

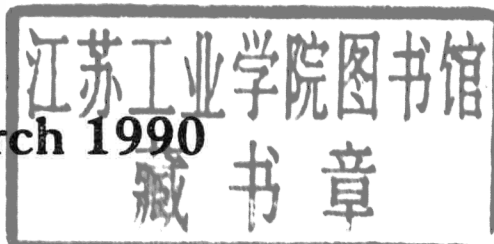
科技资料

Flow and Cure of Polymers- Measurement and Control

FLOW AND CURE OF POLYMERS – MEASUREMENT AND CONTROL

**Papers from a two-day seminar
organised by
Rapra Technology Limited**

22nd and 23rd March 1990



RAPRA
TECHNOLOGY LTD.

©Rapra Technology Limited, 1990

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted in any form or by any means, electronic, mechanical, photocopying, recording, or otherwise, without the prior written permission of the publisher, Rapra Technology Limited, Shawbury, Shrewsbury, Shropshire SY4 4NR, UK.

The view expressed in these papers are those of the individual authors and do not necessarily correspond to those of Rapra Technology Limited. These papers are published on the basis that no responsibility or liability of any nature shall attach to Rapra Technology Limited or the Editor arising out of or in connection with any utilisation in any form of any material contained herein.

FOREWORD

The manufacture of the great majority of polymeric products involves the flow of materials, followed, in many cases, by a curing reaction. It follows, therefore, that a greater understanding of flow and cure mechanisms can enable widespread improvements in processing efficiency and product properties.

Research into these properties has been undertaken at Rapra for many years and is continuing in the form of a number of multi-client projects. As with studies being undertaken at other industrial and academic laboratories, much of this work is now at the stage where it can be applied towards improvements in product design and manufacture.

Advances in measurement methods have allowed a much higher degree of accuracy than was previously possible. For example, isothermal methods are available for the measurement of cure characteristics, thus allowing much more exact predictions to be made of the optimum cure time for large or complex products.

Similarly, research into the combined effects of pressure, temperature and degree of cure on rheological properties is making it possible to perform more exact analysis of viscoelastic behaviour during processing.

At the same time, improvements in modelling techniques have allowed more accurate simulation of polymer processing, particularly in the development of computer-aided design systems and related predictive software. Initially, such software was restricted to the analysis of the injection moulding of thermoplastics. However, systems for more complex materials and a wider range of processes are now becoming available.

Research has also been undertaken on the relationship between factors such as the degree of cure and the final properties of polymeric products. Clearly, a better understanding of these relationships has implications in improving performance and in maintaining consistent quality.

Overall, this conference should provide guidance for delegates seeking to apply the results of recent research in the improvement of product quality and machine efficiency. Rapra Technology is pleased to express its gratitude to all of the speakers whose contributions will enable this aim to be met, in particular those that have travelled from the USA and from Europe to be present. Thanks to their efforts, Rapra is confident that the conference will provide two valuable days for all those attending.

Peter Dickin
Publications Group Head

CONTENTS

Application of Computer Aided Modern Rheometers for Testing the Processibility of Polymers

Herr. H. Luben, Brabender GmbH (West Germany)

Flow and Vulcanisation Studies of EPDM Rubbers using the Shimadzu Capillary Rheometer

M. Nasir, School of Industrial Technology (Malaysia) and Per Albihn, Swedish Plastics and Rubber Institute (Sweden)

Non-Standard Viscosity Behaviour of Polymer Melts and the Treatment in the BASF Database Program

Dipl.-Ing. Hans-Joachim Pitz, BASF AG (West Germany)

Shear and Extensional Flow in Rubber

Don Turner, Avon Rubber plc (UK)

Flow Analysis on Rigid PVC: The Importance of Elastic Effects

Dr. Vito Leo, Solvay & Cie (Belgium)

Torsional Braid Analysis (TBA): A Technique for Relating the Formation to the Properties of Thermosetting and High Temperature Polymers

Prof. John K. Gillham, Princeton University (USA)

Monitoring the Cure of Thermosetting Resins by DSC and Related Methods

Dr. John Barton, Royal Aerospace Establishment (UK)

Studies on Aromatic Amine-Epoxy Novolac Systems

C. Subramaniam, A. Padma and R.M.V.G.K. Rao, National Aeronautical Laboratory (India)

Dynamic Mechanical Analysis using the Bohlin VOR Melt Rheometer

Dr. R. Hall, Bohlin Reologi (Sweden)

The Practical Value of Rheology

Mr. Roy Carter and Dr. Frank Baker, Carter-Baker Enterprises (UK)

The Changes of the DC Resistivity of Rubber in the Curing Process

Ivan Kos, Taurus Hungarian Rubber Works (Hungary)

Modified Cure Rheometer for Isothermal Measurements

Dr. Peter Freilander, Carl Freudenberg (West Germany)

Optimisation of the Vulcanisation Process

J. Steen, Centre for Polymeric Materials (The Netherlands)

FDEMS Sensing for Continuous Online Insitu Monitoring and Control of Cure

Prof. David Kranbuehl, College of William and Mary in Virginia (USA)

Heat Transfer During Autoclave Cure of an Epoxy Resin Prepreg

Dr. Charlotte Morrison, University of Surrey (UK)

Cure Rationalisation Using a Vibrating Needle Curemeter

Dr. Bryan Willoughby, Rapra Technology Limited (UK)

The Application of the Strathclyde Curometer to a Variety of Reactive Systems

Prof. Richard Pethrick, Strathclyde University (UK)

Rheological Testing of Raw Rubber Polymers to Indicate Links Between Their Molecular Structure and their Processibility

Dipl.-Ing. Gebhard Schramm, Haake Mess-Technik (West Germany)

Viscoelastic Properties of Polymers During Cure Using the PL-DMTA Torsion Rheometer System
Dr. John Duncan, PI Thermal Sciences (UK)

Compaction and Consolidation of Thermoset and Thermoplastic Composites
Prof. George Springer, Stanford University (USA)

Mathematical Modelling of the Flow and Cure Phases During the Injection Moulding of Thermosetting Plastics
Dr. Mike Thomas, Rapra Technology Limited (UK)

Understanding the Factors Controlling Pultrusion: Characterisation of the Process by Die Interfacial Shear Stress Measurement and Thermal Modelling
Dr. Geoff Gibson, University of Liverpool (UK)

'Curetrac' - An Automatic Cure Correction Programme for Injection Moulding
Roland S.H. Farrer, REP Machinery (UK) Limited (UK)

On-Line Regulation for Injection Moulding
Dr. Volkmar Pogatzki, CAS (West Germany)

Applications of Computer Aided Modern Rheometers for Testing the Processability of Polymers

Dipl.-Ing. (FH) Heinz E. Luben

Brabender GmbH

Kulturstr. 51-55, Postfach 35 01 62, D-4100 Duisburg 1, West Germany

Approximately 60 years ago the first Torque Rheometer was designed to test the quality of wheat flour. This instrument, the Farinograph®, which means 'Flour Recorder', became the most famous Torque Rheometer in the milling and baking industry around the world.

Then, as the polymer industry grew up, a stronger Torque Rheometer was developed to measure the processability of the new materials. The name given to this instrument was the Plastograph®. During the following decades, many different types of Plastograph® and Plasti-Corder® measuring instruments became well-known in all areas of the polymer industry around the world.

There are also other test procedures available for determining the viscosity of polymers in the plastics and rubber industry. However, some of them use test procedures which are based on test conditions far from industrial practice.

The Torque Rheometers Plastograph® and Plasti-Corder® however, work according to a practice-related dynamic measuring method. The principle is based on the fact that the resistance which the test material puts up against rotating blades, rotors, screws, etc. in mixer measuring heads or measuring extruders can be made measurable. The corresponding torque moves a dynamometer from its zero position. In accordance with established test conditions a typical Plastogram (torque versus time diagram) is traced for every type of material. At the same time the stock temperature is recorded. Thus, a direct relation between viscosity and stock temperature is produced. The torque is measured in Newton-meters, formerly meter-gram, and the temperature in degrees centigrade. The test results appear in either analog or digital form on computer monitors or display instruments, or as diagrams which are recorded or printed.

Whenever a computer is used to support such measurements the software is of utmost importance. There are many different software packages available for the various test procedures. A lot of experience is necessary to develop high quality software packages, and the quality of the software

is responsible for the accuracy of any computerised rheometric measuring system.

A good software program should guide the operator from the very beginning to the end of the particular test. This helps skilled as well as unskilled users gain far more uniform test runs and so increases the reproducibility of test results. The quality of the automatic evaluation of the test results, from such computer aided rheometric measuring systems also depends highly on the quality of the software. It is also very important to increase the objectivity of the evaluations of the individual test results, and to make use of new possibilities for computerised evaluation, such as a good correlation software package to correlate individual test results for limit-values, for average values and to see the deviations and to learn about the reasons for their occurrence.

Parameters which influence the viscosity of the material under test such as the temperature of the measuring heads and/or the shear rate can be varied over a wide range. Therefore it is possible to create test conditions similar to the processing conditions of mixers, moulds, calenders, extruders, and screw injection moulding machines. Once the influence of the test conditions on the test material is known, the processing conditions on production machines can be adjusted quickly and reliably. In addition, the influence of changes in a compound on the processability can clearly be demonstrated and the influence of stabilisers, plasticisers, lubricants, fillers, catalysts, pigments, etc. can be determined.

The concept of processability is not defined completely by only testing melt viscosity of the individual material. The total plasticising behaviour, or the fusion rate, for example, is part of the processability. The heat- and shear-stability of polymer compounds, depending on the basic material and on different additives is also part of the processability, which can be determined on the one hand, and influenced on the other hand. The same is true for the flow-curing behaviour of various types of polymers.

Different mixer measuring heads can be used

with one basic instrument, and there are different test methods to obtain the different criteria of the processability of polymers.

Further, it is meaningful to complete the tests with mixers and with laboratory extruder tests. Different types of extruders developed for various applications are available to determine the extrudability of polymers. The flow of the melt through dies is observed, and the extrudate is checked for melt fracture, swelling, gloss, stips, pigment dispersion etc. Stock temperature and stock pressure can be measured in the different die heads and in the barrel of the measuring extruders. Special rheometric capillary die heads are available for research and production control purposes for direct viscosity measurements of the processed polymer.

Steadily increasing quality requirements in the various fields of the polymer industry and, at the same time, the increasing pressure of costs create a growing number of test problems for manufacturers of polymers, for processors of polymers, and last but not least for the polymer machinery industry.

They all need the best information available about the processability of the individual types of polymer in order to be able to produce high quality products under reasonable technical and financial conditions, and, at the same time, energy should be saved as far as possible.

Last and by no means least test instruments and test methods must be easy to handle, and test results must be reproducible. On the one hand

there is a desire for more and better tests using highly sophisticated instruments and test methods. On the other hand, there is a lack of skilled personnel for the evaluation of test results. At the same time it is necessary to reduce the possibilities of subjectively influencing the test results, in order to improve the reproducibility and comparability of results being obtained and evaluated, for instance at different locations.

There are solutions to all of these problems using more and more computerised versions of the different test instruments.

In the following text some of the most important test procedures are presented. We need to discuss how to measure and why, how to evaluate the results, and how to make use of these results to improve the quality of the many different polymer products.

Before we start to discuss the different test methods let's imagine a normal polymer processing machine:

A screw which can have different configurations is rotating in a temperature conditioned barrel. Along the barrel there is first a hopper where the powder or granules are loaded into the machine. This polymer material is compressed in the first zone of such a machine, then it starts plasticising, and, after having travelled through approximately two thirds of the length of such a plasticising machine, all particles are completely molten and then homogenised and finally pushed out through a die of certain shape, or into a mould.

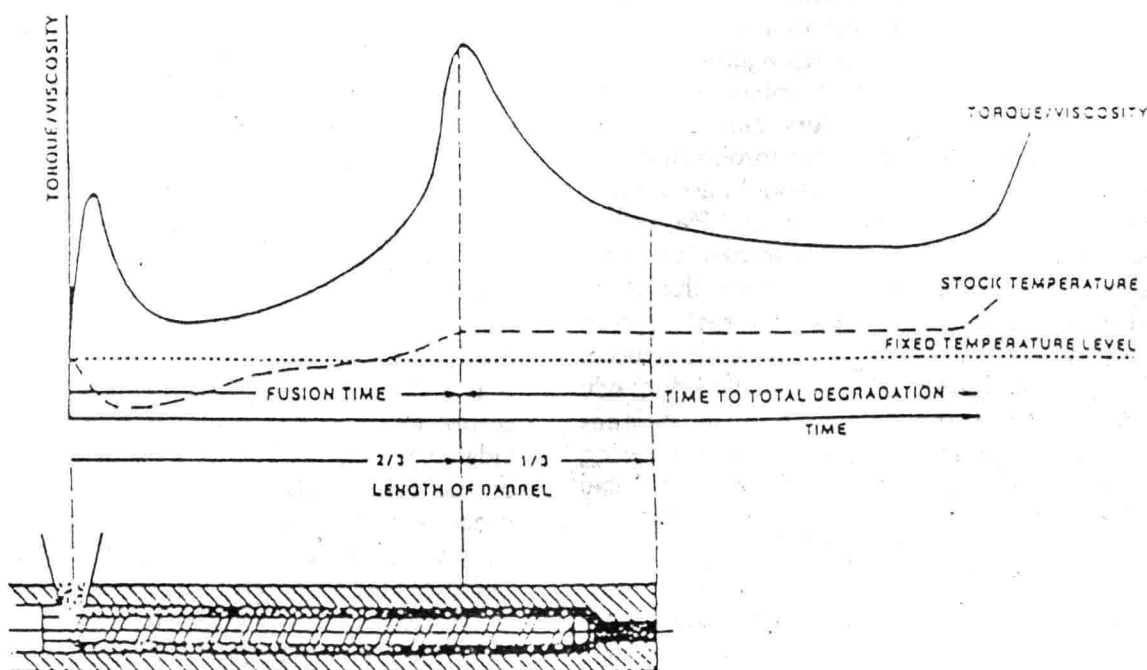


Figure 1 Torque along the screw of a polymer processing machine

What happens to the torque along the screw of such a machine? First there is the loading peak, from loading the material into the barrel. Then the torque drops in the compressing area, and climbs up in the plasticising area to a maximum which is called the fusion point. After this it drops slightly to the homogenising level. For many purposes this is the ideal version of such a process.

Let us imagine, for example, what would happen if we run the same process under higher temperature conditions. This would mean that the whole torque level would be lower, the plasticising time or the fusion rate would be shorter, but eventually the time for homogenising the material in the machine would be too long, so that degradation might take place in the barrel before the material is pushed out. The result would be an unsatisfactory product.

Now for example let's imagine running this process under lower temperature conditions. This would mean that the whole torque level would be higher, possibly too high for a particular machine and the plasticising time or fusion rate would be longer. The time for homogenising the material, then might not be long enough, and the result again could be an unsatisfactory product.

More examples could be demonstrated, but we want to know how our particular material reacts under various shear rates and temperature conditions, and how we can make this measurable in order to be able to influence the behaviour of the particular material, and to compare the results of processing by testing under different conditions.

Because of the fact that we cannot look into such a screw plasticising machine, and because of the fact that the total torque of the rotating screw in such a machine is a mean-value of the different torque levels and peaks along the screw. We need an instrument which is capable of simulating as closely as possible such a dynamic plasticising machine, to test the individual portions of the total processing procedure, for example the plasticising behaviour, the heat and shear stability, the flow curing behaviour, etc. etc., depending on the different types of polymers and additives.

The most suitable device for such measurements is a Measuring Mixer attached to a Torque Rheometer.

TESTS WITH MIXER MEASURING HEADS

There are many different types of exchangeable Measuring Mixers available for the different test methods, and in addition to the Measuring Mixers there are also interchangeable Measuring Extruders and die heads available to run practical tests.

After many years with mechanical Torque Rheometers different types of electronic Plasti-

Corders have been available for approximately one decade. Several years ago Brabender developed the first computerised Torque Rheometer System, the most modern type of which is the Data Processing Plasti-Corder®.

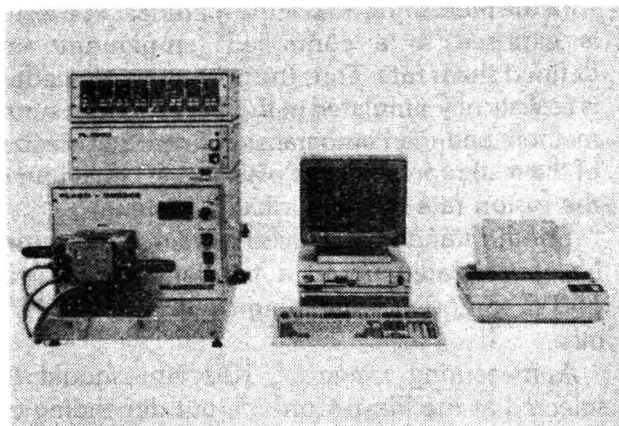


Figure 2 Data-Processing-Plasti-Corder® PL 2000 with Measuring Mixer

The test result is a torque/time and stock temperature diagram which is displayed on the monitor of a computer as the test is running and is printed out using fast printer. This means that the operator can follow the test by watching the diagram developing on the screen. The completed diagram is available at the end of the test, immediately followed by the automatic evaluation which is almost complete by the time the measuring mixer is cleaned.

The result of each test, which is the diagram and all evaluated data, can be saved on a disk for reprinting and/or for correlation at any time. The test parameters of course can be used for the next test without having to re-enter them each time.

PLASTICISING BEHAVIOUR OF PVC

There are many different PVC types offered on the market, and more specifically many possible PVC mixes available which differ in both type- and volume of their components. These mixes either require processing in different types of production machines or it is necessary to find the recipe for a specific mix that gives rise to no problems in an existing machine.

Therefore the PVC mixes must be studied for their processability, in order to achieve a well-balanced optimum regarding the processability as well as the final product quality.

For testing the plasticising behaviour of PVC, (also called the fusion behaviour of PVC), a Measuring Mixer with roller blades is attached to the Plasti-Corder®.

A thermocouple is inserted through the bottom

into the measuring chamber in order to pick up the stock temperature during the test, and this temperature is recorded simultaneously with the torque curve of the test. A smaller mixer (type 30) is best suited for rigid PVC types creating very high torque levels.

In the Measuring Mixer, the material to be tested is exposed to a controlled temperature and defined shear rate. Thus the processing procedure is realistically simulated in this dynamic measuring method, and the Plastogram, (the resulting diagram of the test) represents the plasticising behaviour or the fusion rate of the particular material.

For the standard plasticising test the Measuring Mixer is heated up to a temperature between 140°C to 160°C depending on the type of PVC mix.

A measuring range of 100 Nm should be selected at the Plasti-Corder®, but depending on the type of material a lower torque range should eventually be selected for a good test result resolution.

Experience has shown that a speed of 30 rpm is most suitable when studying the plasticising behaviour of PVC mixes. An increased speed results in shorter plasticising times and a decreased speed results in extended plasticising times.

Eventually some of the test parameters should be changed after the first preliminary test for better evaluation of the diagrams and for better correlation and evaluation of the differences between diagrams of different materials tested.

The sample to be tested is introduced into the Measuring Mixer with the help of a loading chute. In order to obtain reproducible and comparable test results the sample must be loaded into the mixer within 20 seconds, or even better 10 seconds. The loading time tolerance shall not exceed ± 5 seconds in a series of tests. Further it is important that the range of the sample weight does not exceed ± 0.1 g in a series of tests.

The Plastogram will now show clearly the individual parameters of the plasticising behaviour of the particular PVC mix. At the start of the test the torque increases rapidly to the loading peak during the loading phase. The sample then is compressed and heated in the mixer, a process which requires less torque so the minimum point is reached. During plasticising the material, an increasing torque is recorded and the maximum point of the torque curve is reached when all PVC particles are plasticised.

The time from the loading peak to the maximum point of the torque curve is identified and evaluated as the plasticising time, also called the fusion time. After the plasticising point, also called

the fusion point (the maximum torque value), the torque drops to a value which allows further mixing and homogenizing of the melt in the mixer. The maximum torque value of the diagram represents the required power for the processing machine.

The plasticising time enables the user to establish the screw length required to plasticise a particular PVC at a certain screw speed and barrel temperature, or which speed and which output are possible with a given screw length under certain temperature conditions.

With this testing method the most efficient processing conditions can be found for many types of polymer compounds and new recipes can be developed as well.

Looking back to the discussion of the behaviour of the material in a screw plasticising production machine, we clearly recognise that the torque curve along the screw is in principle identical to the torque curve of the plasticising behaviour test with the measuring mixer. The same is true in respect to the following test.

HEAT- AND SHEAR-STABILITY

The heat- and shear-stability of a polymer compound represents the next processing problem to be solved. We need characteristic values which we can use for our production line, and we need a well-balanced formula for the compound in order to make sure that it can be processed without problems on an existing machine, providing the required quality in the final product. To test the heat- and shear-stability of a polymer mix we again use a Measuring Mixer on a Plasti-Corder®.

The plasticising behaviour of a polymer is normally measured at processing temperature conditions. The heat- and shear-stability could be measured under the same temperature conditions, however, under such conditions it would take rather a long time before the degradation or decomposition of the material took place.

In order to save time and to be able to run more tests per day, the heat- and shear-stability is normally tested under higher temperatures and/or under higher speed conditions. Under such increased temperature conditions, for example 180°C or 200°C, the degradation, decomposition or crosslinking of the melt will be initiated much earlier and the diagrams will be easier to evaluate.

Under such high temperatures and/or speed conditions, however, the plasticising behaviour of the material cannot be followed in detail, any more, because this reaction is accelerated at the beginning of the test.

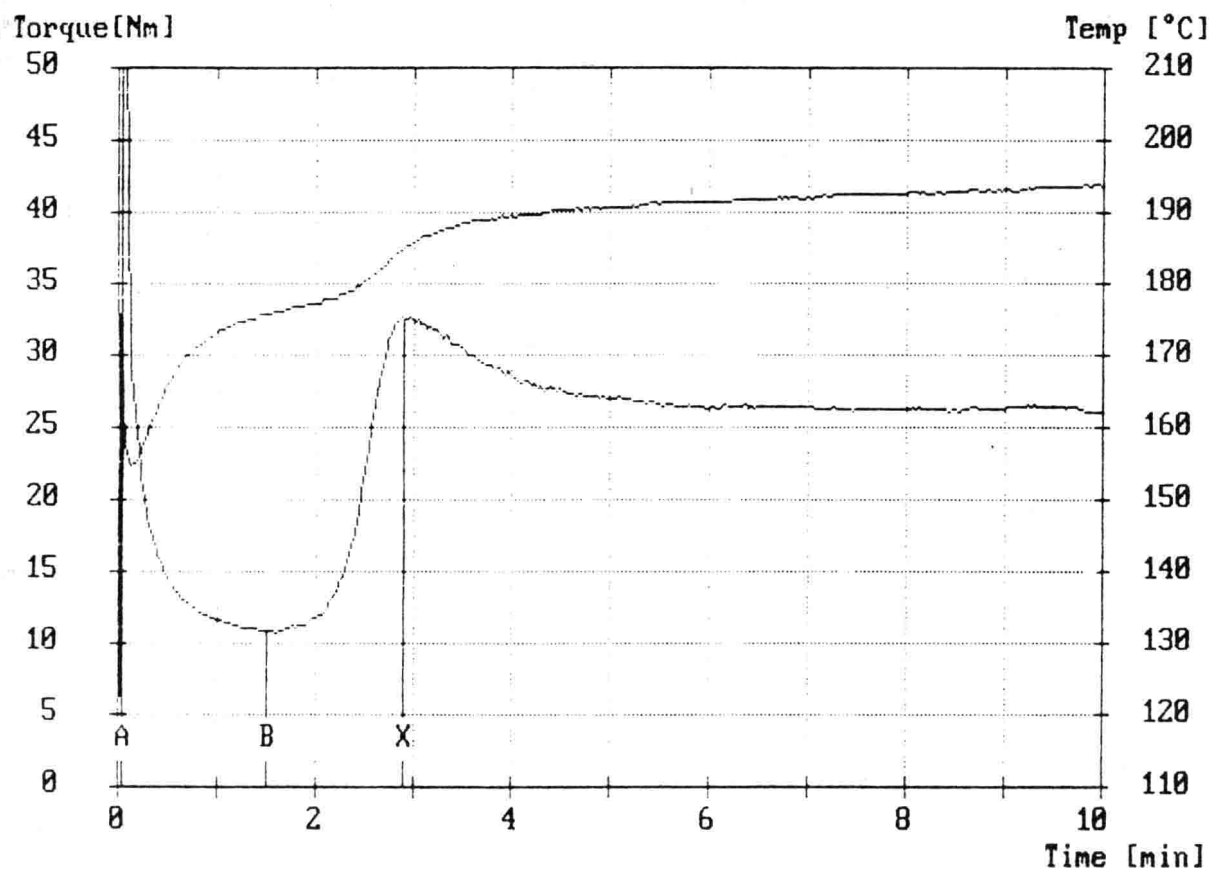
As soon as the degradation takes place in the Measuring Mixer the torque increases more or less

BRABENDER

Data-Processing Plasti-Corder PL2000 and Mixer Measuring Head
Fusion Behaviour / 0.6.7

Test-Conditions

Order	: BRABENDER	Mixer-Temp.	: 180	[°C]
Operator	: EICKMEIER	Speed	: 60	[1/min]
Check-Date	: 16. MAY '88	Meas. Range	: 50	[Nm]
PL-Type	: 2000-3	Zero-Suppr.	: 0	[%]
Mixer-Type	: W 50	Damping	: 3	
Load. Chute	: MANUAL + 5 KG	Test-Time	: 10	[min]
Sample	: PVC	Sample-Weight	: 57	[g]
Additive	:	Codenummer	: 1	
		Start-Temp.	: 178	[°C]



Value	Time [s]	Torque [Nm]	Stocktemp. [°C]
Load.Peak	2	58.0	162
Minimum	90	10.8	176
Maximum	174	32.3	185
End	600	26.1	194

Integration

- Load.Peak	to	Minimum	:	W1 =	8.6	E3 [Nm]
- Minimum	to	Maximum	:	W2 =	9.5	E3 [Nm]
- Load.Peak	to	End	:	W3 =	90.9	E3 [Nm]
- Specific Energy	(W3/Weight)			:	W4 =	1.6 E3 [Nm/g]

Filename : A:\PL2000\MIXEVAL\DATA\TEST1

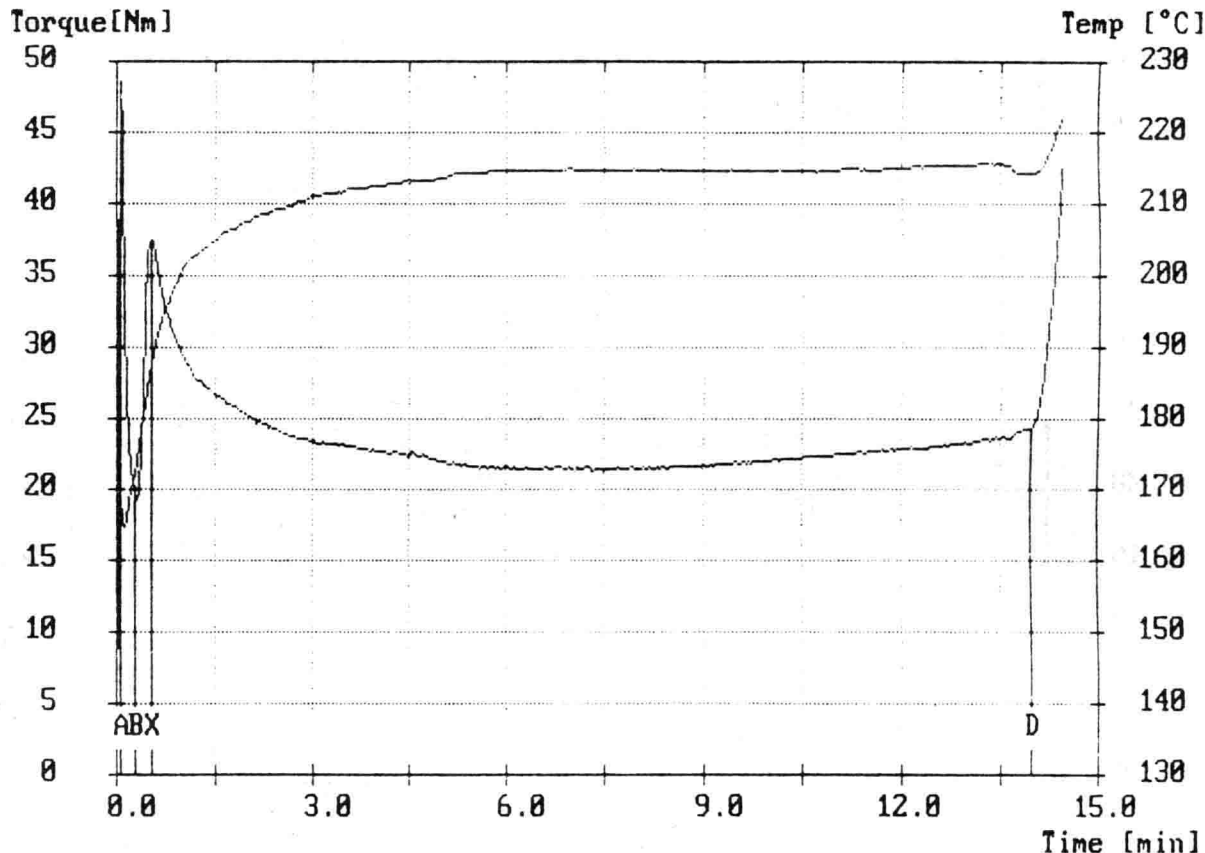
Figure 3 Computer print-out: Fusion behaviour

BRABENDER

Data-Processing Plasti-Corder PL2000 and Mixer Measuring Head
Heat & Shear Stability / 0.6.7

Test-Conditions

Order	: BRABENDER	Mixer-Temp.	: 200	[°C]
Operator	: EICKMEIER	Speed	: 75	[1/min]
Check-Date	: 16. MAY '88	Meas. Range	: 50	[Nm]
PL-Type	: 2000-3	Zero-Suppr.	: 0	[%]
Mixer-Type	: W 50	Damping	: 3	
Load. Chute	: MANUAL + 5 KG	Test-Time	: 15	[min]
Sample	: PVC	Sample-Weight	: 57	[g]
Additive	:	Codenumbr	: 2	
		Start-Temp.	: 196	[°C]



Value	Time [s]	Torque [Nm]	Stocktemp. [°C]
Load. Peak	4	48.5	168
Minimum	16	19.7	173
Maximum	32	37.5	189
Decomposition	838	24.3	215

Integration

- Load. Peak	to	Minimum	:	W1 =	2.5	E3 [Nm]
- Minimum	to	Maximum	:	W2 =	3.7	E3 [Nm]
- Load. Peak	to	End	:	W3 =	159.5	E3 [Nm]
- Specific Energy (W3/Weight)	:			W4 =	2.7	E3 [Nm/g]
- Decomposition Energy (TA to TD)	:			=	152.5	E3 [Nm]

Filename : A:\PL2000\MIXEVAL\DATA\TEST2

Figure 4 Computer print-out: Heat- and Shear-Stability

rapidly, or, depending on the material under test, the torque drops. In any case the heat- and shear-stability of the polymer mix can be clearly detected and evaluated. Here is a typical curve with characteristic values.

Besides the previous well-known characteristic values of the fusion behaviour, (Figure 4) from this diagram we may determine the degradation or decomposition time and, in some cases, also the degree of decomposition, which is the angle of the increasing or decreasing curve after the decomposition point.

In addition to these values the integration of the area under the curve represents the mechanical energy which we have put into this process according to this formula:

$$W = M \cdot T \cdot \omega \text{ (Nm)}$$

W = Energy in Nm
M = Torque in Nm
T = Time in min
 ω = angular speed in min^{-1} which is $2 \pi \cdot \text{RPM}$

Tests with different stabilisers, of course, will show different test results in respect to the torque and time values. Different stabilisers however do not only influence the heat- and shear-stability but they also influence the plasticising behaviour of the various PVC mixtures. Different processing or testing conditions, of course, also show varying results for the same material.

FLOW-CURING-BEHAVIOUR OF CROSS-LINKING POLYMERS

Tests with crosslinking thermoplastic materials show a very similar curve to those with thermosetting materials. Therefore the same test method can be used, and the same characteristic values can be evaluated.

Thermosetting materials are processed more and more under dynamic conditions, and therefore it is very important to test these materials under dynamic conditions also. No static test method will ever give sufficient results for any material being processed dynamically.

When processing a thermosetting material it has to be observed that there are two different reactions running simultaneously: the physical reaction of melting or plasticising and the chemical reaction of curing or hardening. These two reactions overlap each other and they cannot be determined individually.

To run these tests we attach a Measuring Mixer type MB 30 to a Plasti-Corder®. In order to obtain realistic test values, injection moulding compounds, for example, should be tested at a mixer temperature between the cylinder and the mould temperature of the production machine normally being used. Depending on the individual material, however, the evaluation may sometimes be easier under slightly higher or lower temperatures. The Plasti-Corder® should be set to a speed of 30 rpm, but this parameter can also be varied, of course, depending on the particular material.

Figure 5 shows a curve with the characteristic values. The first peak again is the loading peak followed by the decreasing torque that shows the melting or plasticising of the material which leads to the minimum point of the curve. After this, the torque increases again and this part of the curve represents the curing, hardening or crosslinking of the material in the Measuring Mixer. The torque climbs up to a maximum and because the mixer blades are grinding the cured material in the mixer, the torque drops after this rather steeply. Thus the material can be removed from the mixer after the test without problems.

The most important values of such a diagram are as follows. The minimum torque, which represents the viscosity of the melt, the reaction time between the loading peak and the maximum or any other selected point before the maximum point. The reaction time represents the total time from the beginning to the end of the process, which means that – within this time – the material must be melted, homogenised, pushed into the mould and cured.

The delay time at a selected level above the minimum represents the time during which the material is in the molten stage. Within this time the material must be injected into the mould.

Certain levels are recommended for evaluating and comparing the reaction and the delay time in a future DIN Standard.

The effects of varying speeds and temperatures on the test results are related to the varying processing conditions within an injection moulding machine system. The plasticising phase, the flow viscosity, the time of flowability of the melt, the curing phase and the curing speed including the total reaction time are made with this measurable test method. Of course, different testing and processing conditions show different results, and the various crosslinking materials show different reactions under the same processing conditions.

FLOW-CURING BEHAVIOUR OF ELASTOMERS

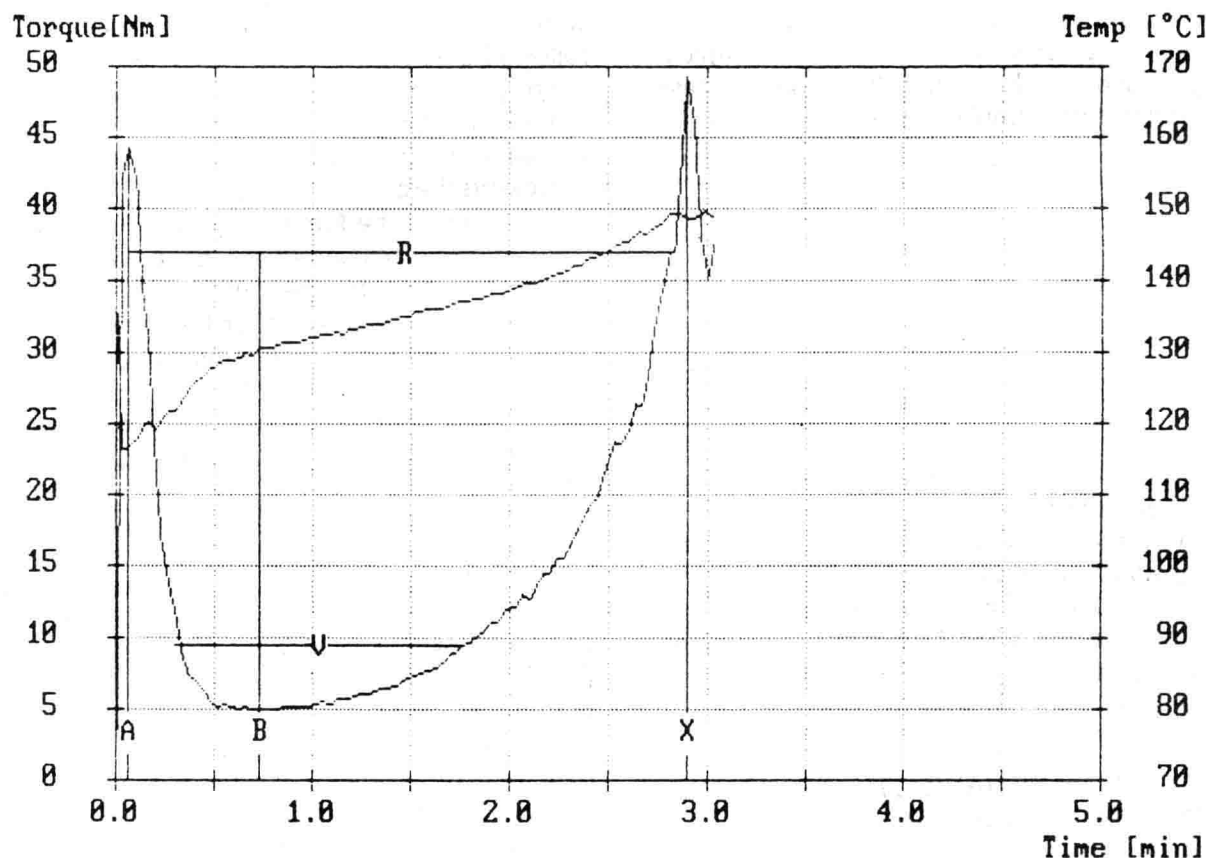
There are many different ways to process elastomers

BRABENDER

Data-Processing Plasti-Corder PL2000 and Mixer Measuring Head
Flow-Curing Behaviour of Crosslinking Polymers / 0.6.7

Test-Conditions

Order	: BRABENDER	Mixer-Temp.	: 140	[°C]
Operator	: EICKMEIER	Speed	: 30	[1/min]
Check-Date	: 16. MAY '88	Meas. Range	: 50	[Nm]
PL-Type	: 2000-3	Zero-Suppr.	: 0	[%]
Mixer-Type	: MB 30	Damping	: 3	
Load. Chute	: MANUAL + 5 KG	Test-Time	: 5	[min]
Sample	: THERMOSETTING	Sample-Weight	: 24	[g]
Additive	:	Codenumbr	: 1	
		Start-Temp.	: 137	[°C]



Value	Time [s]	Torque [Nm]	Stocktemp.[°C]
Load.Peak	4	44.3	117
Minimum	44	5.0	131
Maximum	174	49.3	149
Reaction	170	40.4	150

Integration

- Load.Peak	to	Minimum	:	W1 =	1.6	E3 [Nm]
- Minimum	to	Reaction	:	W2 =	5.1	E3 [Nm]
- Processing Time	TV	:	:	W3 =	1.7	E3 [Nm]
- Specific Energy	(W1+W2)/Weight:	:	:	W4 =	0.3	E3 [Nm/g]

Results

- Processing Time		TV =	88	[s]
- Melt Time	(TA - TB)	TS =	40	[s]
- Reaction Time	(TB - TR)	TR =	126	[s]

Filename : A:\PL2000\MIXEVAL\DATA\TEST3

Figure 5 Computer print-out: Flow and curing behaviour of crosslinking polymers

and there are different methods to test the flow-curing behaviour of elastomers.

Using a laboratory size Measuring Mixer with cam blades or Banbury blades, attached to a Plasti-Corder® torque rheometer again means a practice-related measuring method. A computerised system here is also of great assistance.

Natural rubber as well as synthetic rubber and many other different compounds can be tested this way. Besides testing the flow-curing behaviour of a compound, compounding with different additives can be carried out with such a measuring system, as can other standard tests such as Break-Down-Index or Black-Incorporation-Time. The mixer temperature and speed selection depend on the compound to be tested but they are usually in the range of approx. 80-150°C and of approx. 30-60 rpm, sometimes even higher.

The torque/time and stock temperature diagram in Figure 6 shows the loading peak and the flow behaviour of the compound with a torque minimum. Then the curing starts and the torque climbs up to a maximum after which the torque drops again because the mixer blades are grinding the cured sample. From the curing area of the diagram we can monitor the reaction of the curing additives in this particular compound and we can evaluate the curing speed first as an average value from any selected low torque level near the minimum, to any selected high torque level before the grinding starts.

It may be of interest in further tests to evaluate the curing speed at each point of the increasing curing curve. The computer can do this with the help of the software, and another curve representing the curing speed at each point of the torque/time diagram between the torque minimum and the torque maximum can be printed. The curing speed curve is the first mathematical derivation of the original flow-curing curve.

PVC DRY BLEND PLASTICISER ABSORPTION

Mixing of PVC dry blends has specific significance at the preparation stage. Plasticisers are added to the PVC powders in order to alter their properties. Type and quantity of the plasticising additives influence the properties of the product as well as the processibility of the individual compound.

Torque and time of the absorption process in the mixer are of utmost importance.

Further trouble-free processing of soft PVC is only possible so long as the flow of the powder mix is good enough to enable loading of the mixture into a screw type plasticising machine. Therefore the total plasticiser absorption behaviour of the

different types of PVC powders should be measured.

There is a large number of different PVC types available differing in molecular weight, particle shape and size, and thus these different PVC types absorb a certain quantity of plasticiser at different rates. The maximum plasticiser absorption capacity is also different when a dry mix with good flow is desired.

This means that the required mixing time varies and also the mechanical and thermal energy which has to be provided to produce a mix with the desired well-balanced characteristic of sufficient flow.

In order to identify the different factors influencing the production of a hot mix and to study the behaviour of different raw materials at varying mixing ratios a Planetari-Mixer type or a Sigma Mixer type is attached to a Plasti-Corder® to run the plasticiser absorption tests of PVC dry blends.

A specially designed blade carrying out a planetary movement rotates in the mixer bowl of the Planetari-Mixer which is designed for liquid temperature conditioning by means of circulation thermostat.

According to the ASTM Standard D-2396 the Mixer has to be heated up to 88°C for this particular type of test.

The measuring range selected is 0 to 2 Nm and the speed is set to 100 rpm.

The filling capacity of the mixing bowl is approx. 2500 cc.

Basic tests are normally performed with a total sample weight of 600 g (400 g PVC and 200 g plasticiser). The Planetari-Mixer, however, also permits the use of other sample quantities.

A thermocouple inserted from the bottom into the mixing bowl measures the material temperature during the mixing process.

When the Planetari-Mixer is heated up the PVC powder is poured into the bowl. Then the Plasti-Corder® is switched on and the powder is pre-heated in the running mixer for a period of 5 minutes. Following this the plasticiser is added to the PVC powder.

Figure 7 shows how the torque increases rapidly. The good mixing characteristic of the Planetari-Mixer provides good distribution of the plasticiser all over the surface of all PVC particles within a reasonable period of time. During this phase the compound is similar to a wet snow.

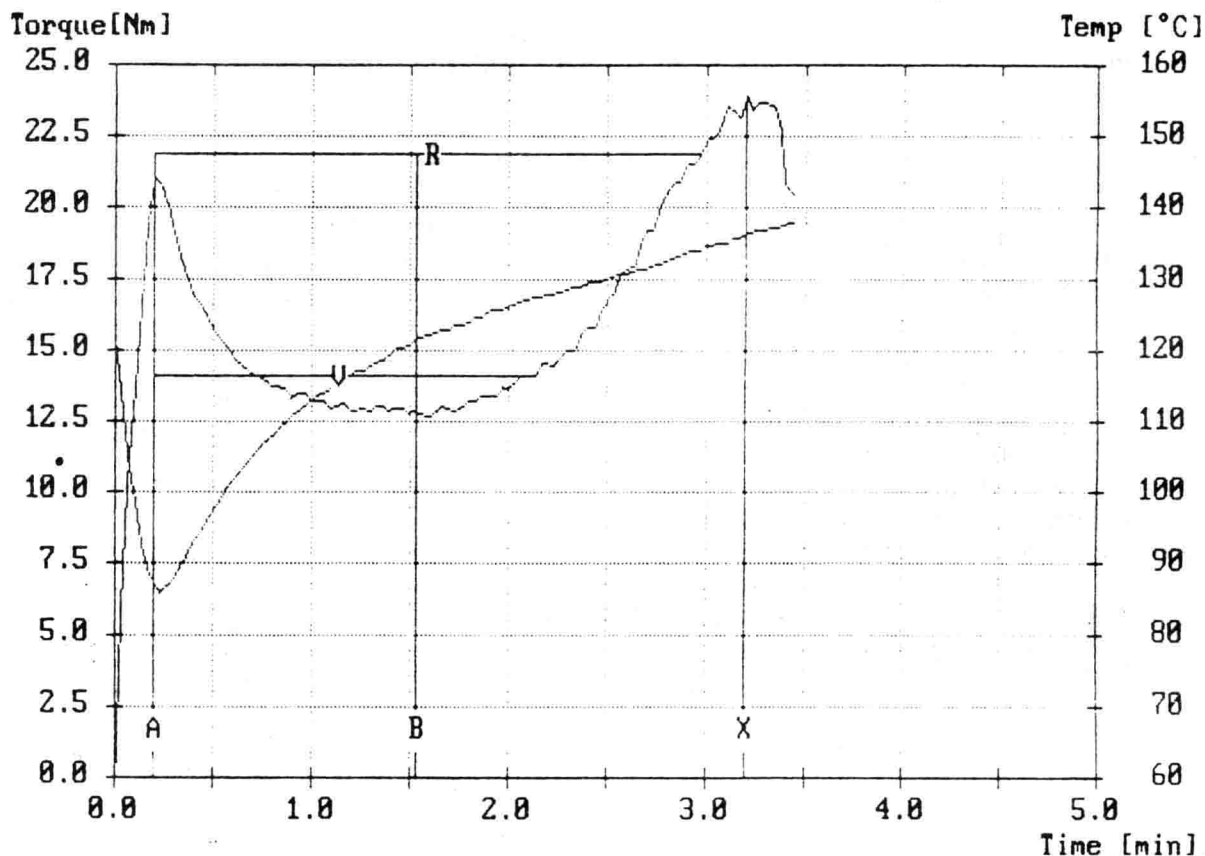
After the absorption procedure the torque drops rapidly to the so-called 'dry-point'. Absorption has taken place, this is followed by an eventual torque increase depending on the type of PVC and the type of plasticiser which gives some

BRABENDER

Data-Processing Plasti-Corder PL2000 and Mixer Measuring Head
Flow-Curing Behaviour of Elastomers / 0.6.7

Test-Conditions

Order	: BRABENDER	Mixer-Temp.	: 130	[°C]
Operator	: EICKMEIER	Speed	: 60	[1/min]
Check-Date	: 16. MAY '88	Meas. Range	: 25	[Nm]
PL-Type	: 2000-3	Zero-Suppr.	: 0	[%]
Mixer-Type	: N 50	Damping	: 3	
Load. Chute	: PRESS.RAM+5KG	Test-Time	: 5	[min]
Sample	: RUBBER	Sample-Weight	: 75	[g]
Additive	:	Codenumbr	: MK 2685	
		Start-Temp.	: 121	[°C]



Value	Time [s]	Torque [Nm]	Stocktemp. [°C]
Load. Peak	12	21.1	87
Minimum	92	12.8	122
Maximum	192	23.9	137
Reaction	178	21.7	135

Integration

- Load. Peak to Minimum	:	W1 =	7.3	E3 [Nm]
- Minimum to Reaction	:	W2 =	8.6	E3 [Nm]
- Processing Time TV	:	W3 =	9.9	E3 [Nm]
- Specific Energy (W1+W2)/Weight:	:	W4 =	0.2	E3 [Nm/g]

Results

- Processing Time	TV =	112	[s]
- Mastication Time	TM =	80	[s]
- Reaction Time	TR =	86	[s]
	TM + TR =	166	[s]

Filename : A:\PL2000\MIXEVAL\DATA\TEST4

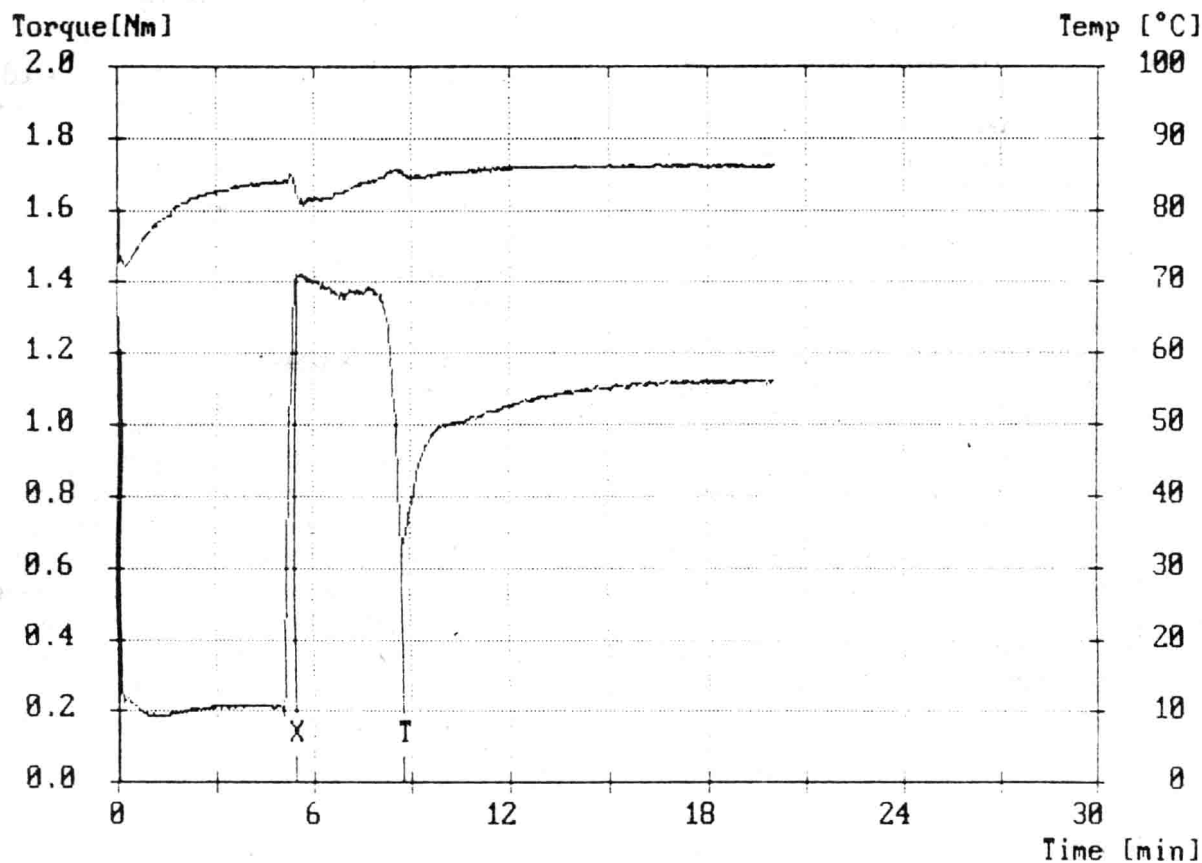
Figure 6 Computer print-out: Flow-curing behaviour of elastomers

BRABENDER

Data-Processing Plasti-Corder PL2000 and Mixer Measuring Head
Plasticizer Absorption / 0.6.7

Test-Conditions

Order	: BRABENDER	Mixer-Temp.	: 88	[°C]
Operator	: EICKMEIER	Speed	: 100	[1/min]
Check-Date	: 16. MAY '88	Meas. Range	: 2	[Nm]
PL-Type	: 2000-3	Zero-Suppr.	: 0	[%]
Mixer-Type	: P 600	Damping	: 3	
Load. Chute	:	Test-Time	: 30	[min]
Sample	: PVC	Sample-Weight	: 400	[g]
Plasticizer	: DOP	Plast.-Weight	: 200	[g]
		Start-Temp.	: 83	[°C]



Value	Time [s]	Torque [Nm]	Stocktemp. [°C]
Maximum	326	1.4	84
Dry Point	526	0.7	95

Integration

- Energy from start to 5 min	:	W1 =	0.7	E3 [Nm]
- Energy from 5 min to Dry Point	:	W2 =	3.0	E3 [Nm]

Results

- Dry Point	:	526	[s]
- Drying Time	:	226	[s]

Filename : A:\PL2000\MIXEVAL\DATA\TEST5

Figure 7 Computer print-out: PVC dry blend plasticiser absorption test