Three-Dimensional Torque Rheometry Mapping of Rigid PVC Compounds

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This paper presents an improved method for presenting torque rheometer data as it applies to rigid PVC extrusion compounds. During extrusion of rigid PVC, the compound is subjected to various degrees of heating, shearing, and compression. Three-dimensional torque rheometry mapping can provide the rigid PVC formulator with greater insights into how a rigid PVC compound will perform on production extrusion equipment.

INTRODUCTION

Three-dimensional graphics take advantage of the human mind's remarkable ability to draw qualitative information and understanding from visual images. The visual impact of multidimensional graphs allows us to intuitively understand the numerous dimensions of nature and the interrelationships of time and space. Three-dimensional presentations of torque rheometry data should allow us to more easily comprehend and understand the complex relationships among time, temperature, shear, and rigid PVC fusion and melt properties. Adding a new dimension to torque rheometry graphics may substantially improve our ability to characterize and solve extrusion processing problems using torque rheometry and greatly enhance the probability of success when taking new rigid PVC compounds out of the laboratory and into the plant. Scaling up from the laboratory to production equipment is not an exact science. When scaling up, the probability of success depends in great part on an intuitive understanding of a compound's rheology as well as the processing characteristics and requirements of the extruders, tooling, and products.

EQUIPMENT SETUP AND DATA COLLECTION

The equipment used in this study was a Brabender Plasti-Corder model number EPL-V5502 with an oil-heated, type 6 mixing-measuring head. This instrument was interfaced with a NEC Powermate 386SX computer with 2 MB of memory, a 40 MB hard disk, VGA display, and a 16 mHz processing speed. Test results were printed on an Hewlett-Packard LaserJet III printer. Data were collected in a spreadsheet compatible format and imported into Lotus Symphony, where the data were analyzed, files combined, and numerical and two-dimensional graphic outputs prepared and printed.

Torque readings were collected at a rate of 120/min. Five readings were averaged to yield each data point. A total of 960 readings were taken in 8 min to yield the 320 data points plotted for each curve. Data from six torque rheometer runs were combined on each two-dimensional and three-dimensional graph. Thus, a total of 1920 data points were plotted on each torque rheometry graph.

PVC PROCESSING ON A TORQUE RHEOMETER VERSUS AN EXTRUDER

Figure 1 is a simple energy balance for a torque rheometer. The test procedure involves compaction of the test material to a fixed volume, during mixing and heating. The amount of work going into the material is indicated by the torque necessary to turn the mixing blades at a preset rpm. The amount of heat transferred into the material is mainly a function of material and bowl temperature. The melt temperature at any time t is dependent on the work and heat transferred into, or out of, the sample.

Figure 2 presents a simple fusion mechanism for rigid PVC being processed in a torque rheometer. Here the PVC resin grains undergo a compaction and densification process, yielding primary particles and primary particle agglomerates that eventually fuse together. The first three fusion transitions can be related to this compaction, densification, and fusion process. As mixing continues, the particle nature of the melt is gradually eliminated, provided a sufficient melt temperature is attained.

A simple energy balance for a twin screw extruder is shown in *Fig. 3*. Heat is transferred into or out of the melt in different sections of the extruder. Work input depends on the configuration of the extruder screws, compound characteristics, and processing conditions. *Figure 4* presents a simplified fusion mechanism for a twin screw extruder. Basically, after the material enters the extruder, it is heated

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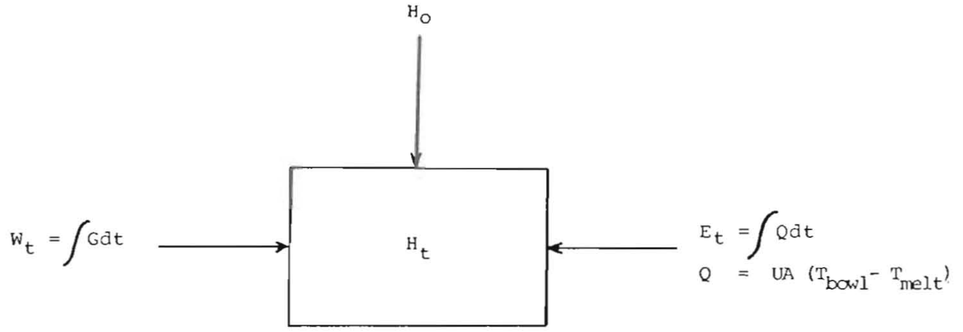
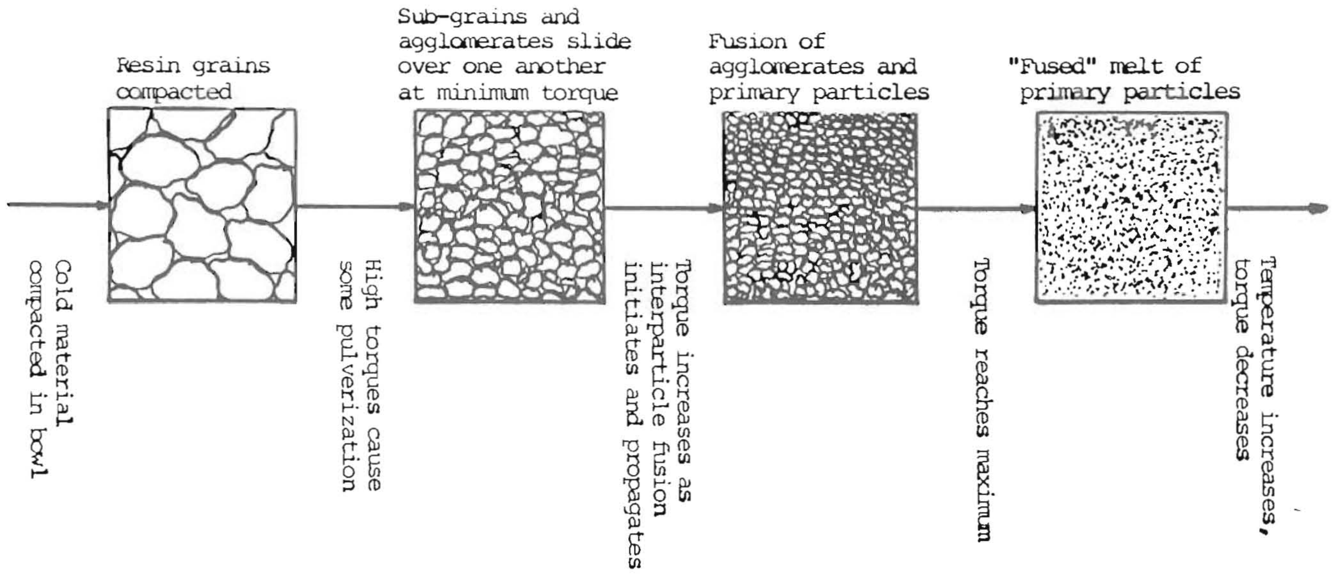


$H_t = H_o + E_t + W_t$
$T_{melt,t} = H_t / mC_p$
$H_o = mC_pT_o + P_oV$

Where: H_o = Initial enthalpy of material, BTU.
H_t = Enthalpy of material at time t, BTU.
E_t = Amount of heat transferred at time t, BTU.
W_t = Amount of work generated at time t, BTU.
m = Charge size, lb.
C_p = Heat capacity of material, BTU/lb°F.
P_o = Pressure upon charging, psi.
V = Bowl volume, ft^3.
T = Temperature, °F.
t = Time, h.
Q = Rate of heat transfer, btu/h.
U = Overall heat transfer coefficient, BTU/ft^2 °F.h.
A = Bowl surface area, ft^2.
T = Temperature, °F.
G = Torque, ft lb.

Fig. 1. Simple energy balance for a torque rheometer.



Where: $E = Q = \int U A\, dT$
= Rate of heat transfer, BTU/h.
W = Work imparted into material, BTU/h.
= f (equipment, compound, lubricants, feed rate, screw speed, etc...)
F = Extruder throughput, lb/h.
$H = F C_p T + PV$
Enthalpy of material, BTU/h.

Fig. 2. Rigid PVC fusion mechanism in a torque rheometer.

and compressed, and free space between the PVC resin particles and subparticles is gradually eliminated. Interparticle fusion initiates as the melt temperature increases. This results in an increase in melt viscosity. The morphology of the PVC subparticles change depending on local shear conditions in the melt.

As demonstrated in *Figs. 3* and *4*, during extrusion, PVC is subjected to a wide range of heating, shearing, and flow conditions. This includes conveying and feeding a free flowing dry blend into the extruder at ambient temperature, to a partially compacted melt/particle mixture that is subjected to a vacuum, to a fully compacted melt at 300 to 400°F+ under a few thousand psi of pressure at the screw tips, and finally to a fused melt exiting the die at 350°F to 440°F, perhaps under slight tension. During the past fifteen years, the author has made many observations in rigid PVC extrusion plants and has often attempted to relate industrial results to torque rheometer test results. In some cases, the torque rheometer and the plant results agreed. However, in perhaps an equal number of cases, the torque rheometer results and plant results did not appear to agree. From these experiences, it became apparent that an improved test method was needed. Given the differences between the heat and material balances and the proposed rigid PVC fusion mechanisms for a torque rheometer compared to an extruder, it appeared that three-dimensional graphics would be of considerable value in presenting, interpreting, understanding, and applying torque rheometer test results.

BRABENDER TESTS AND TWO-DIMENSIONAL GRAPHS

In order to improve the usefulness and reliability of torque rheometer data, it is helpful to run torque rheometry tests under a number of conditions. A broader view of the compound's processing behav-

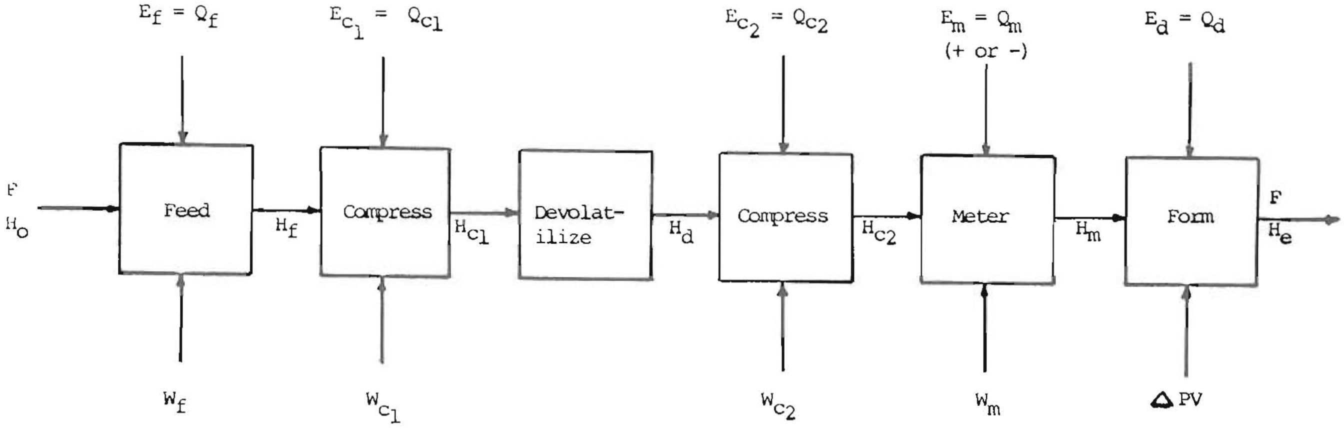


Fig. 3. Simple energy balance for a rigid PVC extruder.

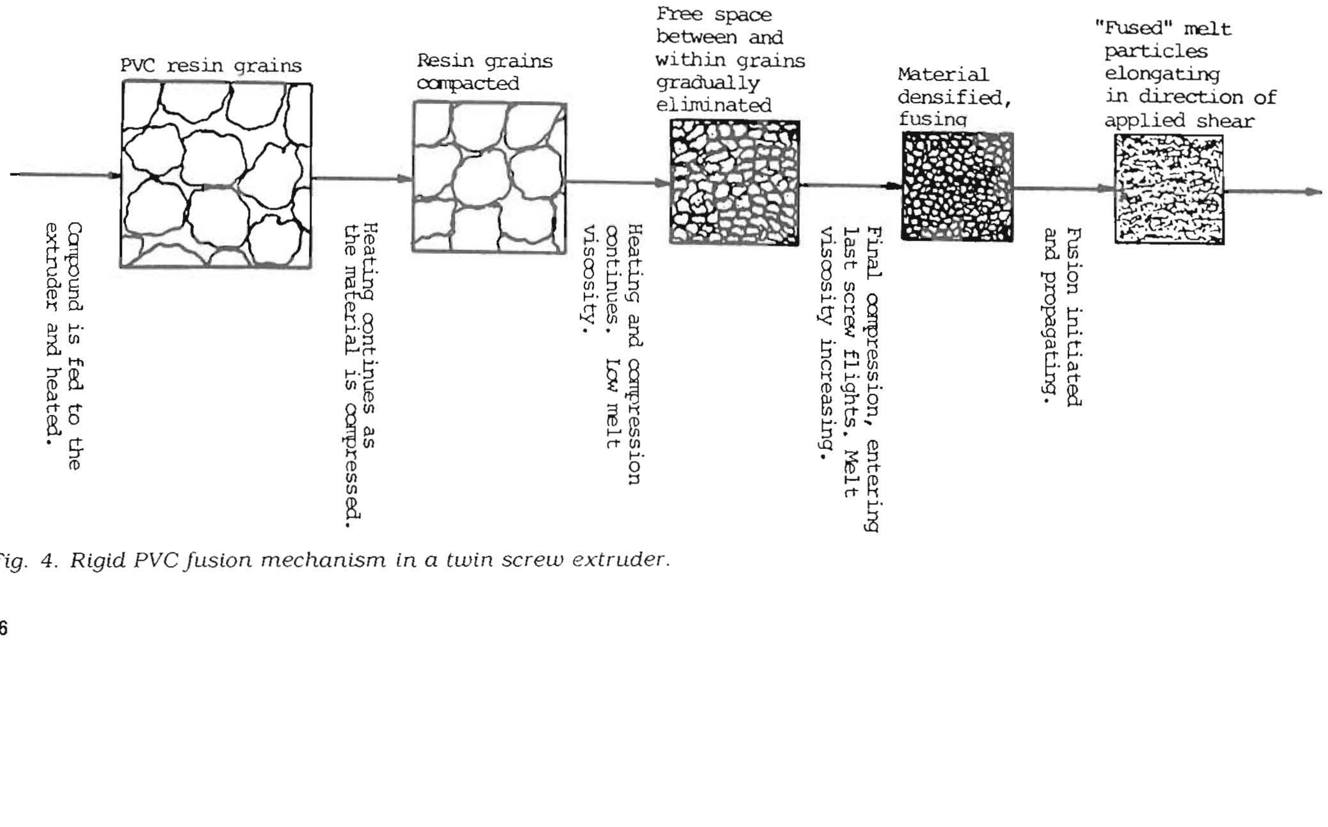


Fig. 4. Rigid PVC fusion mechanism in a twin screw extruder.

ior can obtained if tests are run at different rotor rpms, charge weights, or bowl temperatures. A common practice is to superimpose these test results on two-dimensional plots. This gives a rigid PVC formulator a better view of a compound's performance behavior over the range of test conditions.

To demonstrate the use of three-dimensional graphics, we evaluated a standard PVC pressure pipe compound under a variety of test conditions. The rigid PVC compound was as follows:

Ingredient	Amount
PVC resin	100.0 parts
Tin stabilizer	0.40 phr
TLP-2030 lubricant	2.00 phr
Calcium carbonate	5.00 phr
Titanium dioxide	1.00 phr

The TLP-2030 lubricant is a packaged lubricant system containing calcium stearate, paraffin, and polyethylene waxes. This formulation was evaluated under the following conditions:

1. Variations in rotor rpm: Tests were run using a charge weight of 65 g, an oil temperature of 185°C, and rotor speeds of 20, 30, 40, 60, 80, and 100 rpm.
2. Variations in oil temperature: Tests were run using a charge weight of 65 g, a rotor speed of 60 rpm, and oil temperatures of 165°C, 175°C, 185°C, 195°C, 205°C, and 215°C.
3. Variations in charge weight: Tests were run using a rotor speed of 60 rpm, an oil temperature of 185°C, and charge weights of 60.0 g, 62.5 g, 65 g, 67.5 g, 70 g, and 72.5 g.

The results for the above variations in test conditions, each with six curves superimposed on a two-dimensional graph, are shown in *Figs.* 5 through 7. To prepare these graphs, the test results from six different torque rheometry runs were combined onto one Lotus Symphony spreadsheet. The numerial output for the first 22 s of the variation in rotor rpm evaluations appear in *Fig. 8*. Superimposed two-dimensional graphs provide some greater insight into the processing characteristics of the compound over a range of processing conditions. However, it can be difficult to distinguish between the results from different test runs.

THREE-DIMENSIONAL TORQUE RHEOMETRY GRAPHS

Three-dimensional graphs of the same data that were plotted out in two dimensions appear in *Figs. 9* through *11*. The three-dimensional torque rheometry plots give a broader view, or a map, of a compound's processing behavior. As can be seen in *Fig. 9*, as the rotor rpm increases, fusion times decrease and compaction and fusion torques increase. In *Fig. 10*, the response of the sample to variations in oil temperature varies somewhat. Fusion times decrease with increasing oil temperature. Compaction

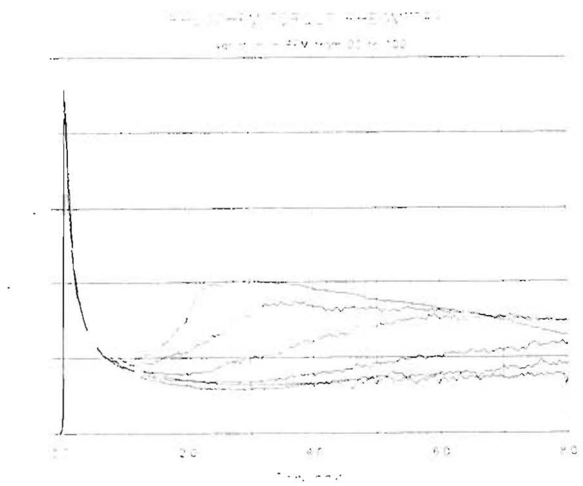


Fig. 5. Two-dimensional torque rheometry chart: Variation in rotor rpm from 20 to 100.

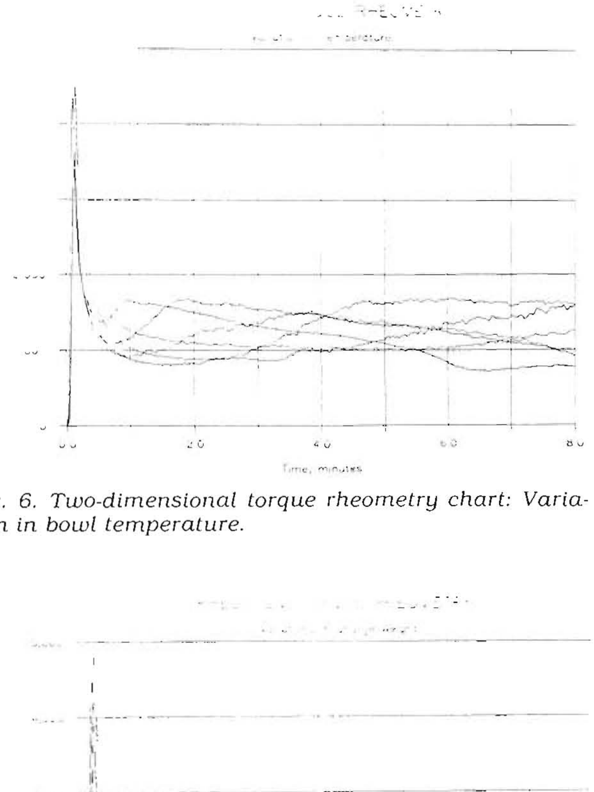


Fig. 6. Two-dimensional torque rheometry chart: Variation in bowl temperature.

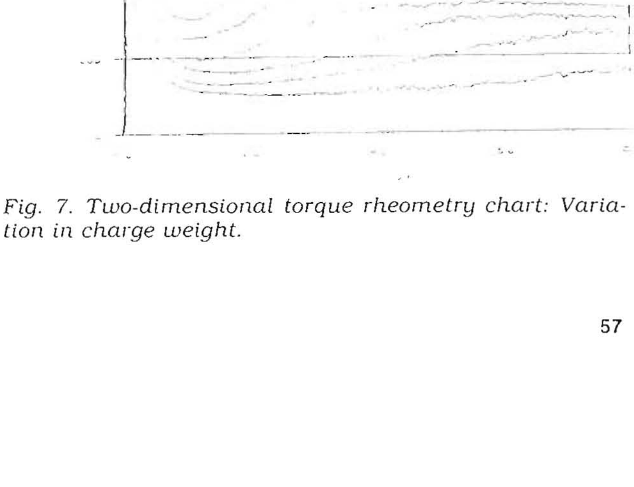


Fig. 7. Two-dimensional torque rheometry chart: Variation in charge weight.

RHEOCHEM MANUFACTURING CO., INC.
TORQUE RHEOMETRY FUSION TEST
VARIATION IN BRABENDER RPM FROM 20 TO 100

65 gram charge and 180°C oil temp

Time Sec	Min	20	30	40	60	80	100
0.0	0.000	0.0	0.0	0.0	12.2	24.4	24.4
0.5	0.008	0.0	12.2	12.2	24.4	24.4	36.6
1.0	0.017	12.2	12.2	24.4	24.4	48.8	61.0
1.5	0.025	0.0	24.4	24.4	36.6	85.5	85.5
2.0	0.033	12.2	36.6	48.8	73.2	122.1	122.1
2.5	0.042	24.4	48.8	97.7	109.9	317.4	268.6
3.0	0.050	24.4	109.9	317.4	170.9	842.3	817.9
3.5	0.058	36.6	317.4	854.5	366.2	1,709.0	1,879.9
4.0	0.067	85.5	793.5	1,623.5	915.5	2,734:4	3,161.6
4.5	0.075	195.3	1,440.4	2,404.8	1,782.2	3,747.6	4,406.7
5.0	0.083	415.0	2,124.0	3,100.6	2,771.0	4,626.5	5,029.3
5.5	0.092	732.4	2,734.4	3,710.9	3,686.5	4,956.1	4,992.7
6.0	0.100	1,123.1	3,247.1	4,235.8	4,467.8	4,748.5	4,663.1
6.5	0.108	1,538.1	3,686.5	4,626.5	4,785.2	4,406.7	4,296.9
7.0	0.117	1,953.1	4,064.9	4,687.5	4,638.7	4,028.3	3,918.5
7.5	0.125	2,282.7	4,406.7	4,614.3	4,321.3	3,723.1	3,601.1
8.0	0.133	2,539.1	4,492.2	4,272.5	3,942.9	3,442.4	3,356.9
8.5	0.142	2,807.6	4,382.3	3,967.3	3,649.9	3,222.7	3,161.6
9.0	0.150	3,027.3	4,113.8	3,698.7	3,381.4	3,027.3	3,002.9
9.5	0.158	3,198.2	3,857.4	3,442.4	3,149.4	2,880.9	2,868.7
10.0	0.167	3,356.9	3,613.3	3,259.3	2,966.3	2,758.8	2,734.4
10.5	0.175	3,430.2	3,405.8	3,100.6	2,807.6	2,648.9	2,612.3
11.0	0.183	3,430.2	3,186.0	2,905.3	2,685.6	2,563.5	2,526.9
11.5	0.192	3,344.7	3,015.1	2,771.0	2,563.5	2,465.8	2,429.2
12.0	0.200	3,125.0	2,880.9	2,661.1	2,478.0	2,368.2	2,343.8
12.5	0.208	2,917.5	2,795.4	2,539.1	2,380.4	2,294.9	2,258.3
13.0	0.217	2,746.6	2,661.1	2,465.8	2,319.3	2,233.9	2,185.1
13.5	0.225	2,648.9	2,575.7	2,368.2	2,258.3	2,172.9	2,124.0
14.0	0.233	2,490.2	2,465.8	2,282.7	2,197.3	2,099.6	2,050.8
14.5	0.242	2,417.0	2,392.6	2,246.1	2,148.4	2,063.0	1,989.8
15.0	0.250	2,319.3	2,294.9	2,185.1	2,087.4	1,989.8	1,953.1
15.5	0.258	2,209.5	2,246.1	2,111.8	2,038.6	1,953.1	1,892.1
16.0	0.267	2,172.9	2,185.1	2,087.4	1,977.5	1,904.3	1,843.3
16.5	0.275	2,148.4	2,136.2	2,050.8	1,940.9	1,855.5	1,818.9
17.0	0.283	2,050.8	2,063.0	1,977.5	1,892.1	1,831.1	1,770.0
17.5	0.292	2,026.4	2,026.4	1,953.1	1,855.5	1,782.2	1,733.4
18.0	0.300	1,965.3	1,977.5	1,904.3	1,818.9	1,757.8	1,696.8
18.5	0.308	1,879.9	1,953.1	1,867.7	1,782.2	1,733.4	1,660.2
19.0	0.317	1,831.1	1,892.1	1,843.3	1,745.6	1,696.8	1,635.7
19.5	0.325	1,831.1	1,879.9	1,806.6	1,721.2	1,660.2	1,586.9
20.0	0.333	1,770.0	1,831.1	1,745.6	1,684.6	1,635.7	1,562.5
20.5	0.342	1,757.8	1,806.6	1,733.4	1,660.2	1,611.3	1,525.9
21.0	0.350	1,721.2	1,806.6	1,709.0	1,635.7	1,586.9	1,403.8
21.5	0.358	1,648.0	1,757.8	1,672.4	1,599.1	1,562.5	1,489.3
22.0	0.367	1,611.3	1,733.4	1,660.2	1,574.7	1,525.9	1,452.6

Fig. 8. Numerical printout of torque rheometer data, first 22 s of run.

torques also increase with oil temperature. Fusion and equilibrium torques vary somewhat, showing peaks and valleys. *Fig. 11* demonstrates the effects of increasing charge weight. As the charge weight increases, fusion times decrease and fusion torques increase.

In order to demonstrate some of the differences between PVC compounds, the following highly filled PVC compound was evaluated:

Ingredient	Amount
PVC resin	100.0 parts
Tin stabilizer	0.40 phr
TLP-2030 lubricant	2.00 phr
Calcium carbonate	30.00 phr
Titanium dioxide	1.00 phr

This highly filled PVC compound was evaluated with an oil temperature of 185°C, a charge weight of 70 g, and rotor speeds of 20, 30, 40, 60, 80, and 100 rpm. *Figures 12* and *13* show conventional torque rheometer test results for the pressure pipe and highly filled PVC compounds, respectively, at a rotor speed of 60 rpm and an oil temperature of 185°C. The 60 rpm conventional torque rheometer results indicate that both compounds have similar fusion times but that the highly filled compound has a higher fusion torque.

Figure 14 shows the three-dimensional torque rheometry map for the highly filled PVC compound. When compared to *Fig. 5*, the results clearly indicate that the highly filled PVC compound has different processing behavior than the low filled PVC pressure pipe compound. Highly filled PVC compounds tend to be more difficult to fuse early in the extrusion process. However, later in the extrusion process, highly filled PVC pipe compounds are

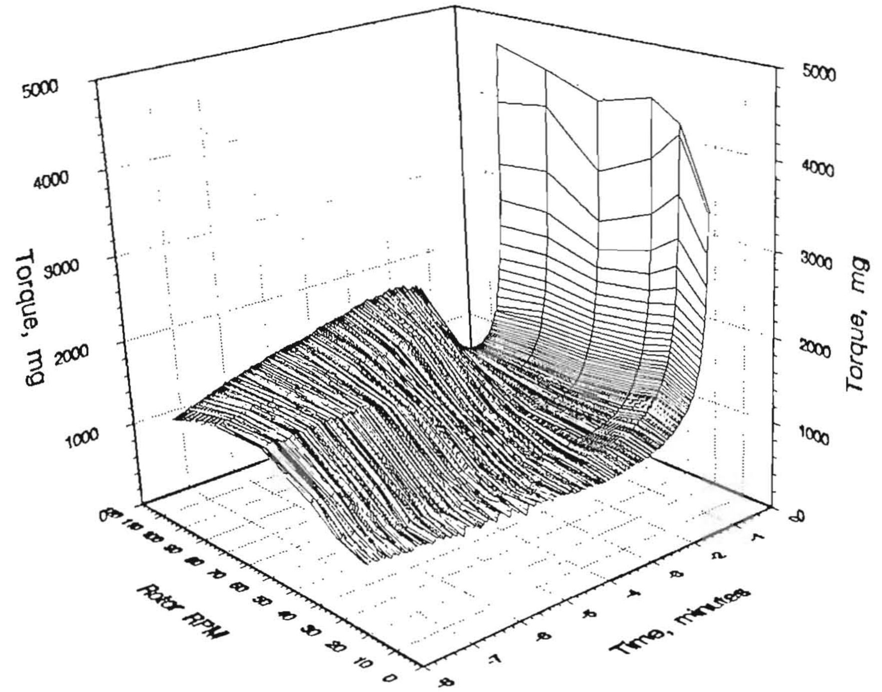


Fig. 9. Three-dimensional torque rheometry map: Variation in rotor rpm from 20 to 100.

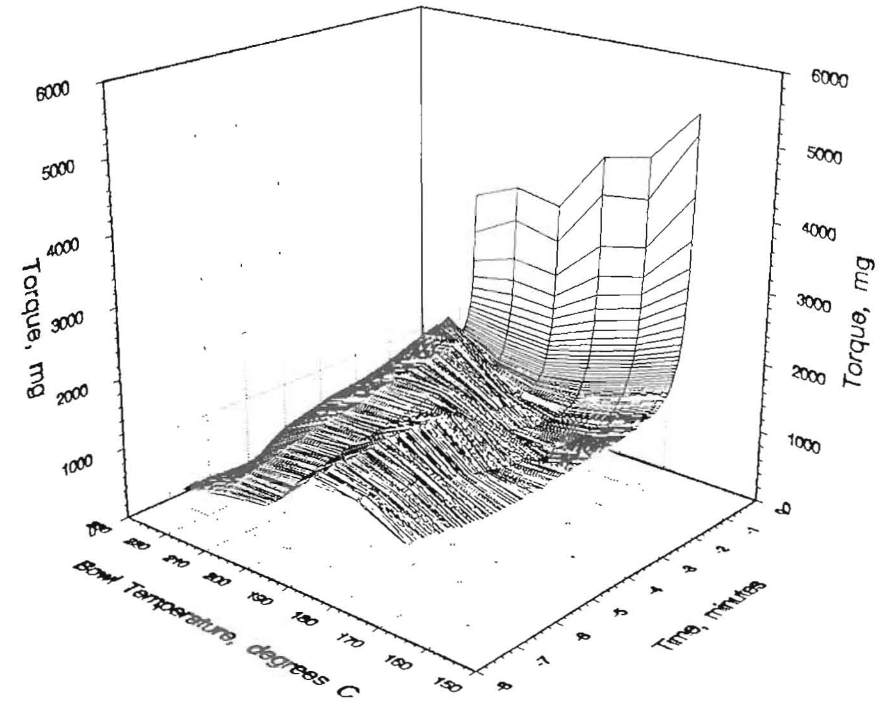


Fig. 10. Three-dimensional torque rheometry map: Variation in bowl temperature.

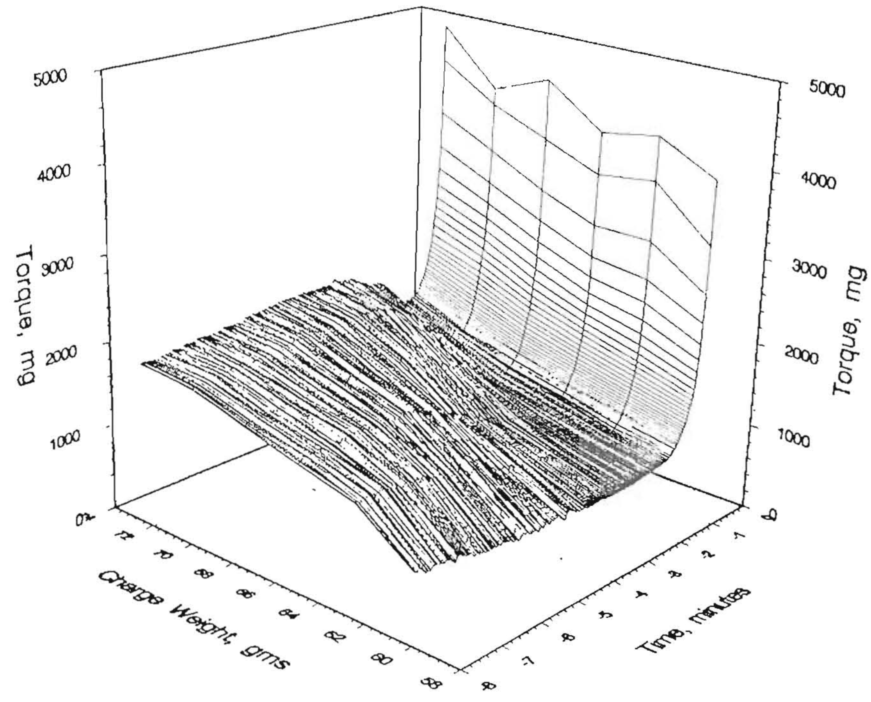


Fig. 11. Three-dimensional torque rheometry map: Variation in charge weight.

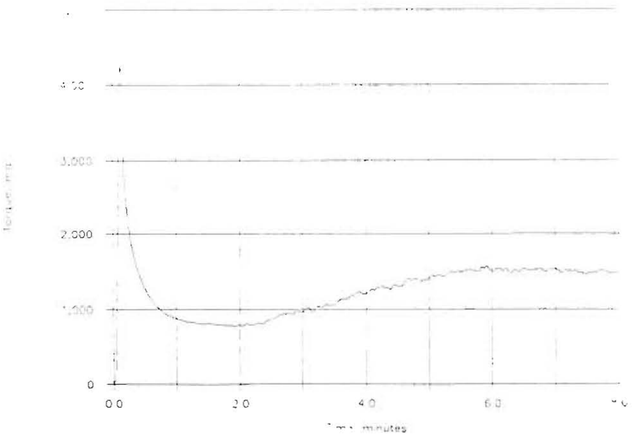


Fig. 12. Single run two-dimensional torque rheometry chart: PVC pressure pipe compound, 60 rpm and 185°C.

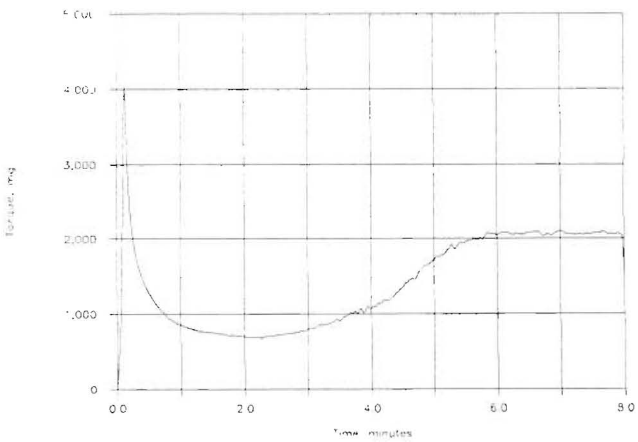


Fig. 13. Single run two-dimensional torque rheometry chart: highly filled PVC pipe compound, 60 rpm and 185°C.

known for their melt "stiffness." When comparing three-dimensional graphs for low filled PVC pressure pipe compound to the highly filled PVC compound, the differences between the compounds become more apparent and relate to known industrial experience. Looking at the three-dimensional torque rheometry results of the highly filled PVC compound, one would expect that in addition to its higher melt viscosity after fusion, the compound might be slower fusing than low filled pressure pipe compound.

CONCLUSIONS

1. Presenting torque rheometer data using three-dimensional graphics gives the user a greater sense of the compound's processing behavior over a range of conditions. This could greatly enhance our ability to understand and solve rigid PVC processing problems and provide greater insights into a compound's processing behavior from the powder stage to the finished product.

2. Variations in bowl temperature and rotor speed provide an interesting and complex range of results that merit further investigation. Variations in charge weight appeared to simply alter fusion time and torque levels depending on how much the bowl was packed.

3. Three-dimensional torque rheometry maps of PVC formulations could enhance a plant's ability to understand and troubleshoot compound problems

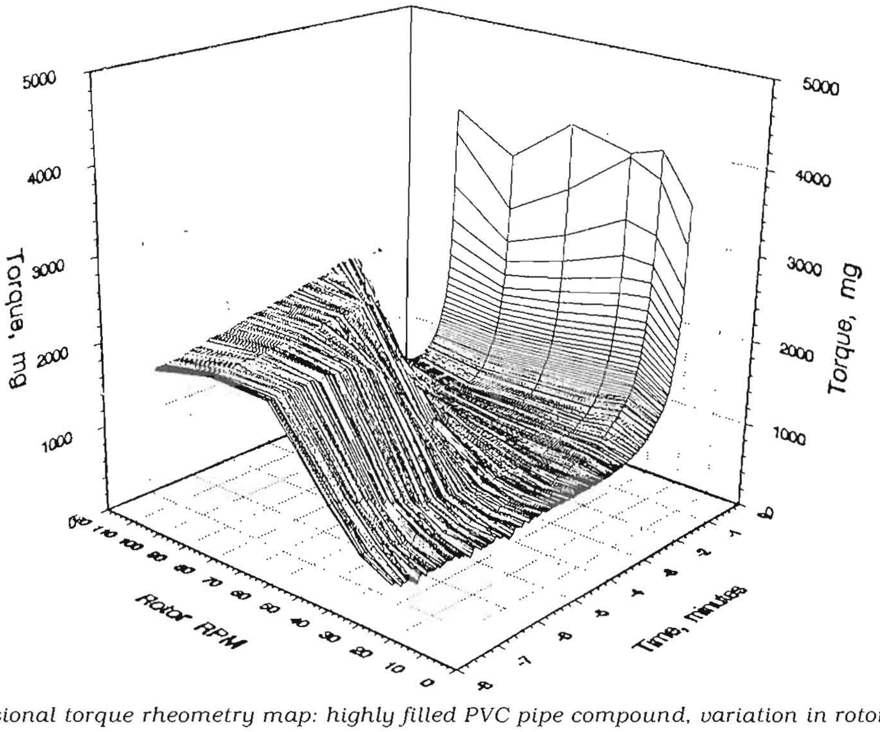


Fig. 14. Three-dimensional torque rheometry map: highly filled PVC pipe compound, variation in rotor rpm.

or reason out potential improvements to a PVC compound or screw and tooling setup. A three-dimensional torque rheometry graph gives one a sense of the stability of a compound's processing behavior over a range of conditions.

4. Three-dimensional presentations of torque rheometry data may prove to be a valuable improvement to the application and use of torque rheometers in the rigid PVC industry.

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