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THERMAL STABILITY OF POWDERED GEON-HYCAR NITRILE POLYBLENDS V:

UTILITY OF DYNAMIC THERMAL STABILITY TESTS

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by

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ABSTRACT

This study was initiated to develop a quantitative method to predict the processing safety and rate of decomposition of flexible Geon compounds that are modified with powdered Hy-car nitrile elastomers. The standard Brabender Dynamic Thermal Stability test was shown to provide the necessary quantitative information. Two indicators of processing safety were developed from this data. Two indicators of degradation rate were also obtained by analyzing the behavior of the compounds during the decomposition phase of the test. This test was then used as a tool to study the effects of changes in both the compound recipe and nitrile elastomer on the thermal stability of the blends. This joint study by the Geon and Hy-car groups clearly demonstrated that processing safety is inversely proportional to the viscosity of the nitrile elastomer. The rate of decomposition was shown to be effected by the PVC stabilizers in the compound and the stabilization system used in the powdered rubber. These studies resulted in the development of a powdered compound of Geon and Hy-car for a customer extrusion application. They also enabled us to accurately define the type of "ideal" powdered nitrile elastomers that are required to optimize the processability of these thermoplastic blends.

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Major Objective

To improve the thermal stability of powdered Geon/Hycar polyblends.

Minor Objectives

To determine the utility of dynamic thermal stability tests as an indicator of compound processability.

To use dynamic thermal stability tests to study the effects of several nitrile rubber variables on the processability of polyblends.

Introduction

This report is one of the results of a joint marketing and development effort of both the Geon and Hycar groups. The specific goal was to develop a powder compound of Geon resin and powdered Hycar nitrile rubber for use in a flexible thermoplastic application. The customer, Cooley Products, requested that the compound contain thirty weight percent nitrile rubber. The first compound was based on Hycar 422, a precross-linked powdered nitrile that was developed for PVC modification. (1, 2) When this compound was extruded in the customer's plant it ran smoothly at the beginning of the run. However, it suddenly degraded with catastrophic results. The customer was not deterred by this problem and requested that we develop a compound which had a slower rate of degradation, so that they had more time to react once they detected the onset of thermal degradation. Therefore, we initiated a study to develop a measure of rate of degradation. Once this was done, we then studied the effects of several Hycar variables on the thermal stability of these compounds during processing. This report covers both aspects of the study.

Conclusions

1. The standard Brebender Dynamic Thermal Stability (DTS) test can be used to obtain quantitative data on the processing safety and rate of decomposition of Geon/Hycar thermoplastic polyblends.
2. The processing safety relative to a PVC compound containing liquid monomeric plasticizer can be determined by the ratio of both the Brebender steady state torque values and the decomposition times of the rubber modified compound to the control.

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3. The rate of thermal degradation during processing relative to the PVC compound containing liquid monomeric plasticizer can be determined by comparing the rate of torque increase and the rate of temperature increase of the rubber modified compound in the degradation region of the curve, as relative to the control compounds.
4. This DTS test can be used in compound development to accurately determine the effects of compound variations as well as rubber changes on the processability of the blend. As a result, a compound was developed by the Geon group and extruded successfully at both Avon Lake and in the customer's plant.
5. The use of this technique revealed that compound processing safety was inversely related to the viscosity of the polyblend after fluxing.
6. The compound viscosity after fluxing was largely controlled by the viscosity of the nitrile rubber portion of the blend.
7. The data indicated that the processing safety and degradation rates were greatly improved by using low Mooney (30 or less) Hycar nitrile elastomers.
8. This study generated a new project aimed at developing powdered forms of very low Mooney viscosity nitrile elastomers for use in PVC compounds.

Future Action

Continue to use the DTS data to study the effects of compound variations on processability.

Generate Instron melt rheology data to study the effects of compound variations on the overall flow behavior of these materials.

Determine the feasibility of preparing free flowing powders of low Mooney Hycar nitrile elastomers.

Experimental Description

A number of powdered dry blends of Geon resin and Hycar nitrile were prepared according to the recipes shown in Table I. These materials were then used in the Brebender Dynamic Thermal Stability (DTS) tests under the conditions outlined in Table II. The changes in both torque and stock temperature were recorded during each DTS run. This data was then analyzed in a manner which gave quantitative information on the degradation process. Several modifications of the Hycar portion of the blend were made and subjected to DTS tests to determine their effect on processing safety.

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TABLE IBASE COMPOUND RECIPES*

<u>COMPOUND</u>	<u>PVC CONTROL</u>	<u>RUBBER MODIFIED</u>
Geon 92	100	100
DIDP	60	26
Hycar Powder	-	72
Tri Octyl Phosphate	10	10
Vinyzene BPSRG**	5	5
Antimony Oxide	9	9
Calcium Carbonate	21	21
Stabilizer	Variable	Variable
Lubricant	Variable	Variable

* Customer Confidential

** Fungicide

TABLE IIBRABENDER CONDITIONS FOR DYNAMIC THERMAL STABILITY EXPERIMENTS

Barrel Temperature	400°F
Rotor	Roller Type
Rotor Speed	80 RPM
Powder Charge Weight*	60 Grams

* Compound added with rotors turning

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Results and Discussion

Interpretation of DTS Test Data

The Brabender Dynamic Thermal Stability test (DTS) is frequently used during the development of PVC compounds. The data obtained from this test are shown in Figure 1 and consist of plots of torque and stock temperature vs time. The test results can be separated into three distinct phases as the mixing time increases. The first phase involves the fluxing of the compound ingredients into a uniform material. This is characterized by a steadily increasing temperature and a maximum in the torque plots. When the fluxing step is complete the material enters a steady state zone of constant torque and temperature. When the stabilizing system is depleted, the compound then enters the last phase, the decomposition zone. This phase is characterized by marked discoloration of the stock and a steadily increasing torque and temperature.

When this test was applied to the PVC control compound, (first recipe in Table 1) we obtained the expected pattern of temperature and torque as discussed above. However, when the rubber modified compound (second recipe in Table 1 with 72 parts Hycar 1422) was tested, we obtained the following qualitative differences.

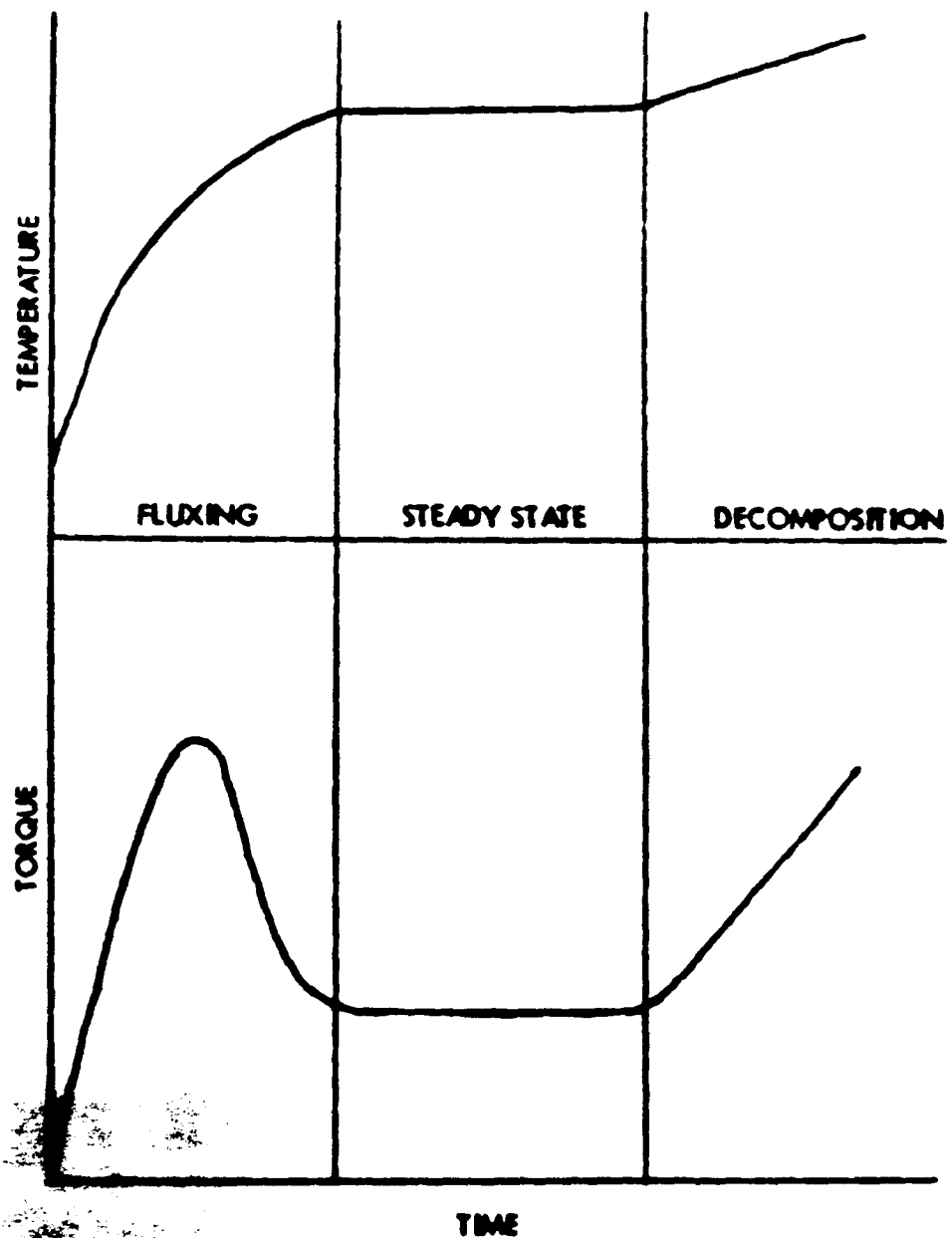
1. The steady state torque values were much greater than those of the PVC control.
2. The duration of the steady state zone was much smaller.
3. The stock temperature increased during the steady state torque region.
4. The slopes of both the torque-time and temperature-time curves in the decomposition range were much steeper.

I, therefore, decided to quantitate these differences to determine if any of the differences would correlate with the problem which arose during the extrusion run in the customer's plant. The results were calculated by the following procedure.

First, the steady state and decomposition zones were determined as shown in Figure 2. I adopted the following nomenclature for this analysis. Torque, temperature, and time values are indicated by M, T, and t respectively. The values of these parameters in the steady state region carry the subscript ss, while those in the decomposition zone use the subscript d.

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TYPICAL DYNAMIC THERMAL STABILITY DATA

FIGURE 1

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The following information is recorded in the steady state region. Note, I have defined the steady state region as one of constant torque.

- a. t_{ss} - the time to reach steady state
- b. M_{ss} - the torque at steady state
- c. T_{ss} - stock temperature at the onset of steady state

Likewise the following data is tabulated for the decomposition region:

- a. t_d - the time where the decomposition begins as evidenced by the increase of the torque above the steady state value.
- b. T_d - the temperature at which the decomposition starts, i.e. the temperature at t_d .
- c. Rate of change of torque with time dM_d/dt . The slope of the linear torque-time curve during decomposition.
- d. Rate of change of temperature with time, dT_d/dt . The slope of the temperature-time curve in the decomposition zone.

The last step is to calculate the following parameters:

- a. The time in the steady state region: $t_d - t_{ss} = \Delta t$.
- b. The rate of change of temperature during steady state:

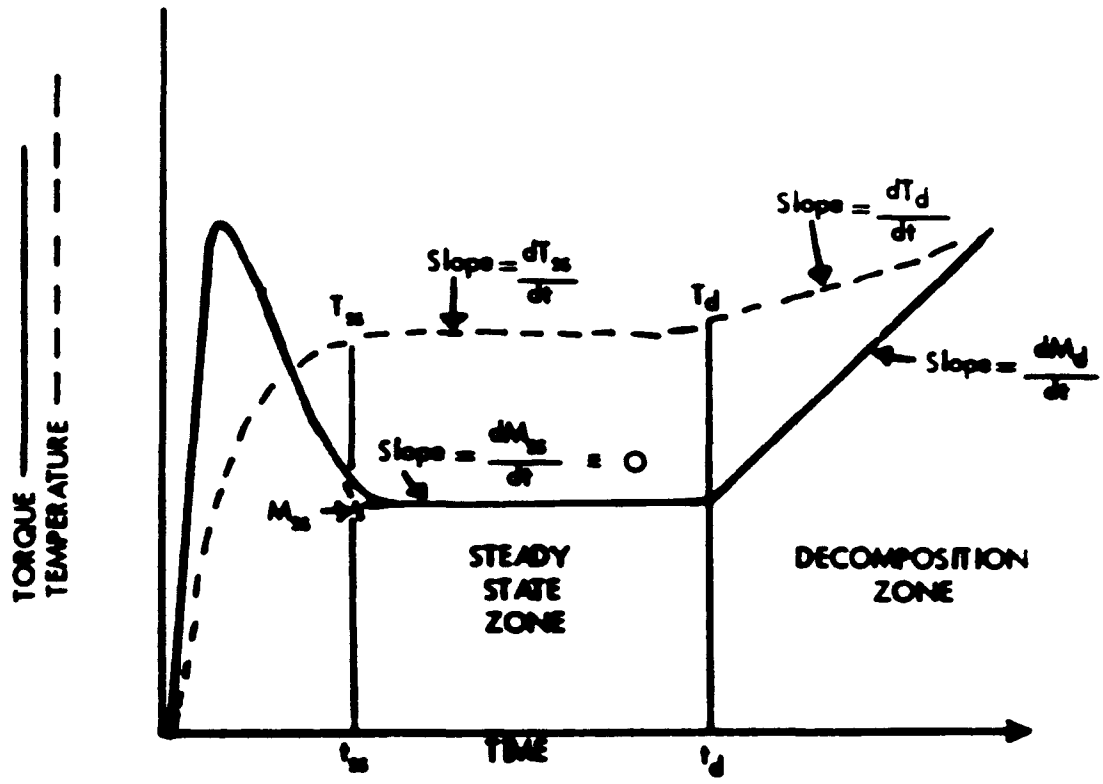
$$\frac{dT_{ss}}{dt} = \frac{T_d - T_{ss}}{t_d - t_{ss}}$$

These parameters were calculated for each of the compounds mentioned above and are tabulated in Table III. The PVC control compound required a long time to reach steady state. It started to decompose at 37 minutes and then entered a less severe mode of decomposition at 45 minutes. On the other hand, the rubber modified compound behaved quite differently. It reached steady state fairly quickly but entered the decomposition zone after nine (9) minutes of total running time vs 37 for the control. The torque at steady state was four and a half times (4-1/2) that of the torque of the PVC control. This torque is directly proportional to the viscosity of the stock which in turn is proportional to shear induced heating. The temperature at steady state is 26°F greater than the control, and more importantly rises at a rate of almost five (5) degrees per minute during the constant torque period. All of these observations indicate that rubber modified compound is less stable than the control because of the greater melt viscosity and the resulting increase in stock temperature due to shear heating.

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DYNAMIC THERMAL STABILITY TEST ANALYSIS

FIGURE 2

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TABLE III

DTS ANALYSIS OF CONTROL AND RUBBER MODIFIED COMPOUND

Sample Number	Composition		Steady State				Decomposition Zone				
	PDR Hycon	Stabilizer	t_{ss}	M_{ss}	T_{ss}	dT_{ss}/dt	t_d	T_d	dM_d/dt	dT_d/dt	
111-17-125-1 ^a	-	340 ^b - 2.35 phs	30 min	340 m-gm	384°F	0	7 min	37 min 45	384°F 388	$18 \frac{m}{min}$ 7	0.8°F/ 0.3
111-17-125-2 ^b	1422-72 parts	340 ^b - 2.35 phs	5'	1530	410	4.7°F/min	4	9	419	185	2.5

^a Table 1 PVC Control^b Table 1 Rubber Modified Compound^c Barium - Cadmium PVC stabilizer

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The behavior of both compounds during decomposition is also illuminating. The control compound goes through two relatively mild decomposition stages. The rubber modified compound undergoes a strong decomposition in which the rate of torque increase is more than ten (10) times that of the control compound and the temperature increase is three times that of the control.

Therefore, based on the above discussion, this method for analyzing DTS data provides us with several indicators of both the relative stability of the compounds plus the rate of decomposition. The suggested indicators are shown in Table IV. The two indicators of relative blend stability are in good agreement and show that the rubber modified blend has one quarter of the stability or processing safety of the control compound. The decomposition rate indicators show that the rubber modified compound degraded much more rapidly than the control. Therefore, the compound extruded at the customer's plant had two major shortcomings: it had only one quarter of the processing safety of a comparable compound which contained liquid monomeric plasticizer; and once it started to degrade, it decomposed at a tenfold greater rate.

Since we felt that the above indicators gave us an accurate picture of the processing safety of flexible PVC blends, we decided to use this interpretation of DTS testing data in our joint effort to develop a less sensitive compound.

TABLE IV

INDICATORS OF BLEND THERMAL STABILITY AND DECOMPOSITION RATE

Sample	Control	Relative Stability Indicators		Relative Decomposition Rate Indicators	
		Torque ^a	Time to Decomp. ^b	Torque Change ^c	Temp. Change ^d
111-17-25-2	111-17-25-1	0.22	0.24	10.3	3

a Steady state torque of control / Steady state torque of compound

b t_d compound / t_d control

c (dM_d/dt) compound / (dM_d/dt) control

d (dT_d/dt) compound / (dT_d/dt) control

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DTS Evaluation of Selected Stabilizer Systems

The Geon group made several stabilizer changes in the base recipe. The DTS data are compiled in Table V. It can be readily seen that none of these stabilizers produced significant reductions of the steady state torque and the resulting shear heating during steady state. The S-8 stabilizer did slow down the rate of torque increase (dM_d/dt) by a factor of three (3). While this is significant, we did not feel that this decrease was sufficient to warrant further evaluation given the reduced t_d values of the S-8 stabilized recipes.

DTS Analysis of Effects of Type and Level of Hycar Nitrile Rubber

All of the previous work was done using Hycar 1422 powder in the recipe. This polymer was specifically designed to give good color stability in PVC compounds,^(1,2) and is also precrosslinked to decrease the amount of nerve in the final compound. We decided to see if switching to a linear rubber would change the decomposition characteristics of the final compound. We looked at this linear polymer, Hycar 1452P-50, at two levels; 72 parts which would give the same amount of rubber as in the 1422 recipe, and 48 parts which should give the same compound hardness as the 72 part Hycar 1422 recipe.⁽³⁾

The results of the DTS analysis are given in Table VI; and the processing indicators are shown in Table VII. There is an interesting apparent contradiction in the data. First, the use of the linear polymer gives a significant reduction in steady state torque. This reduction is increased when the lower level of 1452P-50 is used. This data is in agreement with the data on the time to decomposition as shown in Table VII. However, once decomposition starts, the compounds containing the linear rubber decompose at one and a half to two times that of the compound containing the crosslinked rubber. This apparent contradiction is readily explained by the fact that the crosslinked 1422 was designed to give good thermal protection to PVC compounds. Therefore, it degrades at a lower rate than the linear rubber compound which was not designed for that purpose. This means that we have two (2) alternatives to reduce the rate of decomposition and shear heating in the compounds. We can either make a color stable uncrosslinked version of the Hycar 1422, or increase the overall stabilizer system in the compounds containing the Hycar 1452P-50.

Improvement in PVC Stabilization System

Since it was easier to make a compounding change than to develop a new powdered rubber, we decided to improve the compound stability and use the linear Hycar 1452P-50 as the nitrile portion of the blend. The Geon group, therefore, made several modifications of the stabilization systems. To be thorough, we evaluated their recommended stabilization system in compounds containing both the linear and crosslinked powdered Hycar elastomers.

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TABLE V
DTS ANALYSIS OF STABILIZER VARIATIONS

Sample Number	Connections		Steady State				Decomposition Zone			
	FOR Hy-car	Stabilizer	t_{ss}	M_{ss}	T_{ss}	dT_{ss}/dt	Δt	t_d	T_d	dM_d/dt
111-17-25-2	1422-72 parts	300 ^a - 2.35	5	1530	410	4.7	4	9	419	185
212-18-40-3	1422-72	50 ^b - 3	5	1670	413	3.3	1.5	6.5	418	66
212-18-40-4	1422-72	50 ^b 3 Keramid E-2 TE20G ^c 1	4	1960	418	3	2	6	424	85

^a Barium - Cadmium System

^b Thio Organotin System

^c Rubber Processing Aid

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TABLE VI

DTS ANALYSIS OF EFFECTS OF TYPE AND LEVEL OF HYCAR RUBBER

Sample Number	Composition		Steady State					Decomposition Zone			
	POA Hycar	Stabilizer	t_{ss}	M_{ss}	T_{ss}	dT_{ss}/dt	Δt	t_d	T_d	dM_d/dt	dT_d/dt
212-18-60-3	1422 - 72	SB - 3	5	1670	413	3.3	1.5	6.5	418	66	2.5
212-18-60-2	142750 - 72	SB - 3	8.5	1350	413	0.8	2.5	11	415	125	2.0
212-18-60-1	142750 - 48	SB - 3	10	1150	408	1.0	3	13	411	100	1.4

TABLE VII

PROCESSING INDICATORS OF COMPOUNDS MODIFIED WITH LINEAR AND CROSSLINKED NITRILE RUBBER

Compound	Control	Relative Stability Indicators		Relative Decomposition Rate Indicators	
		Relative Torque	Time to Decomp.	Relative Torque Change	Temp. Change
212-18-60-3	111-17-125-1	0.21	0.18	3.7	3.0
212-18-60-2	111-17-125-1	0.25	0.30	7.0	2.4
212-18-60-1	111-17-125-1	0.30	0.35	5.5	1.7

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The data are given in Table VIII and the PVC control is also included for comparative purposes. The results show that this high proportion of lead stabilizer-lubricant improved the compounds containing Hycar 1422 only a small amount. However, when this new stabilization system was used in the Hycar 1452P-50 compound, several positive changes were obtained. First, the steady state torque decreased by twenty five (25) percent, probably because of the extra lubricant. The decomposition behavior became complex with the first decomposition zone being followed by a second steady state zone before the final decomposition region was entered. This could reflect the action of each of the lead stabilizers. However, even more importantly, the first decomposition region is quite comparable with that for the PVC control. Therefore, the data indicated that this highly stabilized recipe (111-17-139-7) would have processing characteristics only slightly inferior to the PVC control compound. A large sample of this dry blend was prepared for extrusion tests at Avon Lake. The extrusion trial on the 2-1/2" NRM unit was successful. The trial was stopped at the request of the customer because he wanted to run the remainder of the dry blend at his plant. The customer's plant evaluation was also successful. These successful extrusion runs clearly demonstrate the value of the DTS test in predicting processing safety of rubber modified PVC.

Reduction of Compound Viscosity of Hycar 1422 Modified Compounds

The successful extrusion of the compound mentioned in the previous section was not the end of the project, however, because the Hycar 1452P-50 was not as readily dry blendable with the Geon compound. The basic reason for this is that the Hycar 1452P-50 consists of little chunks of rubber with an average particle size of 500 microns. When this material is dry blended with Geon 92 resin the rubber particles can be easily seen with the naked eye.⁽⁴⁾ Therefore, we decided that we would have to use a spray dried powdered rubber because of its smaller (50 micron) ultimate powder particle size. These particles cannot be detected in the final dry blend. Since Hycar 1422 is a spray dried product, we decided to see if we could further reduce the viscosity of these compounds by replacing part of the powdered Hycar with liquid Hycar 1312. A series of dry blends was prepared with different ratios of 1422 to 1312 as shown in Table IX. The processing indicators are given in Table X. A comparison of the first three (3) compounds in Table IX shows that the following changes occur when the ratio of Hycar 1312 to Hycar 1422 is increased: the steady state torque decreases, the time to degradation increases, and both the rates of torque and temperature increase during degradation decrease. These changes are quantified in Table X. It can be seen that the decomposition rates are equal to or less than those of the PVC control when one fourth or more of the powdered rubber is replaced by liquid rubber. The rheological implications of these results will be discussed in the next section.

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TABLE VIII
DTS ANALYSIS OF RUBBER AND STABILIZER VARIATIONS

Sample Number	Composition		Steady State				Decomposition Zone			
	For Hycar	Stabilizer	t_{ss}	M_{ss}	T_{ss}	dT_{ss}/dt	Δt	t_d	T_d	dM_d/dt
212-18-60-3	1422 - 72	S ⁹⁸ - 3	5	1670	413	3.3	1.5	6.5	418	66
111-17-142-3	1422 - 72	S ⁹⁸ - 15 S ⁹¹⁰ - 2 L ⁹³² - 3	4	.580	405	3.7	4	8	420	46
212-18-60-2	1452P50-72	S8 - 3	8.5	1350	413	0.8	2.5	11	415	125
111-17-138-7	1452P50-72	S27 - 15 S10 - 2 L32 - 3	13 26	1000 1210	405 416	0.4 0.3	5 6	18 32	407 418	28 35
PVC Control	-	S ⁹⁸⁰ - 2.35	30	340	384	0	7	37	384	18
								45	388	7

a Thio Organotin Stabilizer

b Lead Stabilizer 810

c Dibasic Lead Stearate

d Butyl Stearate Lubricant

e Barium Cadmium Stabilizer

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TABLE IX

DTS ANALYSIS - EFFECT OF LIQUID RUBBER ON COMPOUND PROCESSABILITY

Sample Number	Curing		Steady State			Decomposition Zone					
	PDR Myear	Stabilizer	t_m	M_m	T_m	dt_m/dt	Δt	t_d	T_d	dM_d/dt	dt_d/dt
212-10-00-3	1422 - 72	SB - 3	5	1670	413	3.3	1.5	6.5	418	66	2.5
212-10-00-11	1422 - 84	SB - 3	5	1150	398	1.7	4	9	405	13	1.0
	1322 - 18							20	416	72	2.5
212-10-00-10	1422 - 26	SB - 3	6	790	390	0.7	7	13	395	7.5	0.4
	1312 - 26		17	830	377	0.3	6	23	399	5.5	0.4
									33	403	86
212-10-00-6	1422 - 26	527 - 15	7.5	640	308	0.4	12.5	20	393	3.5	0.2
111-17-125-1	-	500 - 2.36	30	340	304	0	7.5	37.5	384	18	0.8
								45	388	7	0.3

TABLE X
PROCESSING INDICATORS OF COMPOUNDS BASED ON LIQUID AND POWDERED HYCAR ELASTOMERS

Compound	Control	Relative Stability Indicators		Relative Decomposition Rate Indicators	
		Torque	Time to Decomp.	Torque Change	Temp. Change
212-10-60-3	111-17-125	0.20	0.17	3.7	3.0
212-10-60-11	111-17-125	0.30	0.24	0.7	1.2
212-10-60-10	111-17-125	0.43	0.36	0.4	0.5
212-10-60-6	111-17-125	0.53	0.54	0.2	0.25

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The practical application of these findings to the development of a safe processing compound for Cooley Products caused new problems. Because of the sticky nature of the liquid rubber, it could not be added directly to the dry blend. It was diluted with the plasticizer to thin it out prior to adding it to the Henschel. However, a sticky powder resulted. Therefore, we tried to incorporate the liquid rubber in the powdered rubber by blending the latices prior to spray drying. The blend containing 25% liquid rubber spray dried to give a marginal powder. The 50% blend was much too tacky and did not make an acceptable powder. The 25% liquid rubber powder did not give an acceptable dry blend with the Geon resin because of the "wetness" of the dry blend. Therefore, we will have to come up with another method of preparing a low viscosity powder to improve the processing safety of rubber modified Geon compounds.

Rheological Considerations During Compound Development

A comparison of the processing safety indicators in Tables IV, VII and X show that there was a definite inverse relationship between the compound steady state torque and the time at which thermal degradation occurred. This observation points to the importance of shear induced heating during the extrusion process. The data in this report also indicates that the shear heating is influenced markedly by the viscosity of the rubber used in the blend recipe. We have previously shown that the viscosity of resin/rubber blends is a function of the volume fraction of nitrile rubber in the blend,⁽³⁾ and studies are currently in progress to measure the effect of rubber Mooney viscosity on blend flow properties.

We can, however, obtain an approximate picture of the Mooney effects by examining the data in Table IX in light of the data reported by P. H. Starnes.⁽⁵⁾ This data showed that the Mooney Viscosity of a Hycar 1091 was reduced from 72 to 37 when it was blended with 25 parts of Hycar 1312. He further reported that blends containing more than 33% of liquid Hycar 1312 were very soft and sticky and difficult to handle. This is in agreement with our observation that powdered rubbers containing 25% 1312 were marginal with respect to stickiness and powders based on 50% liquid rubbers were too sticky.

It is also possible to make an estimate of the Mooney viscosity of various solid-liquid rubber blends using the following equation:

$$\sqrt{\text{Mooney Blend}} = \phi_1 \sqrt{\text{Mooney}_1} + \phi_2 \sqrt{\text{Mooney}_2}$$

where ϕ_1 and ϕ_2 are volume fractions of components 1 & 2

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If the value of the viscosity of the Hycar 1422 is 81 and we assume that the Mooney viscosity of the liquid rubber is zero (0), the 50/50 blend will have a Mooney value of 20. The 75/25 blend would have a value of 45. These values are too low to be spray dried into an acceptable powder. Therefore, we have generated a new problem, namely to prepare a powdered form of a low viscosity nitrile rubber which contains the stabilization system of Hycar 1422. Efforts to prepare such a material are currently being evaluated.

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