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STATUS REPORT

VINYLCHLORIDE RESIN

CONTOUR EXTRUSION OF FLEXIBLE VINYL PRODUCTS CONTAINING BAKELITE® RESIN QSOM-7

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SUMMARY

The "O" resins (copolymers of vinylchloride/ethylene) have been developed to the point where field application trials are being made on a selected basis. BAKELITE® resin QSOM-7, Blend 6, has been evaluated in the dryblend and contour extrusion process at the New England Plastics Corporation.

The operation did not successfully produce commercially acceptable extruded products with QSOM-7 resin. However, the run indicated that the application of vinylchloride/ethylene copolymers must be separated into two categories:

1. Unique applications which can take advantage of plasticizer reduction and adjustment of processing conditions and product properties accordingly, as associated with QSOM-7 type copolymers.

2. The replacement of conventional dryblend type extrusion resins will require a grade of "O" resin which will not necessarily afford a plasticizer savings, but will process more readily under the given set of conditions normally employed by the vinyl processor with homopolymer poly (vinylchloride).

Union Carbide Corporation - Chemicals & Plastics
Research and Development Department
Tarrytown, New York

May 18, 1967

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INTRODUCTION

On April 26 and 27 the writer and Mr. S. Krumm supervised plant trials employing BAKELITE® Resin QSOM-7, Blend 6 in the contour extrusion process at New England Plastics Corporation, in Woburn, Massachusetts The investigation included the preparation and extrusion of dryblend compositions in typical vinyl formulations for the manufacture of unfilled (40 PHR plasticizer), filled (80 PHR plasticizer), and rigid vinyl products.

New England Plastics Corporation produces extruded thermoplastic shapes. The plant contains eight Davis Standard extrusion lines (two for extruded sheet stock) arranged spaciously in neat order, but the operation is not fully automated with respect to material feed. The weigh-up facility contains two conventional type mixers (Littletown 120 rpm, and Ribbon 60 rpm); both are about 500 pound capacity, jacketed and are used for preparation of plasticized vinyl dryblend. One intensive mixer (Diosna, 120 pound capacity) is used for preparation of rigid powderblend; it is sometimes employed for reprocessing of an inferior batch of plasticized dryblend, which may have caused extruder feed problems or hard particles in the extrudate. The weigh-up operation is essentially manual, except for resin and plasticizer addition to the Littletown and Ribbon blenders.

All of the vinyl products are prepared from the dryblend and powder blend technique. Thus, two resin tanks are used (Escambia 2225 and Blacar 250-1); Blacar 12-R is purchased in bags. Four tanks hold plasticizer (one with five compartments). Plasticizers are supplied by a local reseller and by USS Chemicals. The following plasticizers are purchased:

Blended DOP/Drapex 4.4:3/1
Blended DOP/DOA/Drapex 4.4:68/11/21
Drapex 4.4 in drums.

The plant trials were coordinated with Messrs. John Gibbons, Vice President, and Bob Alexander, Plant Engineer. All of the personnel were extremely cooperative and helpful in the conduct of the investigation.

DISCUSSION

Formulations:

Base formulations are shown in Table I. The plasticizer level was adjusted downward by 15 per cent in the unfilled chair tubing formulation and by 20 per cent in the softer weatherstrip (#2 channel) formulation when QSOM-7 was substituted for

Escambia 2225. In the rigid (RV-115) formulation, no adjustment was made other than to interchange QSOM-7 for Blacar 12-R.

Blending Operation:

Blending cycles are shown in Table II. It was difficult to determine differences in dryblending characteristics as a function of QSOM-7 vs. Escambia 2225 because the thermocouple was not connected in the ribbon blender due to some previous maintenance service on the blender, not related to the plant trials. However, periodic readings by means of a needle Pyrometer (made by interrupting the continuous operation of the blender) indicated that the heat transfer rate and rate of drying was similar in QSOM-7 and Escambia 2225. The degree of dryness of QSOM-7 appeared inferior to Escambia 2225 when dropped from the ribbon blender in the chair tubing compound, as determined hand tests of caking and dry flow. However, the operators confidently appraised the QSOM-7 dryblends as being at least as good as those based on Blacar 250-1, which is used interchangeably with Escambia 2225. The weigh-up men prefer Escambia 2225 over Blacar 250-1 because of less dusting (due to "pearls").

Normal operating procedure allows the drums of dryblend to stand for 8 to 16 hours prior to extrusion. Immediate use causes feed problems, especially in small throated extruders and when hopper vibrators are not working (although Escambia 2225 is notably less severe in this respect).

In spite of this advice, we tried to feed #87 chair tubing with QSOM-7 into Extruder No. 1 immediately after dryblending. Extruder No. 1 has a small throat, and a malfunctioning hopper vibrator; the QSOM-7 did not feed that day. The preblend was allowed to stand overnight; the next day, the QSOM-7 dryblend was fed into the extruder (machine no. 5) with no difficulty and no indication of caking.

The rigid powderblends were prepared in the Diosna mixer. The QSOM-7 blend showed a significantly lower batch temperature at the outset of the cycle due to extended down time to accomplish thorough clean-up following the Blacar 12-R premix. (This was done with both rigid and channel #2 mixes in order to remove the suspicion that hard particles, previously observed in QSOM-7 extrudates, were a result of contamination in the weigh-up operation). The power requirements (amps) were 50 to 60 per cent greater at a given batch temperature for QSOM-7 in place of Balcar 12-R. No apparent differences existed between the QSOM-7 and Blacar 12-R powderblends.

Extrusion Characteristics

Table III shows the conditions employed in the extrusion process for the three products tested. The unsuccessful "first day" trial of #87 chair tubing with QSOM-7 made on machine No. 1 is not shown in Table III. This abortive attempt was made at barrel temperatures approximately 15 degrees F. below normal operating conditions. The resultant extrudate was poor in gloss and had an exceptionally high level of hard particles.

The flexible vinyl products based on QSOM-7 (chair tubing and channel) were found to extrude best at similar conditions to the control compounds based on

Escambia 2225. However, the flexible products employing "O" resin showed a high level of hard particles and undesirable low gloss level. The gloss was improved somewhat by raising the die temperature as much as 20°F over normal conditions, however, the extrudate still did not reach the desired level of gloss.

At one point in the extrusion of the chair tubing, the screw was adjusted to neutral which resulted in a 25 per cent increase in output rate, but also resulted in inferior quality of the extrudate (gloss and hard particle level).

In the rigid extrusion, the "O" resin showed a significant drop off in power requirements (amps). However, it was necessary to increase extrusion temperatures 10 to 20 degrees F. in order to improve gloss. The elevated temperature caused a slight increase in amperage, but desired gloss was not obtained and the output rate was about 17 per cent below that of the control, Blacar 12-R. The hard particle problem was as prevalent in the rigid extrusion as in the flexible products.

It is recognized that QSOM-7 is not considered the optimum selection to replace Blacar 12-R in the extrusion of rigid products. However, the rigid extrusion with QSOM was close enough to being successful to allow one to predict that a lower molecular weight "O" type resin would provide optimum performance in rigid extrusions.

Thermal degradation was a consistent problem with all of the extrusion trials employing QSOM-7. Four different extruders were employed in all:

Extruder	Product
No. 1	Chair Tubing - Green
No. 5	Chair Tubing - Green
No. 2	Channel - Grey
No. 4	Rigid - White

All four extrusion lines required breakdown within two to eight hours after trials with the QSOM-7 mixes, and all four extruders revealed excessively degraded material (black) in the head and die sections. It appeared that the thermal degradation resulted from stagnation of melt flow in the head and die areas. It is possible that inferior thermal stability is inherent with the "O" resins or with blend 6 of QSOM-7. However, the colors of the flexible extrudates appeared to be right on target and showed no indication of inferior thermal stability. The rigid vinyl extrudate (white) showed two definite streaks indicating localized thermal degradation. These streaks were indicative of "hang-up" within the die, because close examination revealed their presence in the control product (Blacor 12-R). However, they were barely noticeable in the control product, but very obvious in the QSOM-7 product.

Miscellaneous Remarks:

A sample of QSOM-7, blend 6, was taken from an unopened bag at the New England Plastics Corporation and evaluated for fisheye level. Mr. J. Hockersmith has reported the following fisheye ratings:

QSOM-7, Blend 6

	Original*	Return From New England Plastics Corporation
17.5 mil Respro	nil	nil
10. mil Respro Normal Count	40	83
Clear Hard Particle	1	--
High Intensity Light (10 mil)	--	600

* Shown on Blend Data Sheet, 9/15/66

The significant difference in hard particle count indicates the liklihood of contamination by a foreign resin after the bin sample was taken.

Physical properties of the extruded products are being measured and will be reported by Mr. S. Krumm.

CONCLUSIONS

The plant trials employing QSOM-7, Blend 6 in the extrusion of flexible and rigid shapes showed the following:

1. QSOM-7 is not suggested for use in rigid extrusions, but the plant run indicated that a slightly lower molecular weight "O" resin may be useful, and that a lubricant adjustment may be required to optimize processability.

2. The dryblending characteristics of QSOM-7, Blend 6 are inferior to that of Escambia 2225, but were judged to be similar to that of Blacar 250-1 and quite acceptable for conventional dryblend applications.

3. QSOM-7 cannot be substituted directly for medium molecular weight poly (vinylchloride) homopolymers in contour extrusion because the required adjustment of plasticizer causes a significant difference in required processing conditions. For instance, it was impossible to accomplish the desired gloss without exceeding optimum thermal conditions for extrusion of the polymer melt.

4. The heat stability characteristics of QSOM-7, Blend 6 are subject to question. It appears that the thermal degradation difficulties encountered were primarily a result of processing above optimum temperatures for the resin, which was a result of attempts to improve gloss. However, the possibility of inherent thermal stability shortcomings should not be overlooked.

5. The QSOM-7, Blend 6 employed in the plant trials had a significant level of hard particles, which could not be reduced by variations in processing conditions.

The following recommendations are made:

1. QSOM-7, Blend 6 type resin (inherent viscosity = 0.99, ethylene = 1.33%) should be considered a unique resin for applications where plasticizer reduction is exceptionally desirable and unique processing conditions are attainable to produce the desired product.

2. A vinylchloride/ethylene copolymer of a slightly higher molecular weight with an ethylene level of less than one per cent should be investigated as a dry blendable replacement for commonly used medium molecular weight homopolymer PVC resins. It is expected that a resin of this nature will not require significant plasticizer adjustment, but will provide added processing parameters when substituted for homopolymers in contour extrusion of flexible vinyls.

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L. G. Krauskopf

TABLE I

Base Formulations

#87 Chair Tubing, Green	Parts by Weight Standard	"O"
Escambia 2225	100	---
QSOM-7, Blend 6	---	100
Plasticizer*	40	34
Vanstay HTA (Ba/Cd Stabilizer)	2.5	2.5
Vanstay RR (Ba/Cd Stabilizer)	0.8	0.8

* Plasticizer blend = DOP/DOA/Drapex 4.4:68/11/21

#2 Channeling, Grey	Standard	"O"
Escambia 2225	100	---
QSOM-7, Blend 6	---	100
Plasticizer*	80	64
Atomite	33.3	33.3
Vanstay HTA (Ba/Cd Stabilizer)	1.0	1.0

* Plasticizer Blend = DOP/Drapex 4.4:3/1

RV-115 Rigid, White	
Blacar 12-R*	100
Chlorinated Polyethylene	12.5
Acryloid K-120N	3.0
O-292 (Tin Stabilizer)	3.0
Drapex 4.4	3.0
Stearic Acid	0.75
Mg Stearate	0.25
Unitane 450	8.0

* Replaced part for part with QSOM-7, Blend 6

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TABLE II

Blending Cycles

#87 Chair Tubing:

Equipment - Jacketed ribbon blender, blade speed 60 rpm.
Batch Size –Standard 428 pounds; QSOM-7 = 410 pounds

Cycle - Premix dies; then spray in liquids (liquids consist of plasticizer blend and Vanstay RR, which have been premixed and preheated to approximately 100°F prior to addition to batch); apply steam (15 psig) to jacket. Continue mixing with heat until batch reaches 210-220°F. (normally requires 30-45 minutes); apply cooling water and continue mixing until temperature drops to 90-100°F (normally requires 30 minutes); drop into open head drums.

Usage - Move drums to extrusion area and hand feed extruder hoppers.

#2 Channel

Batch size - Standard = 642 pounds; QSOM-7 = 594 pounds

Equipment cycle and usage - the same as that employed for #87 chair tubing.

RV-115 Rigid

Equipment - Diosna Mixer (intensive type jacket neutral)
Batch Size- 120 pounds, standard and QSOM-7 batch.

Cycle - Mix all solids, except lubricants, at 1600 rpm. When temperature reaches 150°F, add liquids (Drapex 4.4) run at 1600 rpm until temperature reaches 175°F. Add lubricant and drop batch into cooling chamber. Cool five minutes and drop into Gaylord container.

Cycle Time (Minutes)	Blacar R-12 Amps	Blacar R-12 °F	QSOM-7 Amps	QSOM-7 °F
0.25	75	170	85	130
0.50	75	170	---	---
	add liquids	---------	---	---
0.75	100	170	90	140
1.00	90	180	95	150
	---	---	add liquids	---------
1.5	85	180	105	150
2.0	83	180	125	170
2.5	85	190	135	190
3.0	95	200	135	200
3.5	100	210	155	200
-------------------	----lubricant	added----	-------------	-------------
4.0	130	230	> 155	230
	drop to cooling chamber			

TABLE III

Extrusion Conditions

#87 Chair Tubing - Green (1/2" ID, .020" Wall)

No. 5 Extruder - Davis Standard, 3.5"; L/D:20/1: Compression ratio 4:1

	Escambia	QSOM-7
Zone 4 (feed)	315 °F	345 °F
Zone 3	320	335
Zone 2	340	320
Zone 1 (head)	350	325
Die	325-345	330
Screw	one full turn cold water	
Speed, rpm	77	77
Speed setting	61	61
Amps	60-70	60-70
Output, fpm	175	175

#2 Channel - Grey .050" wall thickness

No. 2 Extruder - Davis Standard; 3.25 inch; L/D:16/1; Compression ratio 2:1

	Escambia	QSOM-7
Zone 3 (feed)	345 °F	345 °F
Zone 2	355	355
Zone 1 (head)	350	350
Die	355	355-375
Stock Temperature, °F	350	350
Screw	one full turn cold water	
Speed, rpm	106	106
Amps	55	55
Output, fpm	184	184

RV-115 Rigid - White .040" Wall Thickness

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TABLE III

(continued)

No. 4 Extruder - Davis Standard; 3.5 inch; L/D:20/1; compression ratio 2:1

	Blacar 12-R	QSOM-7
Zone 1 (feed)	320°F	310°F
Zone 2	320	330
Zone 3	320	340
Zone 4 (head)	330	340
Die	320	330-340
Screw	no water cooling provision	
Speed, rpm	7	7
Speed selling	19	19
Amps	125	100-110
Output, fpm	12	10

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