stabilizer available from Argus Chemical Company.

RESEARCH AND DEVELOPMENT

RELATIVE PROCESSIBILITY, FLEXIBLE FORMULATIONS

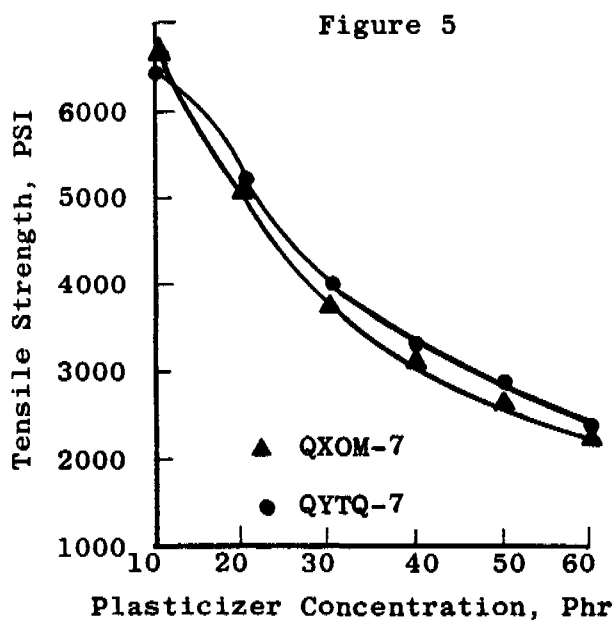


RESEARCH AND DEVELOPMENT

PHYSICAL PROPERTIES FLEXIBLE FORMULATIONS

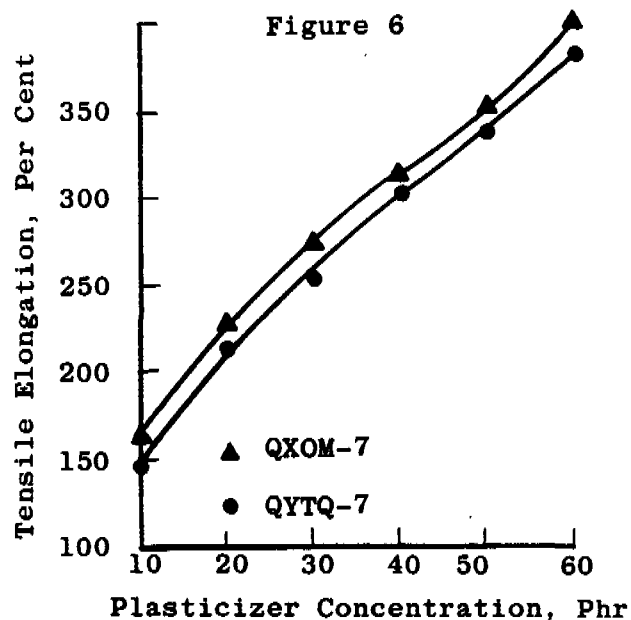
Tensile Strength

Figure 5



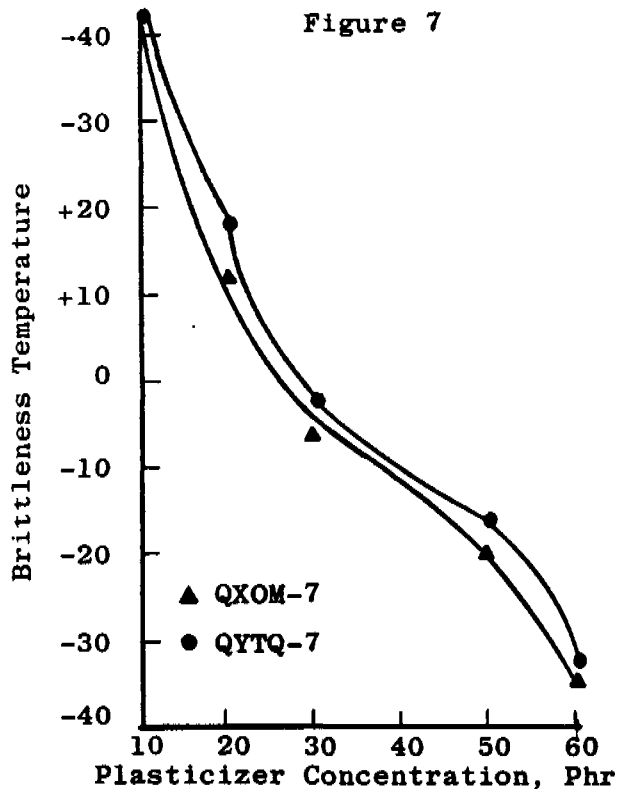
Tensile Elongation

Figure 6



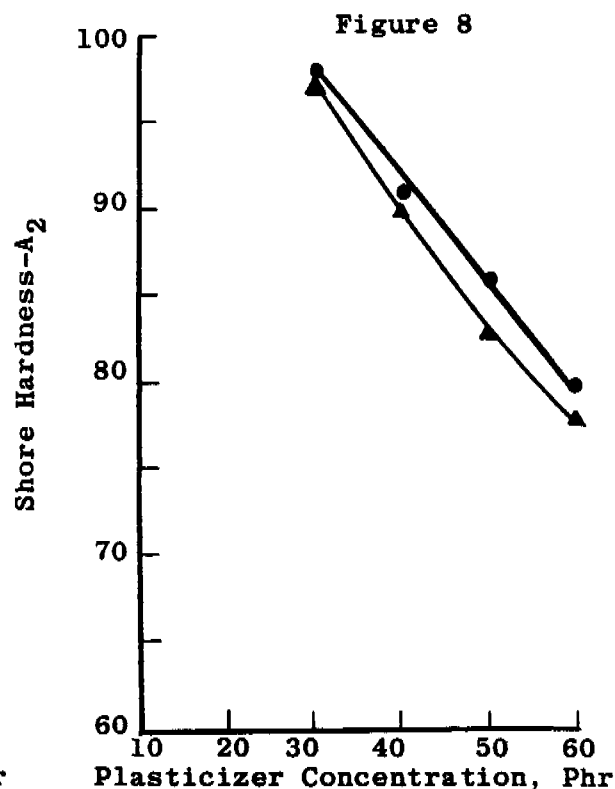
Brittleness Temperature

Figure 7



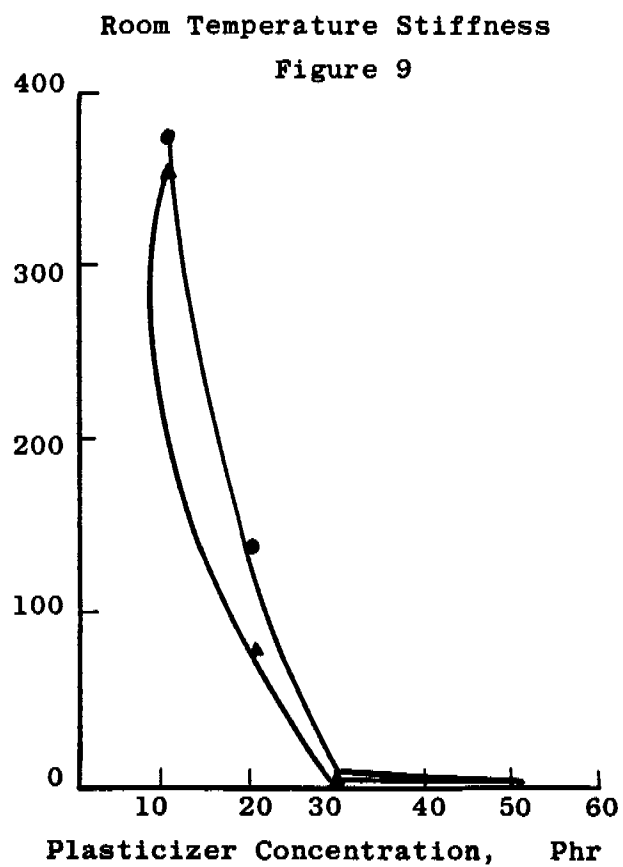
Shore Hardness

Figure 8



RESEARCH AND DEVELOPMENT

PHYSICAL PROPERTIES, FLEXIBLE FORMULATIONS



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