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THE DEVELOPMENT OF A TECHNIQUE FOR MIXING RUBBER COMPOUNDS IN AN INTERNAL MIXER TO A RHEOLOGICAL SETPOINT

by

B R MATTHEWS, ANCRT, MSc, MPRI

A Doctoral Thesis submitted in partial fulfilment of the requirements for the award of Doctor of Philosophy of the Loughborough University of Technology

February 1986

Director of Research: Dr J B Batchelor
Supervisor: P K Freakley, FPRI

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To my parents, I have received a lot of help
from many people along the way, but you were
there in the very beginning when I needed it
most.
I certify that I am responsible for the work submitted in this thesis. Further I certify that no part of this thesis has been submitted to this or any other University for the award of a higher degree.

References to other people's work have been clearly identified and credit given.

............................
B.R. Matthews
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It is always a pleasure to look back when the work is complete, and give credit to those persons who made it possible with their help and advice.

Because I had to design and then install the automation system on the internal mixer, I had to rely heavily on those persons with the required skills. Colin Lines was always ready to advise me on mechanical design matters and build precision components such as the carbon black hopper and the oil injector system. I had no knowledge of electronics but Terry Neale and Pete Patel in the Chemical Engineering Department steered me through this complex area, so that I was able to build a system to control the internal mixer from the computer.

In my early days I had never programmed a computer. I was fortunate to share an office with Suresh Patel who was very adept at programming. His patience and skill made it easy for me to develop the expertise I required in this area. Roger Walton who aided me with the modifications I made on the pneumatic system on the Banbury and to Ron Smith who was always willing to track down raw materials for me. I would like to thank my supervisor, Phil Freakley. Without his persistence the money needed to fund the project would not have been forthcoming, and therefore I would not have been able to have fulfilled my goal. He was always available for discussions when required, and not only gave excellent guidance but much needed encouragement when things did not always go to plan.

Richard Buxton in the Engineering Mathematics Department was able to steer me through the intricacies of experimental design and statistical analysis. He required all his skill and patience with me and I extend my thanks to him.

Also I would like to thank my colleagues from Avon, Dunlop, BTR, Francis Shaw and the SERC. The funding was vital but the interest, guidance and constructive advice given by each person involved was also equally important.
The research evaluated the means in which mild variations in the polymer properties could be modified during the mixing process so that the final mixed compound met the desired rheological parameters.

The initial work developed a model to measure the viscosity of the material in the internal mixer during the mixing process. The model assumed the mixer could be treated as two concentric rotary viscometers. The model had a rotor speed dependence which was caused by the assumption not being valid.

However, this led to the work continuing in a modified form. It was felt that at a given rotor speed and temperature the torque measured on the rotors would be proportional to the viscosity of the material. This was found to be correct and therefore work continued using this principle. Programs were developed for internal mixers with variable speed rotors and fixed speed rotors. The programs were evaluated using natural rubber with different viscosities and were found to be capable of modifying the polymers to obtain a final mixed compound to a target viscosity. The control program was also used to mix a series of batches at different processing conditions. The rheological properties of these batches were compared to a similar group mixed to a specification based on unit work. The result was the control system gave more rheologically uniform material.
CHAPTER 1
AN INTRODUCTION TO RUBBER MIXING AND THE OBJECTIVES OF THE RESEARCH

1.1 REQUIREMENTS OF THE RUBBER MIXING PROCESS

Elastomeric polymers are mixed with particulate fillers, liquid plasticisers, antidegradents; and a number of other ingredients which enable crosslinking to take place, and are known collectively as the vulcanisation system. The behaviour of the resultant rubber compound in further operations such as extruding, calendering, and in the final product, depends on both the constituents of the rubber compound and their treatment in the mixing operation. The work reported in this thesis concentrates upon the influence of mixing on the processing behaviour of rubber compounds.

The ASTM¹ definition of processability is:

"The relative ease with which raw or compounded rubber can be handled in rubber machinery"

However this is too narrow a definition, for it makes no reference to material uniformity, or quality of the components formed.

Norman and Johnson² offer a better definition of processability:

"In extrusion, it means the ease with which the compound can be fluxed, moved by the screw and extruded, together with dimensional stability of the extrudate; in injection moulding, it is the ease of flow and absence of excessive heat build up in the rubbers and gates, together with the speed of vulcanisation".

Elaborating on this definition one can say that for a mixed rubber compound to have acceptable processing characteristics, it must have
uniform and acceptable rheological, and crosslinking properties; where acceptable processing is considered to mean optimum productivity and desired quality levels.

The implications of acceptable processing properties are that components may be produced from downstream processes in the most efficient manner possible, resulting in savings of energy, labour and waste.

It is now possible to define quite clearly the requirements of the rubber mixing process. The process involves the combining of the components of a rubber compound, which range from high molecular weight elastomeric polymers to powders and oils, into an homogeneous mass. The resultant mixed rubber compound must have uniform and acceptable rheological and vulcanisate properties, so that a satisfactory product may be produced in the most efficient manner.

1.2 THE HISTORY OF THE RUBBER MIXING PROCESS

1.2.1 The Early History

The first manufacturer of rubber products appears to have commenced producing elastic bands at St Denis, near Paris, in 1803.

In the early days fillers were added to the natural rubber to reduce both the cost and surface tack or stickiness. The process involved dissolving the rubber in a solvent, such as turpentine, and then stirring in the fillers. The mixture was then placed in a receptacle of the desired shape and the solvent allowed to evaporate off.

However in 1819 Thomas Hancock became interested in rubber and this led, in 1820, to him setting up in business to produce elastic threads from raw rubber. This produced a large amount of trimming waste. To rework this material he developed a device called a "masticator". This was a hand powered device which consisted of a metal cylinder with spikes on the outside, which revolved within an enclosed chamber.
He found that the heat generated massed all the separate pieces of rubber into a lump. By 1821 he had built a larger machine using a horse to supply the motive power. This simple invention provided the basic concepts for the internal mixer; but it was not to be fully exploited for another 94 years.

1.2.2 The Development of the Internal Mixer

In 1915 while employed by Werner Pfleiderer, a Cornish man named Fernley Hope Banbury modified a rubber mixing machine, which prior to modification did not perform in a satisfactory way. He modified the rotors to the same basic configuration as the two winged version used in Banbury internal mixers today, and installed a metal floating weight.

This first modified machine was installed at the American Metal Cap Company in Brooklyn, New York. In 1916 F H Banbury obtained a patent in his name for the mixer. There is no doubt that the development of this machine made possible the massive growth in the rubber industry, required by the development of the automobile. The true significance of this invention can be appreciated when considering the output of a large internal mixer (batch size 445 kg), relative to a two roll mill; which was the main means of mixing at the turn of the century. The output is very dependent upon the type of compound, but typically one could expect 5000-6000 kg/hour from an internal mixer and 200-300 kg/hour from an 80 inch mill.

The other development which made the internal mixer so important, was the discovery, in 1904, by S C Mote of the reinforcing properties of carbon black. This material was not widely used until about 1910; and of course had to be mill mixed. This process is slow and extremely dirty, the air becomes filled with carbon black dust, which creates a hazardous and unpleasant working environment. The advent of the internal mixer made the mixing process both faster and cleaner. Examples of modern internal mixers are given in Figure 1.1.
FIGURE 1.1: SCHEMATIC DIAGRAM OF A BANBURY AND INTERMIX INTERNAL MIXERS
1.3. THE DEVELOPMENT OF MIXER CONTROL METHODS

In the preceding sections the requirements of the rubber mixing process, and the development of the internal mixer have been discussed. In recent years compound formulation and mixing, which had hitherto been considered to be an art, has started to develop into a science. The reason for these changes have been outlined in a number of publications\textsuperscript{5,6,7}. The main reasons are an increase in raw material costs, higher energy costs, and a need to reduce waste and improve quality.

Processability may be divided into two distinct categories: processability during the mixing process, and processability during the successive forming steps. It should be pointed out that these two categories are intrinsically linked. This thesis will only look at processability during the mixing process.

A considerable amount of research has been carried out in the field of processability in the internal mixer. It is generally recognised that the viscoelastic properties of raw polymers\textsuperscript{8,9} do have a significant effect on processability.

A mixing cycle is the process which begins with the individual components of a rubber compound, being introduced into an internal mixer, and ends when the mixing process is terminated with the properly mixed rubber compound being dumped from the mixer. This mixed compound is referred to as a batch. Traditionally the mixing cycle has been controlled by mixing time or batch temperature set-points. This means that the ingredients are either mixed together in the internal mixer for specific time periods, or the temperature rise resulting from putting work into the material is followed, and used as the criteria for deciding when the batch is adequately mixed.

However, in an effort to improve mixed compound uniformity, parameters other than time and temperature have been investigated as a means of monitoring the mixing process.
Improvements in process control were initially set in motion by the manufacturers of the materials handling systems used with internal mixers\textsuperscript{10}; and arose as a result of the demand for automatic materials handling. This in turn had been made possible by the development of electronics technology, and later by the production of microcomputers which could function in the dirty, and often hot industrial environment. These early systems did not measure any new parameters, but they did allow automatic weighing of ingredients and the automatic addition of these ingredients at the selected set-points in the mixing cycle. In terms of reducing error and thereby producing more uniform mixed rubber compounds, this was a significant step forward.

The disadvantage of a mixing cycle using batch temperature as the mixing criteria is that the temperature of the cooling water, the flow rate of the cooling water, and therefore the chamber temperature of the internal mixer, all affect the temperature rise of the material being mixed. Similarly, a mixing cycle which uses time as the mixing criteria is unsatisfactory, because the rate of the mixing process is affected by the temperature of the internal mixer, and by material variations.

By measuring the electrical energy demanded by the motor of the internal mixer during the mixing cycle, it is possible to monitor the mixing energy input to the material in the internal mixer. Power per unit time is the energy input, and the total energy input to a batch expressed in relation to a unit volume is known as 'unit work' and is normally expressed in kJ/m\textsuperscript{3}. This, of course, offers a new parameter for use as a criteria for a mixing cycle, and has been shown to represent a major step forward. The concept was first proposed by Van Buskirk et al\textsuperscript{11,12}, who saw the technique as a means of scaling from one size mixer to another. However, the concept was first utilised as a means of controlling a mixing cycle by Newell, Porter and Jacobs\textsuperscript{13} who developed an instrument which integrated the power input to the mixer with respect to mixing time during the mixing cycle on a continuous basis. They called the instrument a power integrator, and used it in conjunction with a data logging system to develop mixing specifications which
used energy as the mixing criteria. They reported\textsuperscript{14} that using this 
technique gave more uniform mixed rubber compound, when compared to 
mixing cycles using batch temperature as the mixing criteria, and 
significant savings in energy. One of the reasons for the improved 
uniformity of the mixed stock is that an internal mixer cools down 
when it is not mixing. Therefore delays caused by equipment problems 
or changing over to another compound cause a drop in the temperature 
of the substantial metal components of the internal mixer. The result 
is that a batch being mixed to a temperature specification will take 
longer to mix, as the cool Banbury will extract heat from the batch 
at a faster rate than occurs when the mixer reaches an equilibrium 
condition. This is known as the first batch effect, but in fact as 
many as five batches may be affected until the Banbury reaches equi­
librium. These findings of improved mixed compound uniformity at lower 
energy costs when using energy as the mixing criteria, compared to batch 
temperature control, have been confirmed by other authors\textsuperscript{15,16,17}. 
Although mixing energy control is a significant step forward the pro­
cess is still under an open loop form of control.

Raw material variations which are present are not adjusted for, and so 
the variability is carried over into the mixed compound.

1.3.1 Improved Open Loop Control

Werner and Pfleiderer\textsuperscript{18} have developed a control system which enables 
mixing to a specified energy input to be used in conjunction with con­
trol of batch temperature. One of the disadvantages of a mixing speci­
fication using energy as the mixing criteria is that large fluctuations 
in the dump temperature can occur. This means the heat history of the 
batches can vary, producing variable scorch times and curing charac­
teristics. Therefore the method proposed by Werner and Pfleiderer is 
an improvement over normal mixer control using energy as the criteria.
An energy vs batch temperature profile for a batch is determined 
experimentally using a "basic batch". This ideal temperature profile 
is filed in the memory of a microcomputer. As a batch is mixed the batch
temperature is compared to the temperature profile and the microcomputer will, in the case of a variable speed machine, adjust the rotor speed and/or ram pressure to correct deviations; and in the case of a fixed speed machine will adjust the ram pressure.

Although this is a further step forward it is still not a closed loop interactive system, for it makes no adjustments to the mixing cycle to account for material variations.

1.4 SUMMARY OF PRESENT CONTROL SYSTEMS

In the preceding sections an effort has been made to describe the advances in mixing techniques to date. The control systems dealt with have all been open loop systems. They monitor mixing energy and batch temperature, which are machine parameters, but do not measure the characteristics of the batch being mixed, or base control actions on deviations from expected material characteristics. This of course assumes that the raw materials used are uniform in their physical and chemical characteristics, and therefore have no affect on an individual mixing cycle.

The work reported in this thesis describes the development of a control system based on a measure of the changes in flow behaviour of a batch of rubber compound during mixing.

1.5 OBJECTIVES OF THE RESEARCH

To develop a process control system which can:

a) Produce batches of mixed rubber compound within reasonable limits of rheological behaviour by making compensations for mild variations in raw material properties, and mixer conditions.
b) Impart a specified heat history to the vulcanisation system.

c) Control the batch temperature on variable speed machines.
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CHAPTER 2
THE THEORY AND PRACTICE OF INTERNAL MIXING

2.1 REVIEW OF INTERNAL MIXERS WHICH ARE COMMERCIALLY AVAILABLE

There are two basic types of internal mixers in use today, the major difference being in the rotor geometry. The oldest type of rotor configuration is the tangential rotors as developed by F H Banbury. This comes in two and four wing versions as illustrated in Figure 2.1(a) and (b). The tangential configuration is also used by Werner and Pfleiderer.

The other major rotor configuration is the intermeshing system as developed by Francis Shaw and illustrated in Figure 2.1(c).

A study of the relative merits and problems associated with each type of mixer has been made by various authors^2,3,4,5,12.

To obtain equivalent batch sizes with a mixer fitted with intermeshing rotors, compared to a mixer fitted with tangential rotors, one must increase the chamber size by a factor of 1.65^12.

This is due to the difference in geometry of the mixers, and the volume occupied by the rotors. Also mixers with intermeshing rotors function at lower fill factors than comparable machines with tangential rotors. This can be a disadvantage for mixers with intermeshing rotors, since the initial cost of a machine is related to the chamber size. Therefore the initial capital expenditure for an intermeshing machine may be higher.

However, there are also some very positive results of the higher metal surface area of intermeshing machines. The most obvious is better heat exchange during the mixing process, and therefore lower batch temperatures. This of course is critical when mixing compounds that already contain the curatives, since the cure system is activated at a crucial temperature.
FIGURE 2.1: ROTOR CONFIGURATIONS FOR THE MAJOR MANUFACTURERS OF INTERNAL MIXERS
It may be possible to mix the rubber compound in one operation on a mixer using intermeshing rotors, which would not be possible on a machine fitted with tangential rotors, due to the higher batch temperature.

It is also claimed that mixers with intermeshing rotors give better thermal homogeneity in the batch being mixed than with mixers having tangential rotors. A rather interesting point is that mixers using tangential rotors have faster material feeding and discharge characteristics than mixers with intermeshing rotors.

One might expect the nogs on the intermeshing rotors to pull the material into the mixing chamber, while some slippage could be expected with tangential rotors. However this does not appear to happen.

The rate of filler incorporation is generally faster with the tangential rotors.

Comparing the two and four winged versions of tangential rotors, it has been found that, although the four winged rotors reduce the available volume in the mixing chamber, they can be used at higher fill factors. They also have increased mixing efficiency, and therefore can give higher output from a mixer than two winged rotors. However this could mean that higher batch temperatures result for four winged rotors compared to two winged rotors, unless the increased surface area gives better heat transfer.

Therefore it can be stated that there are very specific areas where mixers utilising intermeshing or tangential rotors have an advantage over the other. The machines equipped with tangential rotors are preferable where a high machine efficiency factor is important. This means exploitation of the fast feeding, discharge and filler incorporation characteristics of the machine, such as producing masterbatch or for rapid mixing cycles where temperature is not critical.
Intermeshing machines should be used where heat generation is a problem and lower batch temperatures are desirable. These properties can be exploited with certain types of compounds to reduce the number of steps in a mixing sequence, and thereby obtain greater output of final mix stock with lower labour and energy costs.

Whilst discussing commercially available mixers, there are two other types which at this time are not widely used, but at some time in the future could gain more acceptance. The first, which is illustrated in Figure 2.2, is the Rutilal Monomix. As can be seen from Figure 2.2 the rotor consists of a helical screw arrangement which moves the material from the centre and out to the chamber walls.

The claims made for the machine are that it mixes faster and more homogeneously than twin rotor mixers.

The other machine which is commercially available is the Schiesser IMI Mixer. As can be seen from Figure 2.3 the mixer has a unique action. It consists of a mixing disc or head into which a series of slots have been cut. This mixing head is attached to a shaft which rotates, and moves the head backwards and forwards within a cylindrical mixing chamber. This means the compound is forced through the slots of the mixing head as it moves forwards or backwards. Turbulence created by the rotation brings about a blending action. It is claimed that this mixer can achieve faster, more homogeneous mixing than other designs. However, it is difficult to see how shear stresses of a sufficient magnitude to make it effective can be possible while avoiding excessive equipment downtime for repairs.

2.2 DISCUSSION OF OPERATING VARIABLES AND THEIR EFFECTS ON THE MIXING PROCESS

2.2.1 Ram Pressure

The function of the ram is two-fold. Firstly it seals the mixing chamber and thereby keeps the ingredients in. The volume of a compound decreases as the various ingredients are incorporated, therefore forcing
**FIGURE 2.2:** RUTILAL MONOMIX

**FIGURE 2.3:** SCHIESSER IMI MIXER
the ingredients from the throat into the chamber during the initial mixing period is the second function of the ram. As can be seen a high pressure on the ram during this early stage is extremely beneficial, for it will speed up the process of pushing all the ingredients into the mixing chamber and reduce the number of voids. This in turn will generate the high shear stresses necessary to allow the incorporation process to start. However as the volume decreases it is desirable to reduce the ram pressure, so that the ram merely contains the batch in the mixing chamber. This allows greater rubber to metal contact and so improves heat transfer; and secondly it allows better flow of the batch in the mixer. Improved flow produces better mix homogeneity and better thermal homogeneity.

The effect of ram pressure has been quantified by a number of workers in the field\textsuperscript{1,8,10,11}.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure2.4.png}
\caption{Power Consumed at Different Ram Pressures}
\end{figure}

a) Pressure 1.2 kg/cm\textsuperscript{2}
b) Pressure 0.58 kg/cm\textsuperscript{2}
Figure 2.4 is from the paper by Bebris and his co-workers\textsuperscript{46}. It illustrates two power profiles for the same compound mixed at different ram pressures. It shows that a doubling of the ram pressure produces a higher maximum power peak, while the mixing time is reduced by more than a half. However the resultant energy consumption is approximately equal.

Funt\textsuperscript{9} explains this by saying that increasing the ram pressure increases the contact force between the rubber and rotor surface. This has the effect of increasing the critical stress so that flow begins at a lower temperature. The result is effective mixing begins earlier in the cycle, and with the batch temperature being lower a higher power peak results.

Funt claims that as a result of the higher shear stresses, improved filler dispersion is obtained. This is confirmed by Wiedmann\textsuperscript{12} who showed that increasing ram pressure gave improved black dispersion, higher dump temperature and allowed higher fill factors. This data is illustrated in Figure 2.5.

**FIGURE 2.5: INFLUENCE OF RAM PRESSURE AND FILL FACTOR ON CARBON BLACK DISPERSION AND DUMP TEMPERATURE**
Comes\textsuperscript{8} looked at the effect of rotor speed and ram pressure on mixing time. He demonstrated that large reductions are made in mixing time with increases in ram pressure, up to a critical level. Above this critical level much larger increases in ram pressure are required to obtain additional mixing time reductions.

This work was confirmed by Whitaker\textsuperscript{11}, who also evaluated increased ram pressure with higher rotor speeds. He concluded that increasing rotor speed, decreased mixing time to a given dump temperature. Therefore increasing ram pressure produces a reduction in mixing time, higher power peaks but equal energy per batch, improved black dispersion, higher dump temperatures, and allows the use of higher fill factors.

### 2.2.2. Rotor Speed

Until recently it was only possible to have internal mixers that operated at either one or two preset rotor speeds. However, it has become possible, to vary the rotor speed continuously during the course of a mixing cycle by the use of a DC motor and a thyristor drive. This is becoming an option which many rubber manufacturers are including on new internal mixers. It is therefore important to examine the effects of rotor speed on the mixing process.

As is pointed out by Johnson\textsuperscript{5}, rotor speed has a direct effect on the shear rate and therefore the rate of mixing. The rate of mixing controls the rate of heat generation, and this along with the ability of the cooling system to extract heat from the batch, will determine the temperature rise of the batch. This has been confirmed by this worker and others\textsuperscript{12,14,15,8}. Certainly the first work with increased ram pressure and some of the earliest work, if not the first work, with increased rotor speeds was done by the Farrel Corporation\textsuperscript{1,8}. The work had been initiated at a customer's request to resolve a black dispersion problem, and a general need to improve output from the
mixers. The results were termed "high horsepower mixing", and they recognised the need to put as much work into the batch per unit time while keeping the temperature as low as possible. In a way this is a contradiction in terms and led them to search for more efficient means of cooling the mixers. This ultimately resulted in spray sides giving way to drilled sides.

As the rotor speed increases, the shear stress will increase, but since elastomeric compounds are essentially pseudo-plastic, the corresponding increase in the shear stress will be lower. In turn the increase in rotor speed will cause an increase in temperature with a resultant reduction in apparent viscosity. This in turn will tend to offset the increase in shear stress. This line of reasoning tends to support those who have reported no improvement in dispersion with higher rotor speeds\textsuperscript{11,17}. However Comes\textsuperscript{2} reports that particularly with softer compounds, increasing rotor speed does improve filler dispersion. It could be that with very soft compounds where temperatures increase are not as great, increasing rotor speed may be the only means of generating high enough shear stresses to give satisfactory dispersion. Other workers have also reported improvements in filler dispersion with increased rotor speed\textsuperscript{12,14,18,19}.

In summation, it may be stated that increasing rotor speed generates higher shear stresses which reduce mixing time, increase dump temperatures, and in certain situations, when soft compound is involved, may improve carbon black dispersion, but for most situations no improvement in dispersion is likely.

### 2.2.3 Fill Factor

The fill factor is a ratio of the actual volume of the total ingredients of a batch and the theoretically available volume in the mixing chamber. The critical nature of fill factor is well documented\textsuperscript{12,20,21,22,23,25,10,15}. At the beginning of a mixing cycle the
volume of the ingredients is much greater than later in the cycle. The bulk density of the carbon black has a big influence on this initial volume. The individual carbon black pellets contain 66-76% voids. The structure of the carbon black also contributes to the physical bulkiness of the particles. Thus the higher the structure of the carbon black the greater its bulk. As the cycle proceeds the polymer is occluded within these voids and the volume decreases. Initially part of the batch will remain in the throat of the Banbury, until such times as sufficient black is incorporated to reduce the volume and allow the ram to become seated. For this reason the consensus is that fill factors should be in the region of 0.6 to 0.8. There is definite evidence that oversize batches allow material to remain in the throat of the mixer and produce batches with poor uniformity and dispersion. While justifying the need for fill factors of less than 1.0, Palmgreen stated:

"The empty space provides room for the batch to be turned around and is necessary for good simple mixing action"

Confirmation of this was provided by Freakley and Wan Idris in the flow visualisation studies they carried out. They found the fill factor should be such that there was adequate material in front of the rotor tip to allow the generation of the high stresses required for the fracture of particle material aggregates. They also pointed out that the formation of voids behind the rotor tips appeared to be essential in causing the disordering of streamline flow patterns, and for promoting effective axial transfer of material. This formation of voids behind the rotor tips was found to be very dependent upon fill factor. When the fill factor was too high to allow adequate voiding to take place, very poor extensive and dispersive mixing took place.
The viscoelastic properties of the polymer are known to have a very significant effect on the selection of fill factor\textsuperscript{12}. The greater the elasticity of a polymer, the lower the fill factor and vice versa.

There are workers who have indicated that fill factors of unity or even slightly higher may be beneficial with certain synthetic polymers such as butyl\textsuperscript{24}. Freakley\textsuperscript{21} explained this by saying that polymers which fracture at low extensions such as butyl, EPDM and certain grades of nitrile rubber could be fractured behind the rotor tip. The formation of voids could promote this fracture process and therefore reduce mixing efficiency.

It is possible then that in these cases a higher fill factor could be advantageous.

Undersize batches also create problems because it becomes impossible to create the shear stresses necessary for efficient filler incorporation and dispersion to take place. Therefore ram pressure becomes ineffective, and slippage on the rotors may result giving longer mixing times and poor dispersion\textsuperscript{15}. Because of this it is not possible to keep the area in front of the rotor tip full, and mixing becomes inefficient\textsuperscript{21}.

In summary, fill factor is critical if efficient mixing is to be achieved. The normal range will be 0.6-0.8. The exact fill factor will be dependent upon the compound filler loading and elastomer type. The higher the loading the higher the fill factor, and the greater the elasticity of the polymer the smaller the fill factor. The ideal fill factor is that which gives the the shortest mixing time or the smallest between batch variation\textsuperscript{15}.

2.2.4 Cooling Water Temperature

The effect of cooling water temperature and the flow rate of the cooling water has been evaluated, but not as thoroughly as the other
operating temperatures. Therefore the subject is not so well understood, and consequently it is an area that in a manufacturing environment is often ignored. Most internal mixers are simply cooled by service water, the temperature of which is dependent on the ambient air temperature. The flow rate is often dependent upon the call for service water in other areas of the plant. This means that operating conditions in the winter, when the water is cold, will be significantly different to those in the summer when the water is warmer. A simplified equation for the energy balance of a batch is given below: \[ E_0 = E_1 + E_2 + E_3 \] (2.1)

where

- \( E_0 \) = total energy input
- \( E_1 \) = energy to the cooling system
- \( E_2 \) = energy losses by convection through the machine body, inefficiency in the gears, etc.
- \( E_3 \) = the energy remaining which raises the temperature of the batch to its dump temperature.

If one considers heat transfer from the batch to the cooling water the greater the thermal gradient, the greater the heat flow across that gradient. Therefore a change in the temperature of the cooling water, or flow rate of the cooling water, will either cause the batch dump temperature to be higher or lower than expected, or it will increase or reduce the time required to reach a given batch temperature. Therefore variability in cure rate and state of mix will result. The other variability caused by cooling water is the non-equilibrium conditions created when a run of one type of batch is complete, and the mixer cools off while the next run is set up. This results in the "first batch effect", which means that while the internal mixer is brought back to an equilibrium condition, more heat is extracted from the first few batches, and therefore these batches
will be overmixed\textsuperscript{27,15}. Therefore it would seem that significant improvements could be made in reducing mix time variability and batch-to-batch uniformity by having a closed loop cooling system on each internal mixer; which would be capable of either heating or cooling the water as required, to ensure that cooling was consistent regardless of climatic conditions.

Having established the need for better control of the cooling system it is necessary to consider what are the factors which determine the conditions the system should function at. It might seem reasonable from the preceding section that the lower the water temperature the better. It has been suggested\textsuperscript{9} that transfer of heat from the batch to the mixer is accomplished by the rotor tip forming a layer on the Banbury wall, with heat transferred from the layer to the cooling water via the chamber wall. The layer is then removed on the next revolution of the tip and is blended into the bulk of the batch. This means that the process is very dependent on tip clearance and that the rubber must adhere to the wall. This was confirmed by Ellwood\textsuperscript{2,16} who showed that wall slippage occurred at about 120°C. This gave ineffective mixing and cooling. Therefore there is a lower limit for the cooling water. The actual value for this lower temperature limit is no doubt dependent on the polymer type, rotor speed, and the actual compound. There are workers who suggest that cooling water should actually be warm\textsuperscript{13,28} and others have indicated the optimum temperature to be 50-80°C\textsuperscript{29}.

However other workers have indicated negative effects when using warm water for cooling\textsuperscript{11}.

The temperature dependence of polymers is given by an Arrhenius type relationship\textsuperscript{15}

\[ n_a = A \exp \left( \frac{E}{RT} \right) \]
where \( \eta_a \) = apparent viscosity
\[ A = \text{a constant} \]
\[ E = \text{activation energy} \]
\[ R = \text{gas constant} \]
\[ T = \text{absolute temperature} \]

From this it can be seen that viscosity drops exponentially with temperature. This will mean that as the viscosity drops so the shear stress will drop, and the ability to improve carbon black dispersion will be reduced.

Therefore, in summary, cooling water temperature and the flow rate are extremely important in the mixing process and should be controlled rather than allowed to operate at the prevailing conditions. Further the cooling temperature must be above a critical value below which slippage occurs.

It may well be advantageous to operate at some temperature above ambient room temperature providing the water flow rate is increased to allow adequate removal of heat from the batch.

2.3 MIXING THEORY - A DISCUSSION OF THE EFFECTS OF POLYMER AND FILLER PROPERTIES ON THE RATE OF MIX AND STATE OF MIX

2.3.1 Mixing Theory Overview

In the mixing process there are two basic types of mixing taking place, namely dispersive mixing and distributive or simple mixing. At any point in time during a mixing cycle, both of these mechanisms will be occurring. Which one predominates will be determined by the stage reached in the mixing cycle. An excellent definition has been given for dispersive and distributive mixing by Hindmarch and Gale\(^{30}\).
Dispersive Mixing: An operation which reduces the agglomerate size of the minor constituent to its ultimate particle size, e.g. compounding carbon black into polymer in an internal mixer.

Distributive Mixing: An operation which is employed to increase the randomness of the spatial distribution of the minor constituent within the major base with no further change in size of that minor constituent e.g. distributing black masterbatch or accelerator masterbatch throughout a compound.

These are excellent definitions which contain the precise essence of the mixing mechanisms, and are the only terminology that will be used throughout this dissertation.

The mixing process can be broken down into four distinct steps, as illustrated in Figure 2.6. The four steps are:

- Subdivision
- Incorporation
- Dispersion
- Distribution

Subdivision Stage

When first introduced into the internal mixer the rubber and filler, exist in two separate phases, and are pulled through the rotors and shredded. This is a necessary step because the polymer is normally cold, and in large blocks. This initial step serves to subdivide the polymer, making filler incorporation possible. The work input required to reduce the polymer size generates heat, which causes the apparent viscosity of the polymer to drop, such that it flows under the shear stresses created in response to the movement of the rotors.
FIGURE 2.6: SCHEMATIC OF THE ELASTOMER MIXING PROCESS
Incorporation Stage

After subdivision the filler is incorporated into the continuous rubber matrix, causing the viscosity to increase\textsuperscript{9}. The rate of this step is determined by the magnitude of the shear stresses. The higher the shear stress the more rapid the incorporation. The shear stress is related to the rotor speed, polymer viscosity, and temperature.

As the filler is incorporated, so heat is generated. The result is that on the one hand viscosity is increasing due to filler incorporation, and on the other is being reduced due to the increase in temperature.

Cotten\textsuperscript{14} states that:

"In general the incorporation process is the wetting of carbon black with rubber and displacement of entrapped air".

This is confirmed by Hess et al\textsuperscript{32} who states:

"During incorporation, encapsulation and wetting takes place as the polymer breaks into small pieces and seals in the carbon black".

At the end of the incorporation the majority of filler is present as pellet fragments in a rubber matrix\textsuperscript{5}. It has been suggested that the filler forms large aggregates\textsuperscript{14} and that rubber is occluded into these aggregates. The result is a decrease in batch volume.

Dispersion Stage

This stage involves dispersive mixing where the filler aggregates are broken down to a size less than 1 \(\mu\text{m}\)\textsuperscript{14}. The final level of dispersion is determined by characteristics of the carbon black, mixing
conditions, and polymer properties. Specifically what is required is the highest shear stress that is possible, so that effective rupture of the filler aggregates can be achieved. McKelvey has postulated that there is a critical shear stress, below which it is not possible to rupture the aggregates, and therefore improvements in dispersion become untenable. In his excellent review paper Johnson points out that:

\[ \text{Shear stress} = \text{Apparent viscosity} \times \text{Shear rate} \]

He also points out that the relationship of temperature on the viscosity of polymers is given by an Arrhenius type relationship. This means that the viscosity is reduced exponentially as temperature increases.

The other major factors influencing the viscosity of the compound are the molecular weight of the polymer, the particle size and structure of the filler, and the level of filler present in the compound. Since effective dispersive mixing requires a high shear stress, then it would seem that high rotor speed and high viscosity would be the solution. However high rotor speed can be counterproductive since it could increase the temperature to the point that the viscosity drops, resulting in a lower shear stress and therefore less effective dispersive mixing. One observation that can be made is that the more effective the heat transfer properties of the mixer, the higher the shear stress and the more effective the dispersive mixing.

Distribution Stage

Eventually a point is reached, when due to the reduction in viscosity, it is no longer possible to obtain the high shear stresses necessary for dispersive mixing. At this point distributive mixing becomes the dominant mode. As Freakley et al. demonstrated axial flow produces a blending action leading to a more homogeneous blend of the compound ingredients. This includes a better uniformity of the filler
throughout the mix as well as distribution of the cure system and other additives.

2.3.2 Effect of Polymer Viscoelastic Properties on the Mixing Process

To understand the mixing process better it is necessary to understand how polymers perform under differing processing conditions, and how this relates to the viscoelastic properties of the polymers.

Tokita and White\textsuperscript{35} define four different regions of mechanical behaviour for an elastomer on a two-roll mill. These regions are illustrated in Figure 2.7. These regions are created by increasing the temperature.

\begin{itemize}
  \item \textbf{REGION 1} - High elastic memory, will not enter nip
  \item \textbf{REGION 2} - Good tight band
  \item \textbf{REGION 3} - Tends to bag or crumble
  \item \textbf{REGION 4} - Becomes transparent and forms a fluid coat on roll
\end{itemize}

The significance of this work is that it may be applied to the rotor of the internal mixer\textsuperscript{36} as illustrated in Figure 2.8.

If the polymer behaves as in Region 1, it will be reluctant to pass between the rotor and the wall, making incorporation impossible. Region 2 is the optimum state, but in Region 3 the polymer would crumble on passing between the rotor and wall. Once in this condition the polymer particles would not coalesce and it would become impossible to generate the shear stresses necessary for efficient dispersive mixing to take place. In Region 4 mixing is possible, but highly unlikely because the temperatures necessary to achieve Region 4 would give a low viscosity. This in turn would produce
FIGURE 2.7: ILLUSTRATION OF PROCESSING REGIONS OF ELASTOMERS
(a) Region 2 - Behaviour with Satisfactory Flow Characteristics

(b) Region 3 - Behaviour with Crumbling Taking Place

FIGURE 2.8: ILLUSTRATION OF THE BEHAVIOUR OF RUBBER IN AN INTERNAL MIXER
low shear stresses and therefore very inefficient dispersive mixing. The result is that the only acceptable processing region is 2. Therefore what polymer properties produce processing characteristics as defined by Region 2? In the original work Tokita\textsuperscript{35} found that polymers with narrow molecular weight distribution gave the worst type of processing characteristics as defined by Region 3. White\textsuperscript{36} felt that polymers with too high an elastic memory would respond as a solid and resist the rotor in an internal mixer. The problem in Region 3 was related to small elongation to break in tensile tests. From this it appears that the processing characteristics of a polymer in the mixing process are related to its failure mechanism, and so the physical characteristics which affect the failure mechanism will directly relate to processability.

Along this line Tokita and Pliskin\textsuperscript{38} state:

"Polymers normally do not deform smoothly or flow steadily, but yield and rupture".

They concluded that molecular weight distribution was very influential in determining failure characteristics, and in particular, extension to break. The reason given is that polymers with broader molecular weight distributions exhibit more non-Newtonian behaviour i.e. the shear stress is less dependent on shear rate. Therefore polymers with a broader molecular weight distribution will experience a lower shear stress at the same shear rate, than a polymer with narrow molecular weight distribution. The other reason given is that the tear propagation rate of a polymer with a broad molecular weight distribution is slower than that for a polymer with a narrow molecular weight distribution due to its higher relaxation time.

Tokita\textsuperscript{38} proposed the use of a deformation index which was a ratio of the elastic to viscous component. This in conjunction with the extension to break, was used to define areas of good processability.
Figure 2.9 shows the plot of deformation index versus extension to break. Polymers with high elastic component and, therefore, a deformation index approaching 1, and with a low extension to break, are classified as "dry". These polymers would conform to Region 1 behaviour as earlier defined\(^{35}\).

The region with a deformation index reflecting a higher viscous component but still having low extension to break is classified as "cheesy". This would conform to Region 3. Therefore they concluded that for good processing characteristics narrow molecular weight distribution was unacceptable, and this has been confirmed by other workers\(^ {37,39,35,40}\). They also found that as the molecular weight distribution becomes broader, the deformation index, and the elongation to break increase. This is perfectly consistent with the four region view of polymer processing characteristics.
2.3.3 Carbon Black Incorporation Theory

As discussed earlier in Section 2.3.1 filler/rubber mixing takes place in three phases: incorporation, dispersion, distribution. Cotten\textsuperscript{14} sees the incorporation phase as:

"The wetting of carbon black with rubber and the displacement of entrapped air".

This reinforced the work of Boonstra and Medalia\textsuperscript{41} who concluded that in the incorporation stage the rubber was penetrating into the interstices between carbon black aggregates. Therefore in theory the higher the structure of the carbon black, the more rubber would be trapped in the voids of the carbon black. Cotten\textsuperscript{33} elaborates on this and claims that the incorporation phase is really two processes. In the first he claims the carbon black is encapsulated by the polymer but the interstices within the agglomerates are still filled with air, which gives a weak, crumbly composite.

The rubber is then forced into the interstices during the latter stage of the incorporation process. Hess et al\textsuperscript{32}, also see the incorporation stage as one of encapsulation and wetting which takes place as the polymer breaks into small pieces and seals in the carbon black. Nakajima\textsuperscript{13} has proposed a model which is consistent with the work of Tokita and White\textsuperscript{35}, and the work described in this section.

Mixing can only take place in Region 2, and that the important polymer criteria is the elongation characteristics. As the material passes between the rotor and the chamber wall it undergoes an elongation deformation. This is illustrated in Figure 2.10 which shows that once through the gap between the rotor tip and the chamber wall, the material is subjected to a lower stress, allowing the material to recover from the deformation. The deformation may or may not
FIGURE 2.10: FLOW LINES THROUGH THE GAP BETWEEN THE ROTOR AND CHAMBER WALL WITH THE RESULTANT DEFORMATION AND BREAKING OF RUBBER PARTICLES

FIGURE 2.11: SCHEMATIC REPRESENTATION OF COMMINUTION AND LAMINAR MODELS
involve failure of the material. This will be dependent upon the imposed strain and whether it exceeded the ultimate strain of the material. The incorporation process is therefore seen as one which involves two mechanisms. These are illustrated in Figure 2.11. The first mechanism involves a large deformation of the polymer, followed by a relaxation after passing through the clearance between the rotor tip and the chamber wall, which encapsulates the carbon black particles. The second mechanism involves the failure of the polymer into small particles, and the subsequent mixing with carbon black. Both of the processes are going on simultaneously, and the relative level of each mechanism is dependent on machine variables. These include the size of the clearance between the rotor tip and the chamber wall, and the rotor speed, which controls the deformation elongations the material will be subjected to, and on the elongation properties of the material.

After the incorporation stage the dispersion process involves a large deformation of rubber domains in order to create a stress high enough to break up the carbon black aggregates. For each type of compound there is a critical shear-stress level below which dispersion will not occur. The factors which affect shear stress are shear rate and the viscosity of the compound.

Shear rate is proportional to the rotor speed, therefore in theory the faster the rotor speed the higher the shear rate and the better the dispersion. However viscosity decreases exponentially with temperature, and since an increase in rotor speed means an increase in batch temperature, the result may be counterproductive.

2.4 DUMP CRITERIA

Until recently batches have been dumped on mixing time or batch temperature. The object of the dump criteria is to select the point at which a batch will be dumped, such that a batch
of acceptable quality is obtained, with good batch to batch uniformity. It is well established that the state of mix of a batch has a very significant effect on processing properties, such as extrusion rate and die swell\textsuperscript{38,32}.

The factors which affect the state of mix include polymer variations, such as molecular weight and molecular weight distribution\textsuperscript{38}, equipment variations such as cooling water temperature, and flow rate\textsuperscript{29}, and variation in the filler such as fines, pellet hardness etc.

All of these factors affect the mixing rate and therefore rate of rise of batch temperature. Therefore the use of mixing time and mix temperature as dump criteria can lead to significant variability in the state of mix. For this reason mixing to unit work was introduced\textsuperscript{44}. The advantage of unit work is that it is largely independent of equipment variables such as cooling water temperature, and the flow rate of the cooling water. This effectively ensures the first batch effect is eliminated\textsuperscript{27}. However it does not make any adjustments for material variability.

If the viscosity of the polymer varies, then the polymer with the highest viscosity will consume power at the fastest rate. This means that the unit work will be achieved with a lower total strain rate, and therefore a lower state of mix is possible, relative to another batch, utilising polymer of lower viscosity.

Therefore the ideal dump criteria is one which will vary the unit work until a fixed viscosity is obtained while maintaining a target dump temperature.
REFERENCES

30. R S Hindmarch and G M Gale, a Paper given to the Rubber Division, ACS, Philadelphia, 4-8 May (1982).


CHAPTER 3

ASSESSMENT OF THE STATE OF MIX

3.1 A REVIEW OF METHODS AVAILABLE FOR ASSESSING THE STATE OF MIX

There are many criteria that have been used over the years to assess the state of mix of rubber compounds. The methods used include, evaluation of rheological properties, assessment of vulcanisation characteristics, degree of carbon black dispersion and the measurement of certain physical properties.

Evaluation of Rheological Properties

In Section 2.3.2 the major effect that state of mix has on the viscous flow properties of a mixed rubber compound was discussed. The effect of polymer microstructure and carbon black dispersion on the processing properties of mixed rubber compounds is well established. Various instruments are available for measuring the viscous flow properties of mixed rubber compounds. They include the Mooney viscometer, the dynamic stress relaxometer, the Monsanto SRPT and the instrument used in this study, the TMS biconical rotor rheometer. Machines such as the Mooney viscometer operate at shear rates which are too low to relate to actual processing conditions. This means the relevance of such instruments is in question. The TMS rheometer may be operated over a range of shear rates and may be used to obtain stress relaxation measurements. This makes the instrument very versatile, being able to obtain basic viscosity measurements and a viscoelastic property which has relevance in certain forming operations.

Methods for Assessing the Degree of Vulcanisation

To obtain a uniform level of crosslinking throughout the vulcanised product, it is necessary to have a uniform distribution of the ingredients which constitute the vulcanisation system. The first instrument
for continuously measuring the cure-rate of a mixed rubber compound was called the Vulkameter and was developed by the Bayer Company in the 1960s. A similar instrument called the Curemeter was developed by RAPRA. The instruments measured the dynamic shear modulus of a heated rubber sample. This was achieved by having a small paddle move back and forth within the sample. The most widely used instrument for assessing level of crosslinking today is the Monsanto oscillation disc rheometer. This measures the torque on a biconical rotor which oscillates through an arc of 30°. The rotor is surrounded by rubber which is encased in a heated die. This is the instrument used to assess the level of crosslinking in this work.

Methods of Assessing Carbon Black Dispersion

Various methods have been used to assess the level of carbon black dispersion in a mixed rubber compound. They include electrical resistance, microscopic techniques, surface roughness analysis, and electron microscopy.

Electrical resistance is particularly sensitive to the level of carbon black dispersion since rubber has very poor electrical conductivity and carbon black has excellent electrical conductivity. As the degree of carbon black dispersion improves, so the electrical resistance drops. With the advent of the microcomputer it has become possible to obtain and process multiple-data points. This has enabled the development of microscopic techniques which evaluate a larger area and are therefore more statistically sound than previously. The method used in this work is a microscopic technique using dark field reflected light and is discussed in detail in Section 3.4.2.

Physical Tests Used to Assess State of Mix

Tensile strength has been used as a means of assessing the state of mix. Tensile properties improve with increased mixing time, up to a point, and this reflects the improvement obtained in the carbon
black dispersion. However the method can only be used for mixed rubber compounds containing a vulcanisation system and is a slow and laborious technique. A physical test which is particularly sensitive to carbon black dispersion is the abrasion test\textsuperscript{22,27,28}. Other physical tests which have been used to assess state of mix include hardness, tear and compression set.

\textit{Summary of Methods for Assessing State of Mix}

In this brief review of methods available for assessing state of mix, it can be seen that many methods exist. In this work the state of mix has been assessed in terms of viscous flow measurements, level of crosslinking and carbon black dispersion. In the following sections each of these areas is discussed in detail.

3.2 \textbf{RHEOLOGICAL PROPERTIES}

3.2.1 \textit{Basic Viscous Flow Relationships}

The word 'rheological' comes from the fusion of two Greek words, rhein meaning to flow and logos meaning the science of. So rheology is the science of flow and deformation of matter.

![Rheological property diagram]

\textbf{FIGURE 3.1}
Consider a polymer fluid contained between two parallel plates as shown in Figure 3.1. The bottom plate is fixed and the layer of molecules in contact with each plate is also fixed. The application of a force to the top plate produces a deformation.

The force per unit area required to produce this deformation is known as the shear stress $\tau$ and is given by

$$\tau = \frac{F}{A}$$

The deformation produced by this shear stress is known as the shear strain $\gamma$

$$\gamma = \frac{\text{Amount of displacement } S}{\text{Distance between plate faces } d} = \tan \theta$$

The rate of change of shear strain with respect to time is known as the shear rate $\dot{\gamma}$ and is given by

$$\dot{\gamma} = \frac{d\gamma}{dt}$$

For a Newtonian fluid the viscosity is given by $\eta = \frac{\tau}{\dot{\gamma}}$. In other words viscosity is a ratio of the shear stress to the shear rate, and for a Newtonian fluid, the viscosity is independent of shear stress. This is demonstrated graphically in 3.2a. However high molecular weight elastomeric materials do not act in a Newtonian manner. Most elastomeric compounds act in a non-Newtonian way termed pseudoplastic. This is illustrated in Figure 3.2b. In the region where the shear rate is low, the material may act as a Newtonian fluid, but at higher shear rates the viscosity decreases as the shear rate increases.
This means that when a viscosity is quoted for a non-Newtonian polymer, the shear rate, and temperature must be given. For this reason it is not an absolute viscosity measurement, but an apparent viscosity \( \eta_a \). This decrease in viscosity is not permanent, and is caused by the molecular chains being "uncoiled" from their equilibrium conditions, and being aligned to the direction of the applied stress. A log-log plot of \( \eta \) versus \( \gamma \) produces a linear relationship over most of the shear-rate range. The relationship only becomes non-linear when the shear rate is high enough to produce a rise in the temperature of the sample causing a reduction in the resultant stress.

Therefore a linear log-log relationship gives an intercept \( \eta_0 \), which is the apparent viscosity at 1 sec\(^{-1}\), and a slope with a dimensionless constant normally referred to as \( n \).

Within this shear-rate range where a linear relationship exists, the relationship of shear stress to shear rate is given by the power-law equation:
\[ \tau = \eta_0 \dot{\gamma}^n \]  

(3.1)

The power law equation is an empirically derived equation which has no theoretical basis. However, it is a convenient method of representing non-Newtonian behaviour.

One can see that if \( n = 1 \), the fluid is Newtonian. For pseudoplastic fluids \( n \) is always less than 1. The smaller the number the more non-Newtonian is the behaviour.

At any point on the curve the apparent viscosity is given by

\[ \eta_a = \frac{\tau}{\dot{\gamma}} \]  

(3.2)

or

\[ \tau = \eta_a \dot{\gamma} \]  

(3.3)

If equation 3.3 is substituted into equation 3.1 one obtains a version of the power law equation which allows the calculation of the apparent viscosity at any point on the curve i.e.

\[ \eta_a = \eta_0 \dot{\gamma}^{n-1} \]  

(3.4)

Having obtained the power law constant experimentally for a fluid in a given system, then a prevailing apparent viscosity can be related to a reference viscosity, by one of the forms of the power law relationship given below:

\[ \eta_{\text{prevailing}} = \eta_{\text{reference}} \left( \frac{\dot{\gamma}_{\text{prevailing}}}{\dot{\gamma}_{\text{reference}}} \right)^{(n-1)/n} \]  

(3.5)
\[ \eta_{\text{prevailing}} = \eta_{\text{reference}} \frac{\dot{\gamma}_{\text{prevailing}}}{\dot{\gamma}_{\text{reference}}}^{n-1} \] (3.6)

The other factor which affects the viscosity is temperature. For many polymers and particularly if the molecular weight is not excessively high, the temperature effect may be given by a simple Arrhenius type relationship:

\[ \eta = A e^{-(E/RT)} \] (3.7)

where \( E \) is an activation energy for viscous flow, \( A \) is a constant, \( R \) is the universal gas constant, and \( T \) is the temperature in absolute degrees.

For relating a prevailing apparent viscosity to reference viscosity, the equation may be modified as below:

\[ \eta_{\text{prevailing}} = \eta_{\text{reference}} e^{\frac{E}{RT} \left( T_{\text{reference}} - T_{\text{prevailing}} \right)} \] (3.8)

From equation 3.7 it can be seen that viscosity decreases exponentially as temperature increases. The reason for this is that increasing temperature causes a decrease in the Van Der Waals forces. This allows the molecular chains to move apart. The result is that when a stress is applied, the chains are able to move and slip producing a greater deformation.

Looking at this from another perspective, for the same deformation a smaller stress is required. If one considers the implications of this...
in relation to the mixing process, where high shear stresses are required for good dispersive mixing, increasing temperature can reduce mixing efficiency. Therefore the effect of temperature on viscosity is critical in the mixing process.

3.2.2 Viscoelasticity

If a material is perfectly elastic, then applying a stress would produce a deformation. The stress required to maintain the deformation would be identical to that used to produce it. For a perfectly elastic material the stress is independent of the rate of deformation. Therefore they are termed Hookean and are described by equation 3.9.

\[
\tau = G\gamma
\]  

(3.9)

where \( G \) is the static modulus of elasticity.

If the method is completely elastic then the process is totally reversible.

At the other end of the spectrum one has a viscous fluid, which requires no perceptible stress to maintain the deformation. In reality a static deformation with a viscous fluid does not exist. The process is irreversible, and energy cannot be stored in the viscous fluid or recovered. The work in this irreversible process is dissipated as heat. A perfectly viscous fluid is termed Newtonian, and the stress is proportional to the rate of deformation as given in equation 3.10

\[
\tau = \eta\gamma
\]  

(3.10)

where \( \eta \) is the viscosity of the fluid.
However real materials are not perfectly elastic solids, nor are they perfectly viscous fluids. Instead most polymers are a combination of the two, and therefore the resulting rheological properties are referred to as viscoelastic properties.

A major characteristic of polymers is the dependence of their properties on time. Consider a polymer sample, to which a constant stress is applied. A large initial deformation will be obtained when the stress is applied. However the deformation will continue to increase with time. This phenomena is known as creep.

Now consider a polymer which is instantaneously strained to some value, and held at a constant strain. There would be an initial peak stress which would gradually decrease with time. This is the phenomenon known as stress relaxation.

The viscoelastic behaviour of polymers is also very temperature dependent. The relaxation time of a polymer is given by:

\[ \lambda = \frac{n}{G} \]

where \( G \) = relaxation modulus.

The viscosity has a temperature dependence which is based on an Arrhenius type relationship and the modulus has a linear temperature dependence. This may be expressed as:

\[ \lambda = \frac{C_e \Delta E/RT}{G_0 \rho T} \]  \hspace{1cm} (3.11)
This means that as the temperature rises the viscosity will be reduced by a greater proportion than the modulus. This means the faster the relaxation and vice-versa. The same applies of course to creep.

It is useful when attempting to visualise the viscoelastic behaviour of a polymer, to use a model utilising the concept of dashpots and springs. The dashpot represents the viscous component with its flow, and damping characteristics, and the springs represent the reversible elastic component.

A model cannot represent polymer behaviour adequately, or completely, but they do serve a useful purpose. The simplest model, Figure 3.3a, is simply a Hookean spring in series with a Newtonian fluid dashpot. This is known as the Maxwell model and is useful for depicting stress-relaxation. The major disadvantage of the Maxwell model is that the dashpot is Newtonian and therefore is not valid for most elastomers which are pseudo-plastic. A solution is to use a Maxwell model with a power law dashpot as shown in Figure 3.3b.

The relaxation time is then given by

\[ \lambda = \frac{n\gamma}{G} \]  

(3.12)

This shows clearly the more non-Newtonian the polymer i.e. the smaller the value of n the shorter will be the relaxation time.

3.2.3 Measurement of Rheological Properties

The equipment used for determining the rheological properties of polymers is of two basic types. Those in which flow is caused by a pressure differential in the fluid, and those where the fluid is contained within a cavity, and a movement within this chamber, creates a relative motion.
(a) Maxwell Model

(b) Maxwell Model with Power Law Dashpot

FIGURE 3.3
The most important rheometers falling into the first category are the capillary rheometers. The second category is represented by the Mooney viscometer, the TMS biconical rotor rheometer, and the dynamic stress relaxometer.

Because polymers are non-Newtonian, the effect of shear rate on apparent viscosity is very significant. A necessary criteria for any type of rheological test equipment, is that it is capable of operating in the shear-rate range of the processing equipment that the polymer will be processed on. Figure 3.4 illustrates graphically the range of shear rates that compounds experience on various items of process equipment. The other criteria for a piece of equipment to simulate successfully the rheological properties of a polymer is that the temperature of the sample remains constant throughout the test. This is because the viscosity decreases exponentially, as temperature increases. Therefore the equipment must have excellent temperature control and, ideally, temperature generation during the test will be minimal.
Capillary Rheometers

There are many types of capillary rheometers, but Figure 3.5a is a schematic drawing of a basic piston type. The capillary rheometer was initially used for characterising the rheological properties of plastics, but today it is finding an increasing use in the characterisation of elastomers. A major reason for the usage in rubber compounds, is the wide range of shear rates over which materials can be evaluated i.e. 1 sec\(^{-1}\) to 1000 sec\(^{-1}\). A review of Figure 3.4 will show that this range is more than adequate to cover the shear rates generated in most production operations. However there are still problems that can arise, as a result of the assumptions which have to be made in order to derive the equations for shear rate and shear stress at the capillary wall. It is assumed that there is no slip at the wall, isothermal conditions exist, and the material flows at a constant rate. The assumption of no wall slip, in light of the work by den Otter\(^8\), may be suspect. It is also necessary to make corrections for the viscoelastic effects which take place at the entrance and exit of the capillary. These were studied by Bagley\(^9,10\) who developed the necessary corrections.

The major disadvantage of capillary rheometers is the time required to ensure total thermal homogeneity. This can be 20 minutes, or longer and therefore makes the test very time consuming. However when rheological properties are required at shear rates in excess of 100 sec\(^{-1}\), the capillary rheometer is the only means of obtaining them.

Mooney Viscometer

The Mooney viscometer, Figure 3.5b, consists of a grooved rotor, which rotates in a heated cavity. The torque generated on the rotor is recorded with respect to time. This produces a torque at some given time, such as four minutes, and is expressed as the Mooney viscosity of a compound; and would be reported in conjunction with the rotor size, preheat time and temperature. For instance a test using the
FIGURE 3.5

(a) Capillary Rheometer

(b) Mooney Viscometer

(c) TMS Rheometer

(d) Monsanto ODR
large rotor at $100^\circ C$, and using a one minute preheat, and a four minute test would be expressed as:

$$ML_{1+4} @ 100^\circ C$$

The test is very simple to use and, because of the low shear rate (1.6 sec$^{-1}$), heat generation is low and temperature control is good. A major drawback is that, due to the geometry of the rotor, the shear rate varies continuously across the radius of the rotor. Therefore the shear stress also varies across the width of the sample. Hence, the use of the term Mooney viscosity is inaccurate, since it is really a torque that is measured. The Mooney "viscosity" test is probably the most widely used rheological production test. The reason is the simplicity, speed, and reproducibility of the test. It is a useful test for ensuring the correct levels of polymer, carbon black and oil are present in a batch of rubber compound.

However there is still considerable doubt whether Mooney torque relates to polymer processing operations, such as mill handling, mixing or extrusion. The lack of correlation is thought to be due to the shear rate in the test (1.6 sec$^{-1}$) being much lower than those experienced in most factory processes.

**TMS Rheometer**

The TMS rheometer is a modification of the Mooney viscometer. This means the rheometer has the advantages of excellent temperature control as does the Mooney viscometer. The relatively large heating platen size, relative to the dies, acts as a heat sink which stabilises the die temperatures, and, as with the Mooney viscometer, the sample is contained within the confines of the two dies, which ensures a sample of uniform dimensions. Figure 3.5c illustrates the principal features of the TMS rheometer and highlights the differences
between the Mooney viscometer and the TMS rheometer. The major difference is that the rotor is a biconical geometrical configuration as opposed to a flat disc shaped rotor. The importance of this is that the shear rate is approximately constant over much of the surface of the rotor. The rotor is designed in such a manner that rotor speed in rev/min equates directly to shear rate in s⁻¹.

The other important feature is that the sample is transferred from a heated chamber above the dies, by the action of a piston into the test chamber. This means that a sample of a uniform size and temperature is injected through heated flow chambers into the die. This injection action produces blending, which results in improved thermal homogeneity. The pressure on the die is not controlled by the pressure to clamp the dies together, but rather by the pressure of the piston. This results in a thermally homogeneous sample of consistent size being contained at a controlled hydrostatic pressure. Therefore the variables that can give poor repeatability of tests in the Mooney viscometer are controlled. The machine can be operated at a range of shear rates. Shear stress is calculated from the torque by equation 3.13

\[ \tau = \frac{\text{Torque}}{1.333 \pi r^3} \]  

(3.13)

However there is a contribution to the total torque by the Couette flow between the edge of the rotor and the cavity wall. Assuming Newtonian flow this is given by

\[ T_e = 2\pi r^2 y \tau \]

where \( r \) = rotor radius and \( y \) = depth of rotor at edge.

The modified equation for shear stress is given by:

\[ \tau = \frac{\text{Test torque}}{1.333 \pi r^2 (r+.66y)} \]  

(3.14)

This is the correction used in this work.

It is possible to test a sample over a range of shear rates and obtain the associated shear stresses. Elastomers for the most part conform to the power law equation

\[ \tau = \eta_0 \gamma^n \]
and so a plot of log $t$ against log $\gamma$ will yield a linear relationship, the slope of which is given by the power law index $n$, and the intercept is the viscosity at 1 sec$^{-1}$.

The major drawback of the TMS is that the higher the shear rate, the greater is the heat generation. This is critical because the poor thermal conductivity of polymers ensures that the temperature of the sample will rise. Since the viscosity of polymers decreases exponentially with an increase in temperature, even a slight temperature rise generates significant errors in the measured shear stress. This means the instrument must be operated at relatively low shear rates i.e. less than 10 sec$^{-1}$, or a means must be developed to correct for increases in temperature of the sample. It is also possible with the TMS rheometer to arrest the rotor motion and follow the decay of the shear stress. This capability of measuring stress-relaxation is important for it is felt to be more indicative of processability than a simple viscosity. However it is unlikely that the key to processability will be discovered until it is possible to determine accurately the viscous and elastic components of the flow behaviour of polymers, and relate them to the molecular structure.

**Other Instruments**

There are several other instruments worth mentioning which have practical significance for evaluating the processing characteristics of mixed rubber compound. The first instrument is the stress relaxation processability tester$^{13}$ which is produced by Monsanto. The second instrument is the dynamic stress relaxometer$^{14,15}$ which has been developed by B F Goodrich. As the names imply both instruments evaluate the stress-relaxation properties of the material under evaluation. This indicates the importance placed on stress relaxation as a means of predicting processing behaviour in forming operations such as milling, and extruding.
3.3 CROSSLINKING BEHAVIOUR

3.3.1 Practical Background

The modern vulcanisation system consists of three components namely a crosslinking agent, an organic accelerator and an activation system.

The most common crosslinking agent is sulphur and will be the only system discussed. The activation system is normally zinc oxide and stearic acid. There are a complete range of organic accelerators which vary from extremely fast acting in the case of zinc dithiocarbamate to delayed action for N-oxydiethylene-2-benzothiazole sulphenamide.

3.3.2 Theory of Sulphur Vulcanisation

Unvulcanised rubber may be visualised as a plate of spaghetti. It consists of a series of high molecular-weight chains, which are able to move freely in relation to one another. The only restrictions to flow are the physical entanglements of one molecular chain to another. This means that the material cannot retain a permanent shape, but will deform when a stress is applied, and will not return to the original shape when the stress is removed.

However if the molecular chains are joined together by rigid links, similar to rungs in a ladder, then the chains cannot move independent of one another. This process is called vulcanisation and the crosslinks are sulphur. The crosslinks are produced by forming the material into the desired shape, and then applying heat to produce the sulphur crosslinks. Once cured in that shape the product has an elastic memory, and will deform when a force is applied, but will return to the original shape when the force is removed.
Sulphur crosslinks exist in a variety of forms as illustrated in Figure 3.6. Before vulcanisation can occur a zinc-accelerator-sulphur complex is formed\textsuperscript{16}. The rate at which this complex forms controls the length of the induction period prior to crosslinking. This induction period is critical for it determines how much time a manufacturer has to convert the batch stock into a form ready for moulding. The length of time initially available is a function of the accelerator system being used, and the heat history during the mixing process. However the formation of this zinc-accelerator-sulphur complex is referred to as the scorch time of the batch stock, and is both time and temperature dependent.

Morrell\textsuperscript{16} refers to this complex as a zinc perthio-salt, which takes the form $X S_x Zn S_x X$, where $X$ is a group derived from the accelerator (e.g. dithiocarbamate or benzthiazyl groups).

\begin{figure}[h]
\centering
\includegraphics[width=0.8\textwidth]{figure3.6}
\caption{Forms of Sulphur Crosslinks}
\end{figure}
FIGURE 3.7: A PROPOSED MECHANISM FOR THE FORMATION OF SULPHUR CROSSLINKS

\[ \text{ZnO} + \text{S} + \text{A}_{\text{cc}} \xrightarrow{\text{Heat}} \text{X}_x\text{ZnS}_x\text{X} \]

\[ \text{X}_x\text{ZnS}_x\text{X} + \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \xrightarrow{\text{Heat}} \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]

\[ \xrightarrow{\text{X} + \text{ZnS} + \text{HS}_{x-1}\text{X}} \]

\[ \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]

\[ \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]

\[ + \]

\[ \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]

\[ \text{Additional heat} \]

\[ \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]

\[ \text{X}_x\text{ZnS}_x\text{X} + \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \xrightarrow{\text{Heat}} \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]

\[ \xrightarrow{\text{X} + \text{ZnS} + \text{HS}_{x-1}\text{X}} \]

\[ \sim \text{CH}_2 - \text{C} = \text{CH} - \text{CH}_2 \sim \]
This complex then reacts with the rubber hydrocarbon, extracting a hydrogen atom from the polymer, normally from a methylene group in the \( \alpha \)-position to the double bond. The result is that the complex breaks up with part attached to the polymer, and the other half forming a perthio-accelerator group. It is likely that this perthio-accelerator group then reacts with more zinc to form a zinc-accelerator-sulphur complex and repeats the previous step. The partial crosslinks thus formed then extract a hydrogen atom from another polymer chain, and form a complete crosslink and the accelerator group is split out. This process is summarised in Figure 3.7.

As can be seen an allylic shift occurs which moves crosslinks which were originally at positions 4 and 5 to position 2 or 3. It can be seen that as the process continues so complexes are regenerated with lower sulphur contents. This results in crosslinks with fewer sulphur molecules. Also the initial polysulphidic crosslinks on further heating lose some of the sulphur molecules which combine with the accelerator from step 3 and react with zinc oxide to form more crosslinks. Therefore the process is an extremely complex one and simply continues to regenerate itself until the sulphur or accelerator is exhausted.

### 3.3.3 Effect of Crosslink Density

A compound which is highly crosslinked with predominantly monosulphidic crosslinks is termed "efficiently crosslinked". This type of system is produced using a low sulphur level, but high accelerator levels. A compound which is predominantly monosulphidic is thermally stable, but because the crosslinks are so inflexible, such a compound has poor flex fatigue properties. It is therefore not acceptable for use in dynamic applications such as a tyre sidewall, but would be satisfactory in a static application such as bridge bearings. The other end of the scale is a cure system which is mainly polysulphidic crosslinks. Such a cure system is produced by using high levels of sulphur and low accelerator levels.
However this type of crosslink system is liable to further decom­position, and as such has poor ageing properties, but excellent flex characteristics, because the polysulphidic crosslinks have a greater degree of flexibility and so do not break when the material is dis­torted.

The normal system used is one which contains a range of monosulphidic crosslinks, and polysulphidic crosslinks. Such a system is known as a semi-efficient cure system.

Figure 3.8 illustrates the affect of crosslink density on various vulcanisate properties. From this it may be seen that properties which are related to energy to break, such as tear strength, and fatigue life show an initial improvement with a slight increase in crosslink density, but then a rapid deterioration as the network develops more monosulphidic crosslinks.

On the other hand hysteresis, and the related property dynamic modulus, both improve with increased crosslink density. This of course is exactly what one would expect. Hysteresis is a measure of the deformation energy which is converted to heat, primarily by the viscous component. In other words a compound which has good energy absorbing characteristics, and poor rebound properties will have high hysteresis. However as the crosslink density increases, so the relative movement of the molecular chains becomes increasingly restricted. This means the ability to deform the material, and have a viscous component to absorb energy is reduced. Therefore the hysteresis properties improve with crosslink density, and with it the hardness will increase, the friction coefficient will decrease, and permanent set will decrease.
FIGURE 3.8: THE EFFECT OF CROSSLINK DENSITY ON VULCANISATE PROPERTIES
3.3.4 The Need for Assessing the State of Cure

In the preceding sections the process and mechanism of vulcanisation have been discussed. In those sections it was tacitly assumed that all the ingredients of the vulcanisation system were distributed in an homogeneous manner throughout the mix. This is not always the case, and today with equipment that can follow the vulcanisation process, this non-homogeneity of the cure system can be seen. As has been seen the relative levels of sulphur to accelerator determine the type of crosslink structure formed. In the case of very poor cure-system dispersion, it may be seen that the crosslink density could vary significantly throughout the sample. Depending on the service conditions experienced by the product this could lead to premature failure. We have seen in the discussion on the vulcanisation mechanism, that the initial formation of a zinc-sulphur-accelerator complex is the factor which determines the induction period of scorch time. The importance of this for producing compounds, which provide sufficient time to perform the forming operations has been discussed.

Prior to the development of test procedures which allow the degree of crosslinking to be followed, it was normal to cure a series of tensile sheets at a range of cure times, and obtain an ultimate tensile strength, and 100% or 300% modulus for each cure time. This data would then be plotted, Figure 3.9, and a determination made as to the correct cure time. For production control other methods simply involved curing a hardness disc and testing it. This method indicated whether the batch would vulcanise, but gave no indication of processing safety or cure-system dispersion.

3.3.5 Methods for Assessing State of Cure

Mooney Viscometer

This instrument has been discussed in the earlier section on the measurement of rheological properties. However it is widely used to
determine the scorch time of a compound. This is done by operating the machine at 120°C or 145°C and obtaining a torque-time curve. Figure 3.10a illustrates a typical Mooney viscometer plot. As can be seen the compound initially undergoes a drop in viscosity due to thixotropic effects before stabilising at some minimum value. When the induction period is complete, the curve will start to rise. The time taken for a 5 point rise is referred to as the scorch time. The longer the scorch time the greater is the processing safety. The test is normally run at the temperature which conforms most closely to those experienced at the extruders, mill lines etc.

Monsanto Oscillating Disc Rheometer

Figure 3.5d shows the salient features of this instrument. Without a doubt it is the most widely used machine for determining the vulcanisation characteristics of rubber compounds in the world today. It consists of a biconical rotor, which is contained within a heated chamber. The rotor oscillates through an arc of 1, 3 or 5°, and the heated chamber is split in the middle with the top half being raised.
FIGURE 3.10(a): EXAMPLE OF A MOONEY SCORCH CURVE

FIGURE 3.10(b): EXAMPLE OF MONSANTO RHEOMETER CURVE
or lowered by an air cylinder. Thus the sample is placed in the chamber, the top die is closed and the samples shaped by the dies, and the excess squeezed out.

As the rotor oscillates the torque generated is measured by a strain gauge, and the electrical signal is amplified and sent to a recorder. The result is a plot as shown in Figure 3.10b. The cure time is taken as the time to reach 95% of the maximum torque. The maximum torque is a measure of crosslink density, and so is related to modulus. The minimum torque is taken as a measure of the viscosity of the compound, and a 3 point rise from the minimum is taken as a measure of scorch time. However it should be pointed out that the test is normally run at vulcanisation temperatures and so the values for viscosity and scorch are not very meaningful at processing conditions.

3.4 CARBON BLACK DISPERSION

3.4.1 The Effect of Carbon Black Dispersion on Rheological and Physical Properties of Mixed Rubber Compounds

The importance of carbon black dispersion, and its effect on rheological and physical properties of rubber compounds is well known\(^{19,20,21}\).

Poor dispersion of carbon black is typified by regions of colloidally dispersed carbon black interdispersed with regions of clear polymer.

As the mixture is subjected to high dispersive stresses, the size of the dispersed regions is broken down until they approach the size of carbon black aggregates \((0.05 - 0.5 \mu m)^{21}\). At this stage the carbon black aggregates should be uniformly distributed throughout the polymer phase if satisfactory dispersion has been obtained.

If carbon black dispersion is viewed in this manner, it makes it easier to understand the changes which take place in the rheological and physical properties.
Mooney viscosity decreases as carbon black dispersion increases\textsuperscript{20,21}. The reason given for this is that poorly dispersed carbon black contains black particles whose voids are filled with rubber, and then dispersed in a matrix of clear polymer.

The effective volume of these areas of carbon black with occluded polymer, plus the polymer immobilised between the aggregates is higher than the carbon black itself. They therefore have a more pronounced effect on the viscous flow properties than well dispersed carbon black. However when examining the effects on die-swell one finds these factors work in the reverse direction.

Initially more polymer is immobilised by the carbon black and so die swell is lower with poor dispersion. As the dispersion is improved so die swell increases. However a point is reached where the work required to improve carbon black dispersion produces significant molecular weight reductions. This serves to reduce the elasticity of the polymer and so has the effect of reducing die-swell. So the effect of improved carbon black dispersions on die-swell is offset by the reduction in elasticity and a plateau is reached where die-swell changes very little with further work. From this last discussion one can see the importance of controlling carbon black dispersion, as well as the polymer molecular weight, if the forming operations such as extrusion, are to be performed with a minimum of waste and at a high level of productivity.

What then is the effect of carbon black dispersion on the physical properties? As one would expect abrasion resistance, tensile strength, elongation to break, fatigue resistance and cut growth resistance all improve with improved dispersion. The reason for this is that with poor dispersion one has areas of high black concentrations including pellet fragments, and areas of clear polymer. The areas with excessive black can act as flaws\textsuperscript{22} when the rubber is stretched. Similarly clear polymer has poor abrasion resistance compared to a polymer with
carbon black reinforcement. So the combination of rapid wearing areas and inclusions which can actually pop out, leaving voids in the compound, produces poor abrasion resistance.

Therefore ideal carbon black dispersion is obtained when a uniform black network is established, with no areas of localised high black levels which could lead to initiation sites for failure.

3.4.2 Measurement of Carbon Black Dispersion Level

There are many ways of evaluating the level of carbon black dispersion. They include electrical resistivity\textsuperscript{27,29}, comparison of photomicrographs with standards, and various microscopic techniques\textsuperscript{30,31} involving reflected light. The only technique that will be discussed fully is the dark field reflected light technique employed in this work.

**Dark Field Reflected Light Microscopy (DFRL)**

Figure 3.11 is a schematic which demonstrates the principles of the technique. A sample of rubber is cut with a single edged razor blade. This is a critical step because the roughness of the surface determines the amount of reflected light. The surface roughness of the sample is dependent upon the type of carbon black, the type of polymer and the degree of dispersion. Therefore one does not obtain an absolute value for carbon black dispersion, but a comparative value.

So while it is excellent technique for evaluating differences between batches of the same compound, it is difficult to make comparisons between data from different compounds.

The freshly cut sample is placed into the holder positioned on the microscope stage. A narrow cone of light is made to be incident on the surface by an arrangement of mirrors as in Figure 3.11\textsuperscript{23}. 
FIGURE 3.11: PRINCIPLE OF DARK FIELD REFLECTED LIGHT MICROSCOPY

a = stop; s = sample; o = objective
FIGURE 3.12: PHOTOMETRIC SYSTEM
SCHEMATIC OF THE CARBON BLACK DISPERSION METER
If the sample surface is perfectly smooth, then no scatter of the light beam occurs, and so no light is collected by the objective lens. On the other hand if the surface contains undulations, then light scattering will occur and light will strike the objective lens; where it is quantified by a photometer.

The poorer the dispersion the greater the surface roughness, and so the more light falling onto the objective lens.

Figure 3.12 is a schematic of the complete arrangement. The image, as seen by the microscope, is transmitted by a television camera to a VDU. The computer controls the movement of the sample so that a series of locations are selected and dispersion levels obtained. In this case 100 readings per sample were taken in an area 1500 A° long and 25 A° wide during 4 sweeps. The information is collected by the computer and processed. The intensity of the light collected by the objective lens is referred to as a reading. The results are presented as the mean reading, the standard deviation of the readings, the maximum and minimum readings, and the value of the most-frequently occurring reading.

These values give a fair picture of the quality of carbon black dispersion. As a general rule a poorly mixed batch will give a higher mean reading and a higher standard deviation, than a batch with a higher level of carbon black dispersion.\textsuperscript{23}

Ebell and Hemsley\textsuperscript{35} evaluated the black dispersion of an SBR compound after different lengths of mixing. They found the DFRL technique correlated very well with physical properties such as tensile strength, elongation at break, fatigue life, and tear strength. Compared to the Cabot dispersion rating, and Leigh-Dugmore dispersion rating the DFRL technique was found to be more discriminating at higher levels of carbon black dispersion. The compounds mixed in this work were one step mixed natural rubber. This produced a very "nervy" compound which made it very difficult to produce a smooth surface. In retrospect a more conventional technique for evaluating carbon black dispersion may have been more appropriate.
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CHAPTER 4
MODEL REFERENCE CONTROL

4.1 THEORY

4.1.1 Review of Related Work

Goodrich and Porter\(^1\) attempted to develop the theory necessary to convert the torque data from a Brabender torque rheometer into fundamental rheological units. In doing so the assumption was made that the torque rheometer could be treated as two irregular, adjacent, concentric cylinder, rotational viscometers. The treatment involved the calibration of the instrument by the use of Newtonian fluids.

This work was successful for Newtonian fluids, but most polymers are non-Newtonian and therefore the shear stress is not directly proportional to shear rate. Since there are a range of shear rates over the surface of the rotors, the treatment of non-Newtonian fluids becomes very complicated.

Blyler and Daane\(^2\) attempted to develop the model for non-Newtonian flow. They again assumed the Brabender could be taken as twin concentric viscometers, but they recognised the shear rate varied across the rotors, and so assigned a minimum and a maximum shear rate to each rotor:

\[
\begin{align*}
\dot{\gamma}_{\text{max}} &= k_1 S \\
\dot{\gamma}_{\text{min}} &= k_2 S
\end{align*}
\]

where \(S\) is the rotor speed, and \(k_1\) and \(k_2\) although related to the power law constant \(n\), were taken to be constant.

They concluded that the power law index \(n\) could be obtained from the Brabender data by plotting \(\log\) Brabender torque versus \(\log\) rpm.
This was done for both linear polyethylene and branched polyethylene. The power law index was determined from capillary rheometer data and agreed closely with the values obtained from the Brabender.

Lee and Purdon\textsuperscript{3} continued the work on the Brabender torque rheometer by relating the torque and rotor speed to average shear stress and shear rate values, respectively.

To obtain the average shear stress they stated that the total force over an imaginary surface area should be balanced by the Brabender torque $M$ at a shaft speed of $N$ rpm. Using the effective rotor radius $R_e$, rotor length $L$, and knowing that one rotor operates at $\frac{2N}{3}$ rpm, one obtains the equation for the area:

$$\text{Area} = 2\pi R_e L \left(1 + \frac{2}{3}\right)$$

The effective rotor radius was used as an adjustment factor to balance the total shear force to the Brabender torque. Therefore:

$$9.80 \times 10^4 M = R_e \tau \text{(Area)}$$

or

$$9.80 \times 10^4 M = 2\pi R_e^2 L \left(1 + \frac{2}{3}\right) \tau$$

which rearranges to:

$$\tau = 1.978 \times 10^3 M R_e^{-2} \text{ (dyne/cm}^2\text{)} \quad (4.1)$$

for the average shear rate they assumed, the shear rate was at the surface of the imaginary cylinder and the flow is laminar. A machine constant, $a$, was introduced to account for the disruption of flow which occurs where the two cylinders interface. The effective chamber
radius was given by $R_c$.

The resulting equation for the average shear rate:

$$\dot{\gamma} = \frac{1.1745 \ a \ N}{n(1 - (R_e/R_c)^{2/n})} \ \text{sec}^{-1} \quad (4.2)$$

If a power law relationship is assumed i.e. $\tau = K\dot{\gamma}^n$, then a substitution of equations 4.1 and 4.2 into the power law equation produces:

$$1.978 \times 10^3 \ M \ R_e^{-2} = K \left(\frac{1.1745 \ a \ N}{n(1 - R_e/R_c)^{2/n}}\right)^n$$

which rearranges to

$$M = \frac{R_e^2}{1.978 \times 10^3} \ \left\{\frac{1.1745 \ a}{n(1 - (R_e/1.975)^{2/n})}\right\}^n K \ N^n \quad (4.3)$$

4.1.2 Development of Viscosity Model

Assumptions:

1. An internal mixer may be represented by two adjacent rotational viscometers
2. The polymers obey the power law model i.e. $\tau = n_a \dot{\gamma}^n$
3. The flow is laminar.

Assuming no speed differential between the rotors and zero wall slip, the average shear rate is given by:

$$\dot{\gamma}_e = \frac{2\omega}{n(1 - (r_e/r_c)^{2/n})} \quad (4.4)$$
where \( \omega \) = angular velocity
\( n \) = power law index
\( r_e \) = effective rotor radius
\( r_c \) = effective chamber radius

However it is known that zero wall slip is not a valid assumption. Therefore making a correction for the effect of wall slip on angular velocity, the "true" angular velocity of the material at the surface defined by the effective rotor radius is given by:

\[
\omega_t = \omega - \frac{V_{se}}{r_e} - \frac{V_{sc}}{r_c}
\]  \hspace{1cm} (4.5)

where \( V_{se} \) and \( V_{sc} \) are the slip velocities at the rotor surface and chamber wall respectively.

The slip velocity may be defined by:

\[
V_s = k \tau^m
\]  \hspace{1cm} (4.6)

and

\[
k = c e^{-b \theta}
\]  \hspace{1cm} (4.7)

where \( m \) = a material constant
\( \theta \) = absolute temperature at rubber/metal interface
\( b \) = temperature dependence of slip velocity index
\( c \) = constant of proportionality.

Substituting equations 4.6 and 4.7 into 4.5, produces an equation for "true" angular velocity which takes into account the temperature dependence of wall slip

\[
\omega_t = \omega - \frac{c e^{-b \theta} \tau_e^m}{r_e} - \frac{c e^{-b \theta} \tau_c^m}{r_c}
\]  \hspace{1cm} (4.8)
where \( \tau_e \) = average shear stress at rotor surface
\( \tau_c \) = average shear stress at chamber wall
\( \theta \) = assumed to be the same at rotor/rubber and chamber/rubber interfaces

If the equation for true angular velocity is substituted back into the equation for shear rate:

\[
\dot{\gamma}_e = \frac{\omega - \frac{c_e^{-b\theta} \tau_e^m}{r_e} - \frac{c_e^{-b\theta} \tau_c^m}{r_c}}{2\ln \left(1 - \left(\frac{r_e}{r_c}\right)^{2/n}\right)}
\]

Shear stress is given by \( \tau = \text{force/area} \), and force may be expressed in terms of torque by the relationship, \( \text{Force} = \text{Torque}/r_e \). The area of each rotor of length \( L \) is given by \( 2\pi r_e L \), and so the shear stress for each rotor is given by:

\[
\tau_e = \frac{T_1}{2\pi r_e^2 L}
\]

where \( T_1 \) is torque on each rotor.

The second rotor only travels 0.87 times as fast as the first, therefore the total shear stress for two rotors is given by:

\[
\tau_e = \frac{T}{2\pi r_e^2 L (1.87)} = \frac{T}{3.74 \pi r_e^2 L}
\]
where $T = \text{total torque on rotors}$.

Substituting for shear stress in equation 4.9 and adjusting the equation to account for the speed differential of the rotors:

$$
\dot{\gamma}_e = \frac{\omega \cdot c \cdot e^{-b \theta} \cdot \left[ \frac{T}{3.74\pi r_e^2 T} \right]^m - c \cdot e^{-b \theta} \cdot \left[ \frac{T}{3.74\pi r_c^2 T} \right]^m}{\frac{r_e}{r_c} \cdot 0.53n \left[ \left( 1 - \frac{r_e}{r_c} \right)^{2/n} \right]}
$$

(4.12)

Apparent viscosity is given by $\eta_a = \tau \cdot \frac{1}{\dot{\gamma}}$

Therefore combining equations 4.11 and 4.12

$$
\eta_a = \frac{T}{3.74\pi r_e^2 L} \cdot \frac{0.53 \left[ \left( 1 - \frac{r_e}{r_c} \right)^{2/n} \right]}{\frac{c \cdot e^{-b \theta} \cdot \left[ \frac{T}{3.74\pi r_e^2 T} \right]^m - c \cdot e^{-b \theta} \cdot \left[ \frac{T}{3.74\pi r_c^2 T} \right]^m}{\frac{r_e}{r_c}}}
$$

(4.13)

Therefore it is possible to calculate the apparent viscosity at any point in the mixing cycle. By mixing a batch which has the desired rheological properties, the set point conditions are obtained for addition of carbon black, oil, curatives and dump.

The set point conditions include a shear stress and temperature prevailing for the measurement of the apparent viscosity.

These then become reference conditions and the prevailing viscosity must be normalised to these conditions.
The form of the equation used to normalise the prevailing apparent viscosity to reference shear stress is:

\[ \eta_a = \eta_{as} \left[ \frac{\tau_{prev}}{\tau_{ref}} \right]^{(n-1)/n} \]

or

\[ \eta_{as} = \eta_a \left[ \frac{\tau_{prev}}{\tau_{ref}} \right]^{(n-1)/n} \tag{4.14} \]

where \( \tau_{prev} \) = prevailing shear stress
\( \tau_{ref} \) = shear stress for reference conditions
\( \eta_{as} \) = apparent viscosity adjusted for shear stress to reference conditions
\( \eta_a \) = apparent viscosity at prevailing conditions of temperature and shear stress
\( n \) = power law index.

Normalising the compound for temperature:

\[ \eta_{ast} = \eta_{as} e^{b(T_{prev} - T_{ref})} \tag{4.15} \]

where \( \eta_{ast} \) = apparent viscosity adjusted to reference temperature and shear stress
\( T_{prev} \) = prevailing temperature
\( T_{ref} \) = reference temperature
\( b \) = material constant
Therefore a comparison of the apparent viscosity normalised to reference conditions, with the reference viscosity, was used to determine the end point of a mixing cycle.

4.1.3 Need for a Temperature Control Model

Control of batch temperature is important because of the temperature dependence of both viscosity and the vulcanisation system. The viscosities of rubber compounds drop exponentially with increase in temperature. This means that shear stresses are also reduced as the viscosity is reduced. Since high shear forces are necessary for dispersive mixing, an increase in temperature can lead to less effective mixing. Therefore control of a batch temperature at an upper limit can possibly lead to more efficient mixing. The ability to add a cure system into a batch is determined by the temperature the batch can be dumped at and the type of curing system being used. If it is not possible to add the curing system, then a second or third pass will have to be made through the internal mixer. Therefore keeping the batch temperature below some maximum level which allows the curatives to be added to the internal mixer can be very cost effective.

It may be shown that the temperature rise of a batch is a function of the rate at which energy is put into it and the rate at which energy is removed by the cooling system. The energy remaining causes a rise in the temperature of the batch, hence

\[ E_0 = E_1 + E_2 + E_3 \]  

(4.16)

where \( E_0 \) = total energy input  
\( E_1 \) = energy to the cooling system  
\( E_2 \) = energy losses by convection through the machine body, inefficiency in the gears, etc.
\[ E_3 = \text{energy remaining which causes a temperature rise in the batch.} \]

Therefore if the energy input is at a lower rate, then the cooling system will remove a higher percentage of the energy, which will result in less energy being available to raise the temperature of the batch.

It has been shown that energy input is influenced by rotor speed and ram pressure. Therefore if energy input is modified, then batch temperature can be modified.

4.1.4 Temperature Control by Variable Rotor Speed

The type of control system used is a 3 term proportional-integral-derivative system. The voltage output specified from the control equation is used to control the speed of the rotors.

**Proportional Term:**

This control mechanism functions within a band, the width of which is typically 20°C. Within that band the power output varies from 100% to 0%. It can be seen therefore that proportional control will approach the batch target temperature very rapidly, but would oscillate quite significantly. It is also possible that a severe overshoot could occur and shut off the rotors completely. Therefore proportional control for this application needs to be modified for other terms.

**Integral Term:**

This term acts as a damping device. The temperature differential between the target temperature and the prevailing temperature is known as the error voltage. The error voltage is summed up during the mixing cycle, such that as the output from the proportional control approaches zero, the integral term continues to make a contribution.
When the set point is exceeded, the error voltage becomes negative, and so the integral term diminishes.

**Derivative Term:**

This term projects ahead and causes a reaction to reduce oscillation, even more than the damping effect of the integral term. It reacts in proportion to the rate of change of the error voltage with time. Therefore the three terms produce a control system which is both responsive and stable.

The model is a standard PID equation, which takes the following form:

\[ V = k_1 e + k_2 \tau_D \frac{de}{dt} + k_3 \int_0^t e dt \]  \hspace{1cm} (4.17)

where
- \( V \) = output voltage to rotors
- \( e \) = error voltage
- \( k_1 \) = proportional gain constant
- \( k_2 \) = derivative sensitivity constant
- \( k_3 \) = integral sensitivity constant
- \( \tau_D \) = derivative time constant
- \( \tau_I \) = integral time constant

It is extremely important to have the correct values for sensitivity constants if the controller is to work effectively.

### 4.2 EXPERIMENTAL

#### 4.2.1 Experimental Objectives and Experiment Designs

In the factory the temperature of the cooling water normally varies with the ambient air temperature. Therefore, for a control system to be considered acceptable, it should be independent of cooling water temperature. The system must also function over a wide range of dump temperatures. This experiment seeks to determine whether
the viscosity model will determine viscosity measurements which are independent of cooling water temperature and set point temperature.

The experimental design used is:

<table>
<thead>
<tr>
<th>Batch $T^0$</th>
<th>Cooling Water $T^0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>-1.42</td>
</tr>
<tr>
<td>0</td>
<td>-1.42</td>
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<tr>
<td>-1</td>
<td>-1</td>
</tr>
<tr>
<td>+1</td>
<td>-1</td>
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</tr>
<tr>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

4.2.2 Farrel Bridge F40 Banbury Experiment

The experiment was run on the F40 Banbury at Farrel-Bridge (Rochdale). This mixer has a chamber capacity of 40 litres. The batch weight was 35.74 kg.

A Digital Equipment Company (DEC) MINC 11 (module instrument computer) was connected to voltage input on the main control panel which sent the signal to control the speed of the rotors. The power output signal line from the Banbury was connected to the computer. The Banbury itself
was not automated, so it had to be operated manually. The output voltages from the computer were used to operate a system of lights, which indicated when various operations, such as polymer addition, pigment addition etc. should be made.

Table 4.1 shows the formulation of the rubber compound used in the experiment, and details of the experimental parameters. The mixing specification is shown in Table 4.2. This is known as an upside down mix, because the carbon black and oil are mixed together prior to the addition of the polymer. The batches were mixed using the viscosity control program described in Section 4.1.2.

4.2.3 Farrel-Bridge BR Banbury Experiment

The experiment was run on the BR size Banbury at Loughborough University. The machine has a chamber capacity of 1.59 litres and a batch size of 1.304 kg. The machine is equipped with a variable speed motor, which was controlled by the MINC II computer. The pneumatic systems of the Banbury were modified so that the ram, and the gate could be operated by signals from the computer. A feed hopper was fitted to the back of the Banbury and this was also operated by a signal from the computer. The instantaneous motor power, and batch temperature were fed to the computer on a continuous basis. The temperature was used to control the rotor speed; and the rotor speed and power readings were used to make the viscosity calculations. The formulation of the compound used is given in Table 4.7. The procedure was to cut the polymer into cubes of approximately 1 cm sides. A remote signal, from a switch at the Banbury, was used to start the computer program. This started the rotors at a set speed, and allowed 20 secs to add the polymer and close the loading door. The carbon black and other pigments were then automatically charged from the black hopper, and the batch was then mixed according to the temperature set point and to the target viscosity.
<table>
<thead>
<tr>
<th></th>
<th>PHR</th>
<th>Actual Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dutral 048E</td>
<td>62</td>
<td>5.42</td>
</tr>
<tr>
<td>Vistalon 5630</td>
<td>49.6</td>
<td>4.34</td>
</tr>
<tr>
<td>FEF black</td>
<td>118.7</td>
<td>10.38</td>
</tr>
<tr>
<td>MT black</td>
<td>60</td>
<td>5.25</td>
</tr>
<tr>
<td>Metasorb C</td>
<td>5</td>
<td>0.44</td>
</tr>
<tr>
<td>Stearic acid</td>
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<td>0.087</td>
</tr>
<tr>
<td>Zinc oxide</td>
<td>7.5</td>
<td>0.656</td>
</tr>
<tr>
<td>Trupar oil</td>
<td>99.0</td>
<td>8.66</td>
</tr>
<tr>
<td>ZDBC</td>
<td>1.7</td>
<td>0.149</td>
</tr>
<tr>
<td>MBTS</td>
<td>1.25</td>
<td>0.109</td>
</tr>
<tr>
<td>BDMC</td>
<td>0.13</td>
<td>0.011</td>
</tr>
<tr>
<td>Sulphur</td>
<td>2.46</td>
<td>0.215</td>
</tr>
<tr>
<td></td>
<td>408.34</td>
<td>35.74</td>
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</tbody>
</table>

**Experimental Limits:**

<table>
<thead>
<tr>
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<th>-1</th>
<th>0</th>
<th>+1</th>
<th>+1.42</th>
</tr>
</thead>
<tbody>
<tr>
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<td>29</td>
<td>40</td>
<td>51</td>
<td>60</td>
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<tr>
<td>Batch temperature °C</td>
<td>70</td>
<td>83</td>
<td>100</td>
<td>117</td>
<td>130</td>
</tr>
</tbody>
</table>

**Reference Values Used:**

- Shear stress: 95055
- Apparent viscosity: 19906 Pa.s
- Temperature: 90°C
**TABLE 4.2: Mixing Specification for EPDM Compound Used in Farrel-Bridge F40 Experiment**

<table>
<thead>
<tr>
<th>Step Description</th>
<th>Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Start: Add black, oil</td>
<td>20 rpm</td>
</tr>
<tr>
<td>60 secs: Add polymer, zinc oxide, stearic acid</td>
<td>60-20 rpm</td>
</tr>
<tr>
<td>Add sulphur, Metasorb C</td>
<td></td>
</tr>
<tr>
<td>Preset viscosity conditions, add ZDBC, MBTS, BDMS</td>
<td>60-20 rpm</td>
</tr>
<tr>
<td>Preset viscosity conditions Dump</td>
<td>60-20 rpm</td>
</tr>
<tr>
<td>Rotor speed range:</td>
<td>20 rpm to 60 rpm</td>
</tr>
</tbody>
</table>

**Note:**

Batches were dumped onto a stationary mill. One pass was made to simply put the batch into slab form.
The batches were mixed in a random order. The outlet temperature of the water was controlled by a water tempering unit, which was capable of heating or cooling the water. The actual temperature control unit was a Eurotherm proportional-integral-derivative controller. The Banbury was allowed to come to a temperature equilibrium, after each change in cooling water temperature.

The experimental design was the same one used in the Farrel-Bridge F40 experiment and is described in Section 4.2.1. The experimental limits and reference values used are given in Table 4.7.

4.3 RESULTS AND DISCUSSION

4.3.1 Farrel-Bridge F40 Experiment

The results are summarised in Table 4.3. They show that the prevailing viscosity is very variable, and even after normalising for temperature and shear stress to the reference conditions, it is still variable. The statistical analysis of the prevailing viscosity, Tables 4.4 and 4.5, shows a dependence on batch temperature and rotor speed. The confidence levels are not very high, and therefore the values generated by the model must be treated with great caution. However the confidence levels are high enough to say the dependence is real and not just a coincidence. The statistical analysis of the adjusted viscosity shows lower confidence levels for both batch temperature, and rotor speed dependence. The confidence level for the batch temperature is significantly lower which indicates the temperature normalisation to the reference temperature is working. The confidence level for the rotor speed is slightly lower, which would indicate the adjustment to the reference shear stress is in the right direction, but not as effective as it should be.

Figure 4.1 illustrates the temperature control function of the control program. The data for this batch is given in Table 4.6. It may be seen clearly that the rotor speed declines as the target
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td>50 range, 30 arc</td>
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<td>37</td>
<td>202</td>
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</tr>
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<td>188</td>
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<td>189.3</td>
<td>7.7</td>
<td>100</td>
<td>Cond. on rts</td>
</tr>
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<td>7.7</td>
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<tr>
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<td>98</td>
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<td>175</td>
<td>28264</td>
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<td>88</td>
<td>20</td>
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<td>25362</td>
<td>146.9</td>
<td>7.5</td>
<td>100</td>
<td>20</td>
</tr>
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</table>
**TABLE 4.4: Statistical Analysis of the Effect of Batch Temperature on Viscosity as Determined by the Viscosity Control Program - F40 Banbury Experiment**

<table>
<thead>
<tr>
<th>Obs No.</th>
<th>Y</th>
<th>Pred Y</th>
<th>Residuals</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>25679</td>
<td>-372.012</td>
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**T - Value - 5.45**  
**Confidence level 83.4%**  
**Form of equation: Prevailing Viscosity = (-526.707 x Batch T°) + 73082.7**

<table>
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<th>Obs No.</th>
<th>Pred Y</th>
<th>Residuals</th>
</tr>
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**T - Value - 3.29**  
**Confidence level 67.39%**  
**Form of equation: Adjusted Viscosity = (-404.09 x Batch T°) + 63166.2**
TABLE 4.5: Statistical Analysis of the Effect of Rotor Speed on Viscosity as Determined by the Control Program - F40 Experiment

Prevailing Viscosity

<table>
<thead>
<tr>
<th>Obs No.</th>
<th>Y</th>
<th>Pred. Y</th>
<th>Residuals</th>
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<tbody>
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T Value - 3.57
Confidence level 70.35%
Form of equation: Prevailing Viscosity = (-857.136 x rotor speed) + 41337.2

Adjusted Viscosity

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T Value - 2.96
Confidence level 63.4%
Form of equation: Adjusted Viscosity = (-733.297 x rotor speed) + 40531.9
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TABLE 4.7: Experimental Information from BR Banbury Experiment

S.G. 1.11

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<td>PVI</td>
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<table>
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<tr>
<th>Experimental Limits</th>
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<tr>
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</tr>
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<td>80 89 110 131 140</td>
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</table>

Reference values used:

Shear stress: 133573 Pa
Apparent viscosity: 20,187 Pa.s
Temperature: 107°C

Mixing specification:

<table>
<thead>
<tr>
<th>Time</th>
<th>Rotor Speed</th>
<th>Remarks</th>
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<tr>
<td>0</td>
<td>30</td>
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</tr>
<tr>
<td>20 seconds</td>
<td>30-85</td>
<td>Add carbon + all other ingredients</td>
</tr>
<tr>
<td>Variable, depending</td>
<td>30-85</td>
<td>Dump</td>
</tr>
<tr>
<td>when viscosity limit</td>
<td></td>
<td></td>
</tr>
<tr>
<td>met</td>
<td></td>
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</tbody>
</table>
temperature is approached. Although the constants in the 3-term PID equation were not set correctly, it can be clearly seen that the concept of controlling batch temperature with rotor speed does work.

As can be seen from the Monsanto Oscillating Disk (ODR) rheometer data, the vulcanisation properties are extremely variable. In retrospect this is what one would expect, adding the curatives at a fixed viscosity whilst varying the dump temperature. However it does serve to show that curatives should be added at a point in the mixing cycle, such that a heat equivalence is obtained.

4.3.2 Farrel-Bridge BR Banbury Experiment

A summary of the results is shown in Table 4.8. Two things are immediately obvious. The first is that the viscosity values, as measured by the model, are extremely variable, and secondly they vary from the reference viscosity ($\eta_0$) values obtained from the TMS biconical rotor rheometer by a factor of approximately 4. Further, a review of the statistical analysis in Table 4.11 shows no correlation between the viscosity measurements determined by the model and $\eta_0$ as determined by the TMS rheometer.

The notes in Table 4.8, show that the experiment was not run as initially intended, because the viscosity dump criteria was rarely met and therefore the majority of batches were dumped manually.

A review of Tables 4.9 and 4.10 show again the very strong dependence of the viscosity, as determined by the model, on rotor speed and batch temperature.

Adjusting the apparent viscosity to the reference temperature and shear stress conditions reduced the temperature dependence, but had no effect on the rotor speed dependence. However the very positive result to come out of this experiment is the excellent control of
TABLE 4.8: Results from BR Banbury Experiment

Targets $\eta_a = 20187; \ \tau = 133573; \ T = 107^\circ C$

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<th>Batch No.</th>
<th>Variables(°C)</th>
<th>Batch Data at Dump</th>
<th>TMS Data @ 100°C</th>
<th>Results</th>
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<td>Temp (°C)</td>
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TABLE 4.9: Statistical Analysis of the Effect of Batch Temperature on Viscosity as Determined by the Control Program - BR Banbury Experiment

**Prevailing Viscosity**

<table>
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T-Value - 5.703 \[ \text{Confidence level } 84.53\% \]

Form of equation: \( \text{Prevailing Viscosity} = (-463.823 \times \text{Batch temp}) + 73533.7 \)

**Adjusted Viscosity**

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T-Value - 3.963 \[ \text{Confidence level } 73.97\% \]

Form of equation: \( \text{Adjusted Viscosity} = (-336.011 \times \text{Batch temp}) + 59886.7 \)
TABLE 4.10: Statistical Analysis of the Effect of Rotor Speed on Viscosity as Measured by the Control Diagram - BR Banbury Experiment

**Prevailing Viscosity**

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T-Value - 10.282  
Confidence level 94.37%  
Form of Equation: Prevailing Viscosity = (-588.807 x Rotor speed) + 42279.5

**Adjusted Viscosity**

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T-Value - 10.227  
Confidence level 94.31%  
Form of Equation: Adjusted Viscosity = (-487.134 x Rotor speed) + 39421.8
TABLE 4.11: Statistical Analysis of the Relationship of Viscosity as Determined by the Control Program and $\eta_0$ as Determined by the TMS Rheometer

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T-Value = .100  Confidence level = 2.78%

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T-Value = .125  Confidence level = 3.48%
TABLE 4.12: Example of Data from BR Banbury at Loughborough University

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FIGURE 4.2

BATCH TEMPERATURE PROFILE - BR BANBURY

ROTOR SPEED PROFILE - BR BANBURY
temperature to a given set point. The data from Table 4.12 is plotted out in Figure 4.2, and illustrates very clearly the rotor speed adjusting to hold the batch temperature accurately at the set point temperature.

### 4.4 CONCLUSIONS

The results from the two experiments show quite clearly that the viscosity model, as proposed, did not work. The major drawback was a dependence on rotor speed, and a dependence on temperature. However the results showed that it was possible to make temperature corrections on the viscosity.

The conclusion one must come to is that assuming the Banbury can be represented by two adjacent rotational viscometers is not valid, and that the resultant model is too simplistic.

In an internal mixer the two rotors run at different speeds, and material is transferred from one side of the chamber to the other side. The model, as proposed, assumed the material simply moved within the one cylinder, and that there is no interchange between chambers or interference in flow. This of course is incorrect and no doubt has a significant effect on the actual shear rate.

The assumption is also made that the rotor may be represented by a smooth cylinder. This means the shear rate is assumed to be given across the surface of the rotor. However this is not the case, and in reality there is a complete spectrum of shear rates. With the cylinder assumption, the flow can be envisaged as a series of concentric circles all moving in a laminar flow field with the rotor. However because of the non-uniform shape of the rotors, the material flows in an axial direction as well as in the same direction as the rotors. Another major drawback was the assumption that the behaviour of a rubber compound during mixing can be represented by a simple,
wholly viscous, power law model. This is not the case. Therefore it is obvious that the flow within the chamber of an internal mixer is very complicated.

The type of model necessary to determine shear rates must take into account the questions raised in the preceding dialogue. However a possible way around the problem, if absolute measurements of viscosity are not required, is to measure the torque at a reference rotor speed and reference temperature. This then eliminates the problem of developing a very complicated model which will remove the dependence of viscosity on rotor speed and batch temperature. The very positive result that emerged from these two experiments is the development of the batch temperature control. The system proved successful on both the laboratory size BR Banbury and the industrial size F40 Banbury.
REFERENCES

CHAPTER 5
THE DEVELOPMENT AND EVALUATION OF THE
BATCH CONDITION CONTROL SYSTEM

5.1 PRINCIPLES OF BATCH CONDITION CONTROL

5.1.1 Overall Control Strategy

The need for a batch condition control system has been established in earlier chapters, therefore it will not be repeated here. The requirements of the control system are that it will mix rubber compound with uniform vulcanisation and rheological properties, despite the raw materials having mild property variations.

It is generally accepted that because of the very high molecular weight of the polymer relative to other ingredients, the polymer has the major influence on the rheological properties of the mixed rubber compound. It has been well established that the molecular characteristics of polymers are related to processing characteristics\textsuperscript{1,2,34}. It has also been shown that the molecular properties of polymers can be modified\textsuperscript{5,6}. So if a compound of uniform rheological properties is to be mixed, the polymer must be within specified rheological limits, and if not, there should be a means of modifying the polymer such that it meets the limits. The batch condition control system is able to do this.

The other parameter, which has a major effect on the manner in which a mixed rubber compound will act at the two roll mill, extruder or other shaping process, is the state of mix\textsuperscript{7,8}. This is controlled by ensuring a rheologically related set point is met during the carbon black dispersive mixing phase, which is before the addition of oil.

Finally in order to obtain a satisfactory cure-rate, the amount of heat history to which the vulcanisation system is subjected must be controlled. In the case of variable speed internal mixers, the maximum temperature the batch is subjected to is controlled by varying the rotor speed. In both the fixed speed and variable speed programs, the point at which the
crosslinking agent is added is calculated from the endpoint, and adjusted to obtain the same heat history for each mixed rubber compound.

Summarising the overall control strategy: the polymer is adjusted to meet rheological limits. The mix cycle is continuously monitored and rheological targets met at each phase to ensure a uniform state of mix, and the vulcanisation system addition time is controlled to ensure a uniform heat history. Finally the batch is dumped when a rheological target is met.

5.1.2 The Relationship of Torque to Apparent Viscosity

In the previous chapters a model for measuring viscosity during the actual mixing cycle was proposed and evaluated. The result was a model which had a marked dependence on both rotor speed and batch temperature. It became obvious that several factors made the measurement of absolute viscosity in the chamber of the Banbury very difficult:

1. The power law constant changes as the incorporation and dispersion of the filler proceeds.

2. The spectrum of shear rates throughout the chamber make it impossible to obtain a shear rate which can be considered representative of the system at any given point in time. One would expect this problem to get worse when mixers of larger capacity are used, and may explain why work using Brabender mixers was more successful.

3. The interference of flow patterns which occurs, when material is interchanged from one side of the mixing chamber to the other, again leads to problems with the shear rate.

Thus one can see that the major problem was related to obtaining satisfactory shear rates. The problem therefore is how to obtain an accurately measurable parameter, which can be related to the rheological properties of the material being mixed. Going back to basics it can be shown that shear stress is given by:
\[
\tau = \frac{\text{Force}}{\text{Unit area}} \quad (5.1)
\]

For an internal mixer the unit area will be a machine constant, which will be directly related to the effective radius of the rotors.

The torque on the rotors during the mixing cycle will be given by:

\[
\text{Torque} = \text{Force} \times \text{Rotor Radius} \quad (5.2)
\]

Therefore combining equations 5.1 and 5.2 we obtain for shear stress:

\[
\tau = \frac{\text{Torque}}{\text{Rotor radius} \times \text{machine constant}} \quad (5.3)
\]

and since for any given machine the rotor radius is a constant, torque should be directly proportional to shear stress.

It is well established that the rotor speed determines the flow properties of a polymer system within a mixing chamber, and thus the resultant shear rates. The difficulty of determining accurately a representative shear rate has been discussed previously. However, the viscosity of a power-law non-Newtonian fluid is given by:

\[
\frac{\tau}{\dot{\gamma}} = \eta_0 \dot{\gamma}^{n-1} \quad (5.4)
\]

This shows the shear rate affects the value of the resultant viscosity.

To avoid the problem of shear rate dependence, the torque can be measured at some reference value of rotor speed. At these conditions, for a given machine, the viscosity should relate directly to torque. However, viscosity is related to temperature by an Arrhenius relationship. This
means that for torque to be related to viscosity it must be at a reference rotor speed, and a reference temperature. The reference temperature should be chosen such that only small corrections in temperature from the actual batch temperature will be necessary.

5.1.3 Torque Control

In the preceding section it has been demonstrated, that at a given rotor speed and batch temperature, the torque on the rotors is proportional to the viscosity of the compound. It follows that for the same compound or polymer it is possible to determine the torque at these reference conditions, and after comparing with a reference torque value, make decisions concerning the viscosity of the batch being mixed.

The points during the cycle in which the torque is compared with a reference value are: during the polymer mastication, during the carbon black dispersion stage, and when determining the point at which the cycle will be terminated. The prevailing torque is given by equation 5.5:

\[
\text{Prevailing torque} = \frac{\text{Power}}{\text{Angular Velocity}} \quad (5.5)
\]

The power is calculated from motor volts x motor amps. The rotor speed is controlled but an adjustment is normally necessary to correct the torque to the reference temperature. The form of this temperature adjustment is given by equation 5.6:

\[
\text{Torque}_{\text{adj}} = \text{Torque}_{\text{prev}} \times \exp(-b(\text{Actual } T^0 - \text{Reference } T^0)) \quad (5.6)
\]
5.1.4 Batch Temperature Control

Because vulcanisation systems are both temperature dependent and time dependent, it is often very beneficial to be able to control the maximum temperature that a batch will reach. This may lead to the possibility of mixing a batch in a single cycle through the internal mixer, as opposed to two cycles through the mixer, where the second cycle would simply be used to add the vulcanisation system. The second reason for keeping the batch temperature down is that as temperature rises, so apparent viscosity is reduced, and shear stresses which are necessary for effective dispersive mixing are also reduced. The batch control system works on the principle that reducing rotor speed will reduce the rate of heat generation. This allows the cooling system to remove a larger percentage of the available heat, thereby slowing the increase in batch temperature or even holding it in an equilibrium state.

The system is controlled by proportional integral-derivative control of the form:

\[
V = k_p \epsilon + k_d \frac{dc}{dt} + k_i \int_0^t \epsilon \, dt
\]

where \( k_p \) = constant for proportional control
\( \epsilon \) = error voltage
\( k_d \) = constant for derivative control
\( k_i \) = constant for integral control

The error voltage is the difference between the voltage for the reference temperature, and the voltage for the prevailing temperature. As can be seen the greater the error voltage the higher the total output voltage \( V \). This output voltage is used to control the rotor speed.
5.1.5 Heat History Control

The basic premise of the system is that to obtain the same heat history for the vulcanisation system, an adjustment must be made in the time the ingredients which constitute the vulcanisation system are added, relative to the time remaining to complete the mixing cycle, if the batch temperature deviates from a "typical" temperature. This means that if the batch temperature is higher than the typical temperature then the crosslinking system should be added at a point closer to the end of the mixing cycle. If the batch is lower than typical the crosslinking system should be added further from the end of the mixing cycle.

When the mixing cycle enters the final phase, a linear regression of log torque vs log energy predicts the energy necessary to reach the target torque and therefore terminate the mixing cycle. When the energy to complete the mixing cycle is determined, it is possible to extrapolate back and determine the energy value at which the crosslinking system will be added. It is this value which is adjusted, based on the temperature prevailing at that point.

The steps in the adjustment are as follows:

1. Determine the number of seconds to cycle end
2. Total secs x (Actual $T^0$ - Typical $T^0$) = deviation from typical.
   This deviation can be positive or negative
3. $\frac{\text{Deviation}}{\text{Typical } T^0}$ = correction in secs
4. Energy/sec x correction in secs = correction in energy.

The positive or negative sign automatically ensures the correction is in the right direction. If the actual temperature is higher than the typical, a positive energy correction is obtained. When this is added to the initial calculated energy for the crosslinking system addition, it means the addition occurs later in the mixing cycle, and therefore closer to the end.
5.2 EQUIPMENT

5.2.1 Banbury

The Banbury is a Farrel-bridge BR Banbury. The power is supplied by a 25 horsepower motor, which is controlled by a thyristor drive. This means that the speed of the Banbury is controlled by the voltage supplied to the motor, and therefore the speed may be varied continuously up to 140 rpm. The current and voltage used, with respect to rotor speed, for an empty Banbury was determined. An Avometer was attached to the positive and negative wires which went directly to the motor. A clip on ammeter was attached to the motor, so that the actual current being used by the motor could be measured. On the control panel a socket outlet gives a voltage in the range of 0-5 volts which is proportional to the amperage being consumed by the motor. It is this voltage which is measured by the computer, and used in the calculation of instantaneous power.

A digital voltmeter was attached to the output socket, such that the voltage could be monitored continuously. The Banbury motor voltage, motor amperage, and socket voltage was then measured at various rotor speeds starting at 20 rpm, and increasing in 10 rpm increments up to 120 rpm, and then back to 20 rpm in the same 10 rpm increments. The rotor speed is controlled by an analogue output from the computer which ranges from zero to 10 volts. The results are given in Table 5.1. The data is averaged from five complete cycles.

Above 80 rpm interference with the signal from the computer caused fluctuations in the rotor speed. For this reason none of the experimental work was carried out above 80 rpm.

A regression analysis was run on each parameter as indicated in Tables 5.2, 5.3, and 5.4. The relationship between the panel voltage output was used in all control programs on the BR Banbury to obtain the current used at any instant in time.

The equation for the motor voltage with respect to rotor speed was also used to determine the voltage the motor was operating at, at any instant
TABLE 5.1: Calibration Data on BR Banbury Motor

<table>
<thead>
<tr>
<th>Rotor Speed (rpm)</th>
<th>Motor Current (amps)</th>
<th>Volts Proportional to Amperage</th>
<th>Motor Voltage</th>
<th>Instantaneous Power (Watts)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>1.0</td>
<td>0.115</td>
<td>58</td>
<td>58</td>
</tr>
<tr>
<td>30</td>
<td>1.2</td>
<td>0.125</td>
<td>65</td>
<td>78</td>
</tr>
<tr>
<td>40</td>
<td>1.5</td>
<td>0.142</td>
<td>95</td>
<td>142.5</td>
</tr>
<tr>
<td>50</td>
<td>1.65</td>
<td>0.152</td>
<td>128</td>
<td>211.2</td>
</tr>
<tr>
<td>60</td>
<td>1.78</td>
<td>0.162</td>
<td>160</td>
<td>284.8</td>
</tr>
<tr>
<td>70</td>
<td>1.90</td>
<td>0.170</td>
<td>195</td>
<td>370.5</td>
</tr>
<tr>
<td>80</td>
<td>2.05</td>
<td>0.178</td>
<td>228</td>
<td>467.4</td>
</tr>
<tr>
<td>90</td>
<td>2.15</td>
<td>0.185</td>
<td>263</td>
<td>565.45</td>
</tr>
<tr>
<td>100</td>
<td>2.25</td>
<td>0.192</td>
<td>295</td>
<td>663.8</td>
</tr>
<tr>
<td>110</td>
<td>2.36</td>
<td>0.199</td>
<td>300</td>
<td>708</td>
</tr>
<tr>
<td>120</td>
<td>2.45</td>
<td>0.204</td>
<td>335</td>
<td>820.8</td>
</tr>
</tbody>
</table>

TABLE 5.2: Regression Analysis of Motor Amperage with Respect to Panel Output Voltage

T-value 73.56:  Confidence level: .9992

Form of equation: Motor amps = (Panel Output Voltage x 15.8869) - .7898

<table>
<thead>
<tr>
<th>Rotor Speed (rpm)</th>
<th>Panel Voltage</th>
<th>Predicted Panel Voltage</th>
<th>Residuals</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>1</td>
<td>1.04</td>
<td>-.0372016</td>
</tr>
<tr>
<td>30</td>
<td>1.2</td>
<td>1.20</td>
<td>3.92914E-03</td>
</tr>
<tr>
<td>40</td>
<td>1.5</td>
<td>1.57</td>
<td>.0338519</td>
</tr>
<tr>
<td>50</td>
<td>1.65</td>
<td>1.63</td>
<td>.0249828</td>
</tr>
<tr>
<td>60</td>
<td>1.78</td>
<td>1.78</td>
<td>-3.88622E-03</td>
</tr>
<tr>
<td>70</td>
<td>1.9</td>
<td>1.91</td>
<td>-.0109814</td>
</tr>
<tr>
<td>80</td>
<td>2.05</td>
<td>2.04</td>
<td>.0119233</td>
</tr>
<tr>
<td>90</td>
<td>2.15</td>
<td>2.15</td>
<td>7.14779E-04</td>
</tr>
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<td>100</td>
<td>2.25</td>
<td>2.26</td>
<td>-.0104935</td>
</tr>
<tr>
<td>110</td>
<td>2.36</td>
<td>2.37</td>
<td>-.0117018</td>
</tr>
<tr>
<td>120</td>
<td>2.45</td>
<td>2.45</td>
<td>-1.13678E-03</td>
</tr>
</tbody>
</table>
TABLE 5.3: Regression Analysis of Motor Voltage with Respect to Rotor Speed

T-value 31.45: Confidence level: 99.55%
Form of equation: Motor voltage = (Rotor speed x 2.966) - 14.7636

<table>
<thead>
<tr>
<th>Rotor Speed (rpm)</th>
<th>Motor Voltage</th>
<th>Predicted Motor Voltage</th>
<th>Residuals</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>58</td>
<td>44.6</td>
<td>13.409</td>
</tr>
<tr>
<td>30</td>
<td>65</td>
<td>74.3</td>
<td>-9.2562</td>
</tr>
<tr>
<td>40</td>
<td>95</td>
<td>103.9</td>
<td>-8.9184</td>
</tr>
<tr>
<td>50</td>
<td>128</td>
<td>133.6</td>
<td>-5.5186</td>
</tr>
<tr>
<td>60</td>
<td>160</td>
<td>163.2</td>
<td>-3.24548</td>
</tr>
<tr>
<td>70</td>
<td>195</td>
<td>192.9</td>
<td>2.0909</td>
</tr>
<tr>
<td>80</td>
<td>228</td>
<td>222.6</td>
<td>5.42728</td>
</tr>
<tr>
<td>90</td>
<td>263</td>
<td>252.2</td>
<td>10.7637</td>
</tr>
<tr>
<td>100</td>
<td>295</td>
<td>281.9</td>
<td>13.1</td>
</tr>
<tr>
<td>110</td>
<td>300</td>
<td>311.6</td>
<td>-11.5636</td>
</tr>
<tr>
<td>120</td>
<td>335</td>
<td>341.2</td>
<td>-6.22723</td>
</tr>
</tbody>
</table>

Equation actually used: Motor voltage = (Rotor speed x 3.3) - 33

TABLE 5.4: Regression Analysis of Instantaneous Power with Respect to Rotor Speed for Empty Banbury

T-value 28.34 Confidence level: 98.84%
Form of equation: Instantaneous power = (6.984 x rotor speed) - 118.854

<table>
<thead>
<tr>
<th>Rotor Speed (rpm)</th>
<th>Actual Power</th>
<th>Predicted Power</th>
<th>Residuals</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>58</td>
<td>20.8248</td>
<td>37.1752</td>
</tr>
<tr>
<td>30</td>
<td>78</td>
<td>90.6642</td>
<td>-12.6642</td>
</tr>
<tr>
<td>40</td>
<td>142.5</td>
<td>160.504</td>
<td>-18.0035</td>
</tr>
<tr>
<td>50</td>
<td>211.2</td>
<td>230.343</td>
<td>-19.1429</td>
</tr>
<tr>
<td>60</td>
<td>284.8</td>
<td>300.182</td>
<td>-15.3822</td>
</tr>
<tr>
<td>70</td>
<td>370.5</td>
<td>370.022</td>
<td>.478485</td>
</tr>
<tr>
<td>80</td>
<td>467.4</td>
<td>439.861</td>
<td>27.5392</td>
</tr>
</tbody>
</table>
in time. It should be noted that the experimentally determined voltage for 20 rpm was greater than the voltages from the higher rotor speeds indicated it should be. Since this one point caused the regression line to be skewed, and since all the other points fitted the line precisely, a model which ignored the 20 rpm value was used. The error caused by this is insignificant because the minimum rotor speed of 20 rpm is rarely approached, and the error diminishes to zero as the rotor speed approaches 30 rpm.

The power with respect to rotor speed model from Table 5.4 is for the power consumed in physically operating the Banbury with no material in it. This is used to correct the instantaneous power readings so that the value represents the power introduced into the material during the mixing process.

5.2.2 Automation of the Banbury

Figure 5.1 is a simplified schematic, which demonstrates the technique used to automate the Banbury. The signals to operate a Banbury function come from an analogue output signal from the computer. This signal is a maximum of 10 volts and 50 milliamps. However for solenoid valves to work effectively they require 240 volts. This was achieved by using transistorised relays, which can be switched with very low voltages and minimum current. The transistorised relays are energised with a regulated twelve volt supply. For simplicity only one set of wires has been drawn in Figure 5.1. However each relay and solenoid was wired in an identical manner. The complete system is contained in an enclosed box, which is grounded.

All the electrical circuitry is contained on a board. When a signal is sent from the computer to a relay, it causes the contacts to be switched, so that a 240 volt current activates the appropriate solenoid. This moves the valve allowing air into the appropriate cylinder. The Banbury gate was put on a separate air line to the other functions. This was to allow the gate to be operated at 90 psi, which produces a more positive closing action. The other functions were operated at 40 psi.
FIGURE 5.1: SCHEMATIC OF LAYOUT OF BANBURY FUNCTION CONTROL SYSTEM
Butterfly valve rotates through 90° by means of a rack and pinion system, and a solenoid activated air cylinder.

Scale: 1 cm = 1 inch

FIGURE 5.2: CARBON BLACK HOPPER SYSTEM FOR BR BANBURY
5.2.3 Carbon Black Addition System

Figure 5.2 is a schematic of the carbon black hopper. The back of the throat of the Banbury was removed and replaced by the panel containing the carbon black hopper. The butterfly valve is operated by a rack and pinion which is driven by an air cylinder controlled by the computer. This results in a very rapid opening and closing mechanism. The carbon black is placed into the hopper, and at the appropriate point in the mixing cycle the butterfly valve opens, and the carbon black drops by gravity feed into the Banbury. The system was very effective and ensured the addition of the carbon black in the shortest period possible.

5.2.4 Oil Injection System

Figure 5.3 is a schematic representation of the oil injection system. The oil is placed in a metal cylinder, which was constructed from seamless pipe with 1 cm thick walls. The cylinder was constructed from heavy gauge steel to provide a large heat sink, and because the oil is pressurised, the cylinder is surrounded by a heater band. A thermostatic device which operates in the temperature range of 45 to 55°C, senses the temperature of the bottom of the cylinder, and turns the heater band on or off. Since the mass of the metal cylinder is large compared to the oil, it is reasonable to assume the temperature of the oil reaches an equilibrium temperature with the metal cylinder. After the oil is introduced into the cylinder, a screw down device seals the cylinder and depresses the button on a two way valve, which allows air pressure to be introduced into the cylinder. Unscrewing the locking device causes an instantaneous release of pressure through the exhaust port of the valve, thereby ensuring zero pressure is reached before the cap is removed from the cylinder.

On the underside of the cylinder is a solenoid valve, which remains closed until activated by a signal from the computer. When oil is released from the cylinder the air pressure forces the oil into a positive displacement pump. The pump is driven by a Kopp-converter coupled to the pump by a rubber coupling. The speed of the Kopp-converter can
Oil container which is surrounded by a thermostatically controlled heater band

Solenoid valve

Flexible coupling

Injection to Banbury

**FIGURE 5.3: SCHEMATIC DIAGRAM OF THE OIL INJECTION SYSTEM FOR THE BR BANBURY**
Oil from the Pump

Stainless steel construction

7/16"

FIGURE 5.4: OIL INJECTOR FOR BR BANBURY
be varied over a wide range, and so the rate at which the oil is injected into the Banbury can be varied. The signal from the computer which is used to activate the solenoid valves also activates the Kopp-converter. The air pressure on the cylinder acts as an air-purge to flush the lines free of oil when the tank and pump are empty. The oil is injected at the side of the Banbury, between the rotors. The actual injector used is illustrated in Figure 5.4. Since it fits into one of the thermocouple holes, the basic shape of the oil injector is that of the thermocouple. The system worked well with the oil injecting accurately each time, and the end of the injector remaining clear between uses.

5.2.5 Temperature Measurement

The thermocouple probe was designed to give a faster response than standard thermocouples.

A standard thermocouple casing was fabricated, and an iron/constantan thermocouple produced. The thermocouple was encased completely with a ceramic sleeve from the tip of the thermocouple, for the complete length of the casing. This was to ensure the thermocouple was measuring the actual batch temperature and was not influenced by the heat sink effect of the Banbury body. The thermocouple was then set in place with epoxy resin. As the resin set, the thermocouple tip was pushed up, shaping the epoxy resin into a radius and leaving a skim of epoxy approximately .01 mm over the thermocouple tip. The thermocouple was connected to an operational amplifier, which was calibrated using the ice reference technique so that 5 volts was equal to 150 degrees C. The operational amplifier was connected to an analogue input channel on the computer. The temperature was then monitored by the computer.

By averaging one hundred readings to obtain a temperature measurement, it was possible to achieve greater accuracy than normal pen recorder techniques.
5.3 SYSTEMS DETAIL-FIXED SPEED

The overall control program is illustrated in the flow chart in Figure 5.5. The system will be broken down into discrete sections and discussed individually.

**Mastication:**

In this step the polymer is forced into the rotors by the ram. The computer then obtains the batch temperature, and the energy used by the motor and calculates the torque on the rotors. The torque is adjusted to a reference temperature using the equation 5.6:

\[
\text{Torque}_{\text{adjusted}} = \text{Torque}_{\text{prevailing}} \times e^{-b(\text{Actual } T_0 - \text{Reference } T_0)}
\]

The value for the adjusted torque is then compared to the torque value used as the setpoint. If the value for adjusted torque is greater than the setpoint the program will stay in the loop and the polymer mastication will continue. The time required to complete each loop, is known precisely. The energy used by the motor is multiplied by the time in seconds to complete each loop. The energy in joules is summed to obtain the total energy used during the mixing cycle.

**Black Addition and Incorporation:**

Upon reaching the setpoint for the mastication step the ram is raised, the carbon black hopper opened, and the ram lowered again. At this point, the incorporation of the carbon black into the polymer commences. As more of the carbon black becomes encapsulated in the polymer matrix the torque on the rotors is increased, and the temperature of the material being mixed rises as a result of the work done on it. This process is followed by the computer, and a running average of the 3 previous torque values is calculated. This is to smooth out fluctuations in the torque.
FIGURE 5.5: FLOW DIAGRAM - FIXED SPEED

Start
Input:
File name
Batch Con.
Close gate
Raise ram

Start signal
Yes
Start rotors
Start timer
Lower ram
Obtain data
From A/N.
Calc.temp.
Obtain power values
Calc.torque
Adjust torque
to ref.
temperature
Store data
in local
array

Torque = target
Yes
Raise ram,
add carbon
black.
Obtain batch
temp.
Obtain power.
Calc. torque
Calc. running ave.
for torque
Store data
in local
array

Torque pre.val
Yes
Energy &
torque. Calc. energy to store
Reg. coeff., 7.9
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc.
torque
Obtain power

No

Obtain batch
temp.
Calc. running ave.
for torque
Calc. torque
Obtain power

No

Obtain batch
temp.
Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

No

Obtain batch
temp.
Calc. running ave.
for torque
Calc. torque
Obtain power

No

Obtain batch
temp.
Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

No

Obtain batch
temp.
Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
Calc. energy to pigment
addition
Obtain batch
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Obtain batch
temp.
Obtain power. Calc.
torque
Store data
in local
array

Calc. running ave.
for torque
Calc. torque
Obtain power

Obtain batch
temp.
values that occur during the mixing process. When the current average torque value is less than the previous average torque i.e. the torque/time curve has passed through a maximum, the program looks for another setpoint. The setpoint is a torque value which ensures the carbon black reaches a predetermined level of dispersion before any further action is taken. The torque is reduced as the carbon black dispersion improves. However as the carbon black dispersion increases so the rate at which oil will incorporate into the compound is reduced so the selection of this setpoint affects the state of mix, and the overall mixing time. When the setpoint is reached the program proceeds to the next phase of the mixing cycle.

**Oil Addition and Incorporation:**

The ram is raised and the oil is injected into the chamber followed by an air purge of 2-3 seconds, and the ram is then lowered. The addition of the oil has a lubricating effect on the contents of the mixing chamber and there is a reduction in the mixing energy going into the batch, with a resultant lowering of batch temperature.

The computer program continually monitors the batch temperature and torque, averaging the last three torque values. The torque increases as the oil is incorporated and passes through a maximum.

**Regression Analysis and Prediction of Endpoint:**

After a negative slope is obtained the program collects six pairs of torque and energy values. The data is put in log-log form and a linear regression analysis, using the least squares method, is used to predict the total energy required to reach a target torque. The torque values are adjusted for temperature using the equation in 5.6. The theory of the linear regression is as follows:

Let $x$ = independent variable (energy)
Let $y$ = dependent variable (torque)
Then \( y \) may be predicted by a relationship of the form:

\[
y = bx + a \tag{5.8}
\]

where \( a \) and \( b \) are constants given by the intercept and slope of the curve.

Applying the least squares principle the following equations are obtained:

\[
\sum_{i=1}^{n} y_i = an + b \sum_{i=1}^{n} x_i \tag{5.9}
\]

\[
\sum_{i=1}^{n} x_i y_i = a \sum_{i=1}^{n} x_i + b \sum_{i=1}^{n} x_i^2 \tag{5.10}
\]

The equations may be rewritten in terms of \( a \) and \( b \):

\[
b = \frac{n \Sigma xy - \Sigma x \Sigma y}{n \Sigma x^2 - (\Sigma x)^2} \tag{5.11}
\]

\[
a = \frac{\Sigma y - b \Sigma x}{n} \tag{5.12}
\]

Equation 5.8 can be rewritten in terms of the dependent variable:

\[
x = \frac{y-a}{b} \tag{5.13}
\]

Therefore having solved equations 5.11 and 5.12 to obtain values for \( a \) and \( b \) and inputting the value of the target torque \( y \) into 5.13, the
equation can be solved to obtain the predicted energy required to reach the target torque. The coefficient of correlation, which numerically estimates how good a fit the model is to the experimental data is given by:

\[ r = \sqrt{\frac{b(\sum xy - \sum x \sum y/n)}{\sum y^2 - (\sum y)^2/n}} \]  

(5.13)

Depending upon the slope of the curve \( r \) may be positive or negative. A value of 1 represents a perfect fit by the experimental data to the model, while a value of zero indicates no correlation at all.

Energy was selected as the means of predicting the endpoint rather than time because time assumes that the mixing process will continue at the same rate. However when the vulcanising system is added the ram is raised, and a significant change in the mixing rate occurs. Also with the variable speed process, the rotor speed changes, which results in changes in the mixing rate. Using energy as the endpoint can avoid these problems.

After the energy required to reach the target torque is obtained, the regression coefficient is calculated. This value must be 0.9 or greater for the program to proceed. If the value is less than 0.9 the program collects a further six pairs of torque/energy data points and performs another regression.

**Vulcanisation System Addition:**

After an acceptable regression coefficient is obtained the energy required to reach the addition point for the vulcanisation system is calculated. Initially a point 40 revolutions from the predicted end of the mixing cycle is obtained. This is then adjusted to obtain a temperature
equivalence as discussed in Section 5.1.5. The cycle then proceeds
with the batch temperature and the energy being continuously monitored.
When the total energy reaches the value calculated for the addition of
the vulcanisation system the ram is raised and the carbon black hopper
opens, depositing the crosslinking system into the Banbury. The ram
is lowered and the carbon black hopper is closed.

*Mixing Cycle Termination:*

The mixing cycle continues until the total energy level is equal to or
greater than the predicted value.

The current torque value is adjusted for temperature using equation 5.6
and compared to the value for target torque. The program stays in this
loop until the value of adjusted torque is equal to or less than the
value for the target torque. At that point the ram is raised, the gate
opens and the batch is dumped. The data which has been stored in the
local memory of the computer is transferred to a permanent file on a
floppy disc, and a summary of the times, and energy levels for the
mixing cycle are printed out. An example of the contents of such a
printout are given in Table 5.5.

5.4 PRACTICAL INVESTIGATION OF BATCH CONDITION CONTROL FOR FIXED SPEED
MIXING

5.4.1 Object of Experiment

1. To prove the hypothesis that a measurement of torque at the same
rotor speed and temperature is related to the viscosity of the
mixed rubber compound.

2. To demonstrate the control system can mix to a rheologically uni-
form endpoint.

3. To demonstrate the system can adjust the addition point of the vul-
canisation system in order to obtain heat history equivalence.
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<th>Time (secs)</th>
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<th>Power (Watts)</th>
<th>Actual Torque (N.m)</th>
<th>Total Energy (kJ)</th>
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TABLE 5.6: Compound Formulation Used in all Process Control Experiments

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</table>

Note:

It should be pointed out that this is not a typical compound. It has a high sulphur level and low accelerator level and a low oil level considering the level of the highly reinforcing carbon black used. The compound was designed to critically evaluate the control system. In practice a natural compound would not be mixed in a single cycle as has been done in this work. This is because the heat generated in a large internal mixer would be excessive and the viscosity of the resulting compound too high for subsequent forming operations. However the work was done in this manner to give the system the most rigorous evaluation.
<table>
<thead>
<tr>
<th>Time (secs)</th>
<th>Batch (°C)</th>
<th>Power (Watts)</th>
<th>Actual Torque (N.m)</th>
<th>Total Energy (kJ)</th>
</tr>
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Compound Formulation and Experimental Setpoints:

The compound formulation used in this experiment is given in Table 5.6. The experiment consisted of mixing ten batches with target torques of 295, 300, and 305 Nm. The parameters were set by mixing batches to the desired endpoint, and collecting the data. One of the set-up batches for this experiment is given in Table 5.7. The mastication in the set-up runs was for a preselected time i.e. 30 seconds. However the set-up program was arranged so that the program would not move to another phase in the mixing cycle until a voltage signal was sent down one of the analogue input lines. By following the energy/time curve of the mixing cycle it was possible to determine when to add the oil, and when to terminate the mixing cycle.

Details of Mixing Procedures:

The water cooling temperature exiting from the Banbury was maintained at 40°C. The rubber was cut into slabs approximately 2 cm thick and 10 cm wide. The rubber was placed on top of the stationary rotors at the start of each mixing cycle. The signal which starts the control program also starts the Banbury rotors turning. This technique ensures the polymer is loaded into the Banbury in a uniform manner for each mixing cycle. The sulphur, stearic acid and zinc oxide were added with the carbon black in the hopper. This ensures minimum material loss, and allows a maximum time in the Banbury to enable the best possible distribution of these ingredients throughout the rubber mix. The target torque was entered at the computer for each batch and the cycle was allowed to run to completion. When the mixed rubber compound was dumped from the Banbury, it was transferred to a stationary two roll mill. The rolls of the mill were set at approximately 0.75 cm. The mill was started, and the mixed rubber compound passed through the rolls once to convert the batch to sheet form. It was then labelled and allowed to cool.
Physical Testing:

Each sample was evaluated on the TMS rheometer, Mooney viscometer and Monsanto rheometer.

TMS Rheometer: the rheometer was operated at 100°C. The actual test conditions are given below:

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<th>Rotor Speed (rpm)</th>
<th>Time (secs)</th>
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<tr>
<td>Relaxation</td>
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Shear stress is related to shear rate and viscosity by equation 5.14.

\[ \tau = \eta_0 \gamma^n \]  

A plot of log shear stress versus log shear rate will give a linear relationship with an intercept \( \eta_0 \) and slope \( n \) which is the power law index. The data was fed into a computer program which ran a linear regression analysis and printed out the value of \( \eta_0 \) and \( n \); as well as the regression coefficient so that the quality of the data could be evaluated.

Mooney Viscometer: the test was run at 100°C using the large rotor. The test consisted of a one minute preheat, and the viscosity taken after four minutes.
Chart No.: 6
Range selected: 50
Stock #: 275
Temperature: 165°C

FIGURE 5.6
Monsanto Rheometer: the tests were run at 170°C, using a 30° arc on the rotor and the 100 mN.m range. The test was to evaluate the vulcanisation properties.

A typical rheometer curve is shown in Figure 5.6. The minimum point in the curve is referred to as the minimum viscosity. The time required for the curve to rise five points from this minimum point is referred to as the $T_5$ value. This is taken as a measure of processing safety, and gives a measure of the amount of heat history that the mixed compound can be subjected to during further forming processes, such as extruding, before it commences to vulcanise. The maximum torque reached by the curve is the measure of the crosslink density and the modulus of the compound is related to the maximum torque. The time taken to reach 95% of the value of the maximum torque is referred to as the $T_{95}$ value. This value is a measure of the optimum vulcanisation for the rubber compound.

5.4.2 Results - Fixed Speed Mixing Part 1

The results for the experiment are given in Table 5.8. A linear regression analysis was carried out on the TMS $\eta_0$ data and the Mooney viscosity data with respect to adjusted torque. The results of the regressions are given in Tables 5.9 and 5.10. The plots of the same data are given in Figure 5.7.

The regression data and linear plot of $\eta_0$ versus adjusted torque show a very good correlation between $\eta_0$ and adjusted torque. The temperature range and spread of torque values is somewhat narrow but the relationship is very definite. The relationship between the Mooney viscosity and the adjusted torque is much more tenuous. In terms of statistical confidence there is no relationship between Mooney viscosity and adjusted torque. However graphically there does appear to be a loose relationship, although the scatter is large.
TABLE 5.8: Mixing to Target Torque at Fixed Rotor Speed

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<th>Batch No.</th>
<th>Target Torque (N.m)</th>
<th>Adjusted Torque (N.m)</th>
<th>Dump Temp. (deg C)</th>
<th>Black Addition Time (secs)</th>
<th>Oil Addition Time (secs)</th>
<th>Pigment Addition Time (secs)</th>
<th>Total Mix Time (secs)</th>
<th>Predicted Energy (kJ)</th>
<th>Actual Energy (kJ)</th>
<th>$n_0$ (Pa.s)</th>
<th>$n$</th>
<th>Mooney Viscosity M1+4</th>
<th>Monsanto Rheometer @ 170°C</th>
<th>$T_{95}$ (mins)</th>
<th>$T_{95}$ (mins)</th>
<th>Relaxation Time to 75% decay</th>
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TABLE 5.9: Regression Analysis of $\eta_p$ with Respect to Adjusted Torque at Dump on BR Banbury Rotor

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Constant Term -31417.1

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TABLE 5.10: Regression Analysis of Mooney Torque with Respect to Adjusted Torque at Dump on BR Banbury Rotors

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Constant Term -42.1804

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FIGURE 5.7

NETA ZERO vs ADJUSTED TORQUE for BR BANBURY 75 rpm

MOONEY TORQUE vs ADJUSTED TORQUE for BR BANBURY 75 rpm
FIGURE 5.8: TARGET TORQUE VERSUS ADJUSTED TORQUE
Figure 5.8 is a graphical representation of the values for adjusted torque as related to the target torque. There are only two values for a 295 Nm target, and one of these is obviously a very poor match for the target. The cause was an error in the computer program which allowed the mixed compound to be discharged prematurely. If this one batch is overlooked then the control system did a creditable job of dumping the batches at the target torque selected. Table 5.11 contains the results of the regression analysis of the major variables vs the adjusted torque. The correlation matrix shows the only statistically significant relationship is between total cycle time and total energy. There are correlations, but not statistically significant between total cycle time and total energy with  \( n_0 \). The plots of total energy versus neta zero and total mix time versus neta zero are given in Figure 5.9.

5.4.3 Discussion of Results for Fixed Speed Mixing - Part I

The experiment proved that a torque measurement at a reference rotor speed and temperature was related to the \( n_0 \) value as determined by the TMS rheometer. The relationship with the Mooney torque data was much less conclusive. The probable reason for this is the Mooney viscometer operates at a very low shear rate \((1.6 \text{ sec}^{-1})\) relative to the shear rates which prevail within the internal mixer. The TMS rheometer on the other hand was operated up to 40 sec\(^{-1}\). Although this is relevant in terms of duplicating the shear rates in the internal mixer, it can lead to heat generation problems.

The heat equivalence function of the program functioned poorly. The reason for this is that the heat equivalence correction was based on the predicted endpoint. However in each case the energy required to mix the batch to the required endpoint was always greater than that predicted. A plot of the power profile and torque profile from batch 3 in this experiment is given in Figure 5.10. The actual batch data is given in Table 5.12. This shows the torque initially dropping quite rapidly and then levelling out towards the end of the cycle. The precise reason for this phenomenon is not understood. The temperature rise from the point
TABLE 5.11: Linear Regression Analysis of Adjusted Torque at Dump Versus a Series of Variables

| Variable 1 | Adjusted torque (Nm) |
| Variable 2 | Dump temperature (degrees C) |
| Variable 3 | Total cycle time (secs) |
| Variable 4 | Total energy (kJ) |
| Variable 5 | \( \eta_0 \) (Pa.s) |
| Variable 6 | \( n \) the power law constant |
| Variable 7 | \( T_{5}\) from Monsanto Rheometer (mins) |
| Variable 8 | \( T_{95}\) from Monsanto Rheometer (mins) |

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FIGURE 5.9

NETA ZERO vs TOTAL ENERGY for BR BANBURY 75 rpm

NETA ZERO vs TOTAL MIX TIME for BR BANBURY 75 rpm
FIGURE 5.10

TORQUE-TIME PROFILE - BR BANBURY

POWER PROFILE - BR BANBURY
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<td>832</td>
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<td>199.79</td>
<td>87</td>
<td>4934</td>
<td>311</td>
<td>887</td>
</tr>
</tbody>
</table>
the regression is made to the point at which the mixing cycle is termi-
nated is not sufficiently large to have any significant affect on dis-
persive mixing. As a result of this experiment it was decided to base
the termination of the mixing cycle on the energy value predicted by
the linear regression analysis.

5.5  SYSTEM DETAILS - VARIABLE SPEED

The flow chart for the variable speed batch control is given in Figure
5.11. The program naturally has many similarities to the fixed speed
program. However when the program commences it is necessary to input
the minimum and maximum rotors speeds the machine will operate at in
order to maintain the specified maximum batch temperature. It is also
necessary to specify the rotor speed to be used for the mastication
phase, and also the rotor speed for the black incorporation phase.

Mastication:

Upon receiving the signal to start the mixing cycle, the rotors commence
turning at a fixed speed specified by the operator. The ram is lowered,
compressing all of the polymer into the chamber. The computer monitors
the temperature of the polymer, and the power used by the motor of the
internal mixer. The torque on the rotors is calculated and adjusted
for temperature using equation 5.6. The torque value, adjusted for
temperature, is compared to a reference value, and when equal to or
less than the reference value the program proceeds to the next phase.

Carbon Black Addition and Incorporation:

The ram is raised, the carbon black discharged from the hopper, the ram
lowered, and the rotor speed set as specified by the operator. The
computer monitors the batch temperature and power consumption of the mixer
motor. The torque is calculated and a running average of the last three
values is maintained. As the carbon black becomes fully incorporated the
FIGURE 5.11: FLOW DIAGRAM - VARIABLE SPEED

Start
Input:
File name
Batch Cond.

Close gate
Raise ram

Check for status

Start rotors
Start timer
Lower ram

Obtain data
from A/N & calc. batch

Collect A/N for
power, calc. torque

Adjust torque to ref. temperature

Collect data in
local array

Is tor.val.
< step?

Change rotom speed, raise ram, add carbon black

Obtain batch T0

Obtain power read.
& calc. torq.

Calc. running ave. for torque

Collect data into
local array

Av. tor.<pre.
value

Read data
into local array

Average torque

Obtain power
readings

Calc. voltage
for PID con.
Adjust rotor speed

Obtain batch temperature

Energy to adsorb
Yes

Raise ram

Open hopper

Lower ram

Close hopper

Obtain batch temp.

Adjust rotor speed

Obtain power values calc. torque

Store data

Total energy pre

RS to ref.

Obtain power
and temp.

Calc. torque

Dum
torque curve passes through a maximum. This is detected when the value for the average torque is less than the preceding value. As the computer continues to monitor the mixing process, the value of torque adjusted for temperature is compared to a reference torque value. The reference torque is used to ensure a uniform state of mix prior to the addition of oil. When the temperature adjusted torque is equal to or less than the reference torque the program proceeds to the next phase of the mixing cycle.

Oil Addition and Incorporation:

The ram is raised and the oil is injected into the mixing chamber followed by an air purge of 2-3 seconds, and the ram lowered. The rotor speed is now controlled by the voltage output from the proportional-integral-derivative (PID) control. The function of the PID control system was discussed in greater detail in Section 5.1.4. The computer program monitors the batch temperature. This information is used by the PID function to adjust the rotor speed to ensure the maximum temperature specified is reached as rapidly as possible and then maintained. The program monitors the power consumption of the motor and calculates the torque, maintaining a running average of the last three values. As the oil is incorporated the torque increases and passes through a maximum. At this point the value for the average torque is smaller than the preceding value and the program moves to the next phase of the mixing cycle.

Regression Analysis and Prediction of Endpoint:

The rotor speed is fixed at the reference speed and the six values of energy and torque collected and the regression analysis run as detailed in Section 5.3. After the program has confirmed the regression slope is negative and the regression coefficient is satisfactory, the energy required to mix to the desired rheological endpoint is calculated and the program moves to the next phase of the mixing cycle.
**Vulcanisation System Addition:**

The initial point at which the vulcanisation system will be added is calculated as 40 rotor revolutions from the predicted cycle termination point. Using the theory given in Section 5.1.5, the heat equivalence is calculated. For determining the reference heat history, the rotor speed is assumed to be an average of the minimum and maximum values inputted by the operator. Having determined the point at which the vulcanisation system will be added the program continues to monitor the batch temperature, automatically adjusts the rotor speed, monitors the power consumption and calculates the torque. When the total energy value is equal to or greater than the value for the addition of the vulcanisation system the ram is raised, and the crosslinking system is added through the black hopper. The ram is lowered and the program moves onto the next phase of the mixing cycle.

**Mixing Cycle Termination:**

The mixing cycle continues, with the computer monitoring the batch temperature, and adjusting the rotor speed by means of the PID function to maintain the batch temperature at the temperature specified as the maximum. The power is monitored and torque calculated. At the point when the total energy is equal to or greater than the energy level determined by the regression analysis, the rotor speed is fixed at the reference rotor speed. There is a 3 second delay to allow the rotor speed to come to an equilibrium condition, and then the batch temperature and power consumption are measured. The torque is calculated and adjusted for temperature, the ram is raised, gate opened and cycle terminated. All the data stored in the local memory is transferred to floppy discs and a summary of the mixing cycle and data is printed out.
5.6 PRACTICAL INVESTIGATION OF BATCH CONDITION CONTROL FOR VARIABLE SPEED MIXING - PART 1

5.6.1 Experimental Details of an Experiment Using SMR10 and SMR20

Object of Experiment:
To mix a compound from two different grades of natural rubber (SMR10 and SMR20), to determine if the batch condition control system could compensate for any rheological differences between the polymers.

Compound Formulation and Experimental Design:
The compound used is the one given in Table 5.6. The natural rubber consisted of combinations of SMR10 and SMR20, the details of which are in Table 5.13.

Mixing Procedures:
The variable speed control program was used. The polymers were cut into pieces approximately 2 cm thick and 10 cm wide. The SMR20 was put into the internal mixer first, with the rotors stationary. The program was started by a remote starting device at the Banbury. Upon the completion of the mixing cycle the mixed rubber compound was removed and placed on a stationary mill. The rubber compound was passed through the mill rolls once, and sheeted out to a thickness of approximately 0.75 cm. The batches were mixed in a random sequence. Approximately 30 minutes was allowed to elapse between each mixing cycle to enable the Banbury to come to thermal equilibrium.

Physical Testing:
Each sample was evaluated on the TMS rheometer, Monsanto rheometer and carbon black dispersion tester.
TABLE 5.13: Details of the First Variable Speed Experiment Using Various Levels of SMR10 and SMR20

<table>
<thead>
<tr>
<th>Batch Number</th>
<th>Level of SMR10</th>
<th>Level of SMR20</th>
<th>Weight SMR10</th>
<th>Weight SMR20</th>
<th>Mixing Sequence</th>
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<tbody>
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<td>0</td>
<td>654.5</td>
<td>0</td>
<td>4</td>
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<tr>
<td>2</td>
<td>0.9</td>
<td>0.1</td>
<td>589.05</td>
<td>65.45</td>
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<td>3</td>
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<td>0.2</td>
<td>523.6</td>
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<td>10</td>
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<tr>
<td>4</td>
<td>0.7</td>
<td>0.3</td>
<td>458.15</td>
<td>196.35</td>
<td>1</td>
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<tr>
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<td>392.7</td>
<td>261.8</td>
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<td>0.5</td>
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<td>327.25</td>
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<tr>
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<td>0.4</td>
<td>0.6</td>
<td>261.8</td>
<td>392.7</td>
<td>7</td>
</tr>
<tr>
<td>8</td>
<td>0.3</td>
<td>0.7</td>
<td>196.35</td>
<td>458.15</td>
<td>2</td>
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<tr>
<td>9</td>
<td>0.2</td>
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<td>130.9</td>
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<td>0.1</td>
<td>0.9</td>
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<td>689.05</td>
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<tr>
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<td>0</td>
<td>1.0</td>
<td>0</td>
<td>654.5</td>
<td>3</td>
</tr>
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</table>

Process conditions:
- Cooling water exit temperature 40°C
- Maximum rotor speed 80 rpm
- Minimum rotor speed 35 rpm
- Fill factor 0.65

Setpoint conditions:
- Mastication 300 Nm @ 75 rpm and 45°C
- Dump 265 Nm @ 75 rpm and 100°C
- Maximum batch temperature 90°C
The TMS rheometer was operated at 100°C as follows:

<table>
<thead>
<tr>
<th>Rotor speed (rpm)</th>
<th>Duration (secs)</th>
</tr>
</thead>
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<tr>
<td>1</td>
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<td>3</td>
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<tr>
<td>5</td>
<td>60</td>
</tr>
<tr>
<td>7</td>
<td>60</td>
</tr>
<tr>
<td>10</td>
<td>45</td>
</tr>
<tr>
<td>Relax</td>
<td>60</td>
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</table>

A regression analysis was run with log shear rate as the independent variable, and log shear stress as the dependent variable. The value for \( n_0 \) (viscosity at 1 sec\(^{-1}\)) and the power law index \( n \) was calculated, as well as the regression coefficient. If the regression coefficient was below 98% the test was rerun. The shear rate was reduced from a maximum of 40 sec\(^{-1}\) to 10 sec\(^{-1}\) because of a control problem with the shear rate on the TMS rheometer.

The Monsanto rheometer was operated at 170°C, 100 range and a 30 arc on the rotor. The values for \( T_5 \) and \( T_{95} \) were obtained from each curve as well as the minimum and maximum torques.

### 5.6.2 Results and Discussion

The results are summarised in Table 5.14, and a summary of the linear regression results of various functions with respect to the level of SMR10 are in Table 5.15.

A review of the mastication times indicates no set pattern, which one might expect if there was a significant difference in rheological properties between the two grades of natural rubber.

The TMS data for the two polymers at 100°C is given below:
TABLE 5.14: Results of the First Variable Speed Experiment Evaluating Various Levels of SMR10 and SMR20

<table>
<thead>
<tr>
<th>Compound Number</th>
<th>Feature SR10/SR20</th>
<th>Adjusted Torque (N.m)</th>
<th>Target T°C</th>
<th>Dump to Black Addition Time (secs)</th>
<th>Black Addition Time (secs)</th>
<th>Oil Addition Time (secs)</th>
<th>Pigment Addition Time (secs)</th>
<th>Total Mix Time (secs)</th>
<th>Predicted Energy (kJ)</th>
<th>Actual Energy (kJ)</th>
<th>$n_0$ (Pa.s)</th>
<th>n</th>
<th>170°C</th>
<th>Relaxation Time 75% Delay</th>
<th>Mean Peak</th>
<th>Standard Deviation</th>
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<td>1</td>
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<td>21.6</td>
<td>54.7</td>
<td>111.2</td>
<td>147.5</td>
<td>529.3</td>
<td>548.7</td>
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<td>-63.63</td>
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<td>2.20</td>
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<td>63.8</td>
<td>118.2</td>
<td>143.3</td>
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<td>485.6</td>
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<td>88.9</td>
<td>37.1</td>
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<td>152</td>
<td>527</td>
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<td>-57.07</td>
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<td>52</td>
<td>115.7</td>
<td>140.9</td>
<td>446.4</td>
<td>487.6</td>
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<td>22.0</td>
<td>4.41</td>
<td>2.41</td>
<td>60.478</td>
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### TABLE 5.15: Linear Regression Analysis of Experimental Results with Respect to Level of SMR10

<table>
<thead>
<tr>
<th>Variable</th>
<th>t-value</th>
<th>Confidence Level %</th>
<th>Intercept</th>
<th>Slope</th>
<th>Mean</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adjusted torque</td>
<td>.4209</td>
<td>13.9</td>
<td>264.941</td>
<td>2.8638</td>
<td>266.373</td>
<td>6.83557</td>
</tr>
<tr>
<td>Carbon black addition time</td>
<td>.8601</td>
<td>27.56</td>
<td>31.646</td>
<td>-4.2182</td>
<td>29.5364</td>
<td>5.07627</td>
</tr>
<tr>
<td>Total mix time</td>
<td>.7595</td>
<td>24.54</td>
<td>145.886</td>
<td>8.8817</td>
<td>150.327</td>
<td>12.0027</td>
</tr>
<tr>
<td>Total energy</td>
<td>1.2331</td>
<td>38.02</td>
<td>501.991</td>
<td>60.509</td>
<td>532.245</td>
<td>52.7883</td>
</tr>
<tr>
<td>$n_0$</td>
<td>0.8082</td>
<td>26.01</td>
<td>794.519</td>
<td>760.284</td>
<td>79796</td>
<td>969.341</td>
</tr>
<tr>
<td>Monsanto rheometer $T_{95}$</td>
<td>0.3469</td>
<td>11.49</td>
<td>4.444</td>
<td>-0.06091</td>
<td>4.41364</td>
<td>0.175851</td>
</tr>
<tr>
<td>Relaxation time</td>
<td>1.9731</td>
<td>54.95</td>
<td>13.5909</td>
<td>-4.4546</td>
<td>11.3636</td>
<td>2.68859</td>
</tr>
<tr>
<td>Power law index $n$</td>
<td>3.8562</td>
<td>78.93</td>
<td>0.133545</td>
<td>-0.0134546</td>
<td>.126818</td>
<td>5.65371E-03</td>
</tr>
<tr>
<td>Monsanto rheometer max torque</td>
<td>0.9082</td>
<td>28.98</td>
<td>79.9409</td>
<td>-1.93634</td>
<td>78.9727</td>
<td>2.21642</td>
</tr>
<tr>
<td>Carbon black dispersion</td>
<td>2.9539</td>
<td>70.16</td>
<td>-59.9369</td>
<td>-4.21329</td>
<td>-62.0436</td>
<td>1.99168</td>
</tr>
</tbody>
</table>
As can be seen there is no difference in the $\eta_0$ value of the two polymers although the SMR10 is slightly more elastic than the SMR20.

The assumption had been made that the SMR10 with a lower dirt content than the SMR20 would be subjected to a more intensive cleansing process. This it was felt would translate into a material with lower molecular weight and therefore lower viscosity. However the SMR10 is prepared from a cleaner grade of latex and which does not result in a lower viscosity polymer. The variation in the mastication is therefore due to random variations within the polymer and within the process variables. However the variation within the $\eta_0$ values for the mixed rubber compound is random, with an excellent grouping. Figure 5.12 shows clearly the narrow range of $\eta_0$ values for the mixed compound.

The relaxation data, although not statistically significant, does show a trend of slightly increasing elasticity as the amount of SMR20 increases. This is rather interesting because the data on the raw polymers showed the SMR10 to be the more elastic of the two polymers. Figure 5.13 illustrates the frequency distributions of four measured parameters. The distribution for a five point rise from the minimum value for the Monsanto rheometer curve is excellent. This indicates the point at which the vulcanisation process is initiated, and is an indication of the heat history the vulcanisation system has been subjected to. In this context it is extremely interesting to note the large range in mixing times and mixing energies relative to the rheological properties of the mixed compound. Referring to Table 5.13 the mean for $\eta_0$ is 79796 with a standard deviation of 969.341 or ±1.215%. The mean for the total energy is 532.245 with a standard deviation of 52.7883 or 9.918%.
LEVEL OF SMR10 vs NETA ZERO

LEVEL OF SMR10 vs RELAXATION TIME

FIGURE 5.12
FIGURE 5.13: FREQUENCY DISTRIBUTIONS OF VARIOUS PARAMETERS FOR FOR SMR 10/SMR 20 EXPERIMENT
Therefore it is reasonable to conclude that the variation in total energy is a result of adjusting the mixing specifications to correct for mild variations in the raw materials and the process.

A review of the carbon black dispersion data indicates small differences in the carbon black dispersion. From the statistical analysis it can be seen that the relationship with the SMR10 level is not significant. There is however a trend which indicates the carbon black dispersion improves slightly as the level of SMR10 decreases. This is in the same direction as increasing elasticity and therefore is more likely to represent surface roughness due to viscoelastic effects than a change in carbon black dispersion. The dark field reflected light microscope technique used to determine carbon black dispersion is wholly dependent on surface texture.

It is possible that when testing unvulcanised mixed rubber compounds based on highly elastic polymers like natural rubber, the surface smoothness will be affected by the viscoelastic properties of the compound.

5.6.3 Conclusions

The batch condition control system produced 11 batches from varying amounts of SMR10 and SMR20. The random material variations were modified, resulting in mixed compounds with uniform rheological properties. The function for adjusting the heat equivalence of the vulcanisation system was satisfactory. The maximum torque on the rheometer, which is related to sulphur level, was variable, which caused a larger spread in the $T_{95}$ values than was obtained in the $T_{5}$ values. This is difficult to explain when the sulphur is added early in the mixing cycle with the carbon black, and therefore should be well dispersed.
5.7 PRACTICAL INVESTIGATION OF BATCH CONDITION CONTROL FOR VARIABLE SPEED MIXING - PART 2

5.7.1 Experimental Details of an Experiment Using CV5 and SMR20

Object of Experiment:
To mix a compound from two different grades of natural rubber (CV5 and SMR20), to determine if the batch condition control system could compensate for the rheological differences between the polymers.

Compound Formulation and Experimental Design:
The compound used is given in Table 5.6. The natural rubber consisted of combinations of CV5 and SMR20; the details of which are in Table 5.16. The rheological properties of the two polymers were evaluated using the TMS rheometer. The results obtained are listed below:

<table>
<thead>
<tr>
<th></th>
<th>( \eta_0 ) (Pa.s)</th>
<th>Relaxation Time (secs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CV5</td>
<td>69666</td>
<td>7.4</td>
</tr>
<tr>
<td>SMR20</td>
<td>105550</td>
<td>62</td>
</tr>
</tbody>
</table>

Mixing Procedures:
The procedures are the same as those given in Section 5.6.1.

Physical Testing:
The same tests were carried out as outlined in Section 5.6.1.
TABLE 5.16: Experimental Details for Second Variable Speed Experiment Using Various Levels of CV5 and SMR20

<table>
<thead>
<tr>
<th>Batch Number</th>
<th>Level of CV5</th>
<th>Level of SMR 20</th>
<th>Weight of CV5</th>
<th>Weight of SMR 20</th>
<th>Mixing Sequence</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>0</td>
<td>654.5</td>
<td>0</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>.9</td>
<td>.1</td>
<td>589.05</td>
<td>65.45</td>
<td>8</td>
</tr>
<tr>
<td>3</td>
<td>.8</td>
<td>.2</td>
<td>523.6</td>
<td>130.9</td>
<td>10</td>
</tr>
<tr>
<td>4</td>
<td>.7</td>
<td>.3</td>
<td>458.15</td>
<td>196.35</td>
<td>1</td>
</tr>
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<td>5</td>
<td>.6</td>
<td>.4</td>
<td>392.7</td>
<td>261.8</td>
<td>6</td>
</tr>
<tr>
<td>6</td>
<td>.5</td>
<td>.5</td>
<td>327.25</td>
<td>327.25</td>
<td>11</td>
</tr>
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<td>7</td>
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<td>.6</td>
<td>261.8</td>
<td>392.7</td>
<td>7</td>
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<td>1</td>
<td>0</td>
<td>654.50</td>
<td>3</td>
</tr>
</tbody>
</table>

Process conditions:

- Cooling water exit temperature: 30°C
- Maximum rotor speed: 80 rpm
- Minimum rotor speed: 35 rpm
- Fill factor: .65

Setpoint conditions:

- Mastication: 250 Nm @ 45°C and 75 rpm
- Dump: 265 Nm @ 100°C and 75 rpm
- Maximum batch temperature: 80°C
5.7.2 Results and Discussion

A summary of the results are given in Table 5.17.

A review of the data shows that the minimum time in which the control system is able to react and raise the ram for carbon black addition is 14.3 secs. It was found that the first four batches with the highest level of CV5 were masticated to below the target torque for mastication in less than 14.3s. This means for those batches the control system has been unable to modify the polymers successfully. This is illustrated quite clearly by Figure 5.14a which depicts black addition time versus level of CV5. As one would expect the replacement of CV5 with SMR20 produces a polymer blend with higher average viscosity. This means the work inputted to the polymer in order to obtain the desired target torque increases as the level of CV5 decreases. Because the control system was unable to control the mastication of the first three polymer blends successfully, the linear regression analysis on the results was made on the last eight batches only.

The results of the linear regression analysis are summarised in Table 5.18. The relationship of carbon black addition time with level of CV5 is confirmed by the statistical analysis, although the experimental scatter is reflected in the confidence level of 84.26%. The correlation between \( n_0 \) and level of CV5 is completely random, which indicates the control system is functioning and modifying the polymers. However the spread of the \( n_0 \) values for the mixed compound are higher than the previous experiment with a coefficient of variance of 2.895%. The analysis of the relaxation data is very interesting since it shows no correlation with the level of CV5. A review of this data in Table 5.17 shows the relaxation times are quite variable but random. When one considers the difference in the relaxation times of the two raw polymers, the results are excellent. The analysis of level of CV5 with respect to total energy indicates a definite relationship, but with a confidence level of 89.12% the precise terms of the relationship must be treated with some caution. However if the control system works, it must put more work
<table>
<thead>
<tr>
<th>Compound No.</th>
<th>Feature</th>
<th>Adjusted Torque (N.m)</th>
<th>Target To (°C)</th>
<th>Dump To (°C)</th>
<th>Black Addition Time (secs)</th>
<th>Oil Addition Time (secs)</th>
<th>Total Mix Time (secs)</th>
<th>Predicted Energy (kJ)</th>
<th>Actual Energy (kJ)</th>
<th>n</th>
<th>( n_o ) (Pa.s)</th>
<th>170°C</th>
<th>M100</th>
<th>144</th>
<th>Relaxation Time</th>
<th>Carbon black dispersion</th>
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<td>1</td>
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<td>124.2</td>
<td>149.4</td>
<td>347.6</td>
<td>494.7</td>
<td>69814</td>
<td>.0905</td>
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<td>71.3</td>
<td>4.26</td>
<td>67.1</td>
</tr>
<tr>
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<td>0.9/0.1</td>
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<td>14.4</td>
<td>54</td>
<td>112.8</td>
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<td>419.2</td>
<td>487.6</td>
<td>77402</td>
<td>.0902</td>
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<td>6.1</td>
</tr>
<tr>
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<td>81.2</td>
<td>14.4</td>
<td>54</td>
<td>112.8</td>
<td>138</td>
<td>419.2</td>
<td>487.6</td>
<td>77402</td>
<td>.0902</td>
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<td>4.2</td>
<td>76.7</td>
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<td>53.9</td>
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<td>415.5</td>
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<td>81734</td>
<td>.105</td>
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<td>83.8</td>
<td>8.2</td>
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<td>73</td>
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<tr>
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<td>83.5</td>
<td>20.8</td>
<td>45.1</td>
<td>117.6</td>
<td>142.8</td>
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<td>80078</td>
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<td>42.9</td>
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<td>79</td>
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<td>528.6</td>
<td>78879</td>
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<td>22.0</td>
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<td>4.37</td>
<td>81.2</td>
<td>8.7</td>
</tr>
<tr>
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<td>0.1/0.9</td>
<td>279.6</td>
<td>78.5</td>
<td>29.3</td>
<td>64.4</td>
<td>119</td>
<td>197.6</td>
<td>636.5</td>
<td>648.5</td>
<td>79367</td>
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<td>69.0</td>
<td>4.32</td>
<td>81.4</td>
<td>3.2</td>
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<tr>
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<td>0/1.0</td>
<td>279.2</td>
<td>80.5</td>
<td>33.5</td>
<td>68.7</td>
<td>123</td>
<td>168.2</td>
<td>608.6</td>
<td>620.5</td>
<td>79118</td>
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<td>22.3</td>
<td>80</td>
<td>4.05</td>
<td>86.1</td>
<td>9.6</td>
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</tbody>
</table>

* Adjusted to ref temperature

**TABLE 5.17:** Results of the Second Variable Speed Experiment Evaluating Various Levels of CV5 and SMR20
NETA ZERO vs LEVEL OF CV5

BLACK ADDITION TIME vs LEVEL OF CV5

FIGURE 5.14
TABLE 5.18: Linear Regression Analysis of Experimental Results with Respect to Level of CV5

<table>
<thead>
<tr>
<th>Variable</th>
<th>t-value</th>
<th>Confidence Level %</th>
<th>Intercept</th>
<th>Slope</th>
<th>Mean</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adjusted torque</td>
<td>0.197</td>
<td>8</td>
<td>280.35</td>
<td>4.071</td>
<td>281.775</td>
<td>12.463</td>
</tr>
<tr>
<td>Carbon black addition time</td>
<td>3.832</td>
<td>84.26</td>
<td>31.0925</td>
<td>-21.2928</td>
<td>23.63</td>
<td>6.190</td>
</tr>
<tr>
<td>Total mix time</td>
<td>1.547</td>
<td>53.39</td>
<td>181.742</td>
<td>-67.7262</td>
<td>158.037</td>
<td>31.073</td>
</tr>
<tr>
<td>Total energy</td>
<td>4.347</td>
<td>87.12</td>
<td>632.675</td>
<td>-261.607</td>
<td>541.113</td>
<td>73.56</td>
</tr>
<tr>
<td>(n_0)</td>
<td>0.813</td>
<td>31.51</td>
<td>78683.2</td>
<td>2969.53</td>
<td>79722.5</td>
<td>2308.06</td>
</tr>
<tr>
<td>Power law index (n)</td>
<td>2.284</td>
<td>68.2</td>
<td>0.1206</td>
<td>-0.03055</td>
<td>0.10995</td>
<td>0.01097</td>
</tr>
<tr>
<td>Monsanto rheometer (T_{95})</td>
<td>2.97</td>
<td>77.11</td>
<td>4.1675</td>
<td>0.482144</td>
<td>4.336</td>
<td>1.5315</td>
</tr>
<tr>
<td>Relaxation time</td>
<td>0.11</td>
<td>4.49</td>
<td>7.25</td>
<td>0.428568</td>
<td>7.4</td>
<td>2.339</td>
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<tr>
<td>Monsanto rheometer max. torque</td>
<td>0.189</td>
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<td>73.8083</td>
<td>1.012</td>
<td>75.1625</td>
<td>3.227</td>
</tr>
<tr>
<td>Carbon black dispersion</td>
<td>6.56x10^{-3}</td>
<td>&gt;1</td>
<td>54.425</td>
<td>-0.078583</td>
<td>-54.4525</td>
<td>7.183</td>
</tr>
</tbody>
</table>
Figure 5.15:

(a) NETA ZERO vs ADJUSTED TORQUE

(b) TOTAL ENERGY vs LEVEL OF CV5
TABLE 5.19: Regression Analysis of Torque at Dump as \( \eta_0 \)

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### Multiple R-squared .787862

### Standard Error of Estimate 1743.08

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Constant term 26468.2

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into higher viscosity polymers to modify them and meet the desired mastication targets. This relationship shows the control system is doing just that. The relationship is shown in Figure 5.15a.

Table 5.19 is a regression analysis of $\eta_0$ for the final mixed stock vs adjusted torque at dump. The confidence level of 88.76% shows a strong relationship between the two parameters. This is represented graphically in Figure 5.15b. It demonstrates the experimental variability one can expect when basing data on a single torque value at the end of a mixing cycle. However it clearly shows there is a definite relationship between torque at reference rotor speed and temperature and basic rheological properties.

5.7.3 Conclusions

The experiment has demonstrated the control system is capable of producing a product with a uniform endpoint despite large variations in the rheological properties of the raw polymers.

The relationship between torque at a reference rotor speed and temperature to basic rheological properties has been confirmed again.

5.8 PRACTICAL INVESTIGATION OF BATCH CONDITION CONTROL FOR VARIABLE SPEED MIXING - PART 3

5.8.1 Experimental Details of an Experiment which Evaluates the Batch Condition Control Versus Unit Work

Object of Experiment:

Unit work has been shown\(^1\) to produce mixed rubber compound with more uniform rheological properties than other methods of controlling the mixing cycle. Therefore it was selected as the best technique to use for comparison with the batch condition control system.
**Compound Formulation and Experimental Design:**

The compound used is given in Table 5.6. The experimental design consists of two variables (cooling water temperature and target batch temperature), each at three levels. The details are given in Table 5.20. Each point was run in duplicate for a mixing specification based on unit work and also for a mixing specification utilising the batch condition control. The mixing cycle setpoints used are given in Table 5.21. The initial experiment had the batch temperature targets as 80, 90 and 100°C. However it became evident that 100°C was not possible and so the limits were changed to 70, 80 and 90°C. For this reason there are additional batches included in the results.

**Mixing Procedures:**

The mixing procedures are as described in Section 5.6.1. All the mixes from both the batch condition control and the mixing specification using unit work were treated as one group and mixed in a random order. This is shown in Table 5.20 which includes the extra mixes that were prepared as a result of changing the experimental setpoints for batch temperature. The batches were mixed over a three day period. This ensured that approximately thirty minutes was left between each cycle to allow the Banbury to achieve thermal equilibrium.

**Physical Testing:**

The testing procedures are as described in Section 5.6.1. The Mooney torque tests were carried out at the Avon Rubber Company.

**5.8.2 Results and Discussion**

The total results are summarised in Tables 5.22 and 5.23. The regression analysis on the experimental results is given in Tables 5.24 and 5.25.
TABLE 5.20: Experimental Design for Experiment 3 - Variable Speed

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Mixing Cycle Setpoints:

i) **Batch condition control**
   - Mastication: 250 Nm @ 45°C and 75 rpm
   - Dump: 265 Nm @ 100°C and 75 rpm

ii) **Unit work**
   - Black addition: 60 kJ
   - Oil addition: 210 kJ
   - Pigment addition: 400 kJ
   - Dump: 520 kJ

Process Conditions:
- Cooling water exit temperature: 40°C
- Maximum rotor speed: 80 rpm
- Minimum rotor speed: 20 rpm
TABLE 5.21: Details of Actual Experimental Setpoints

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<td>563.8</td>
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<td>73.1</td>
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</table>

Coefficient of Variability for the Test

|               | .86% | 2.45% | .79% | .99% | 2.92% | 1.39% | 7.35% |

**Notes:**
- The table represents the data collected for variable speed process control in a manufacturing setting.
- The columns include various process parameters and outputs measured using a Monsanto Rheometer.
- The final columns provide relaxation temperature, cooling time, and batch temperature.
- Coefficient of Variability indicates the consistency of the test results.
| Exp No. | Feature Code | Target Temp (°C) | Target n° (N.m) | Dump T°C | OIl Addition Time (secs) | Pigment Addition Time (secs) | Total Mix Time (secs) | Predicted Energy (kJ) | Actual Energy (kJ) | n_0 (Pa.s) | a | b | c | d | e | f | g | h | i | j | k | l | m | n | o | p | q | r | s | t | u | v | w | x | y | z |
| 1      | 40           | 80              | 82.2            | 37.1     | 65.1                      | 117.1                      | 173.2                    | 521.9                | 78144               | .129         | 22.0 | 72.9 | 2.40 | 4.73 | 87.9 | 11.0 | -56.04 |
| 2      | 40           | 90              | 84              | 62.2     | 90.4                      | 135.8                      | 171.1                    | 538                 | 78606               | .134         | 22.1 | 74.9 | 2.31 | 4.41 | 86.9 | 14.5 | -58.34 |
| 3      | 40           | 100             | 89.2            | 36       | 65.1                      | 108.3                      | 143.6                    | 540.5               | 80586               | .125         | 22.1 | 75.7 | 2.44 | 4.57 | 89.2 | 11.4 | -66.67 |
| 4      | 30           | 80              | 75.2            | 37.1     | 65.2                      | 106.3                      | 141.7                    | 541.2               | 78793               | .130         | 22.3 | 79    | 2.30 | 4.20 | 89.4 | 12.0 | -52.82 |
| 5      | 30           | 90              | 81.0            | 67.3     | 112.7                     | 148.2                      | 358.5                    | 77440              | .124         | 22.1 | 75.5 | 2.35 | 4.29 | 87.2 | 9.9  | -64.22 |
| 6      | 30           | 70              | 70.6            | 33.9     | 59.8                      | 76.7                      | 164.4                    | 537.5               | 78531               | .130         | 22.3 | 78.1 | 2.37 | 4.31 | 91.1 | 12.9 | -57.76 |
| 7      | 20           | 80              | 64.3            | 37       | 62.9                      | 104                       | 137.2                    | 539                 | 79467               | .130         | 23    | 74    | 2.35 | 4.77 | 91.9 | 10.9 | -65.63 |
| 8      | 20           | 70              | 70.6            | 37.1     | 63                       | 104.1                     | 135.1                    | 533.4               | 79337               | .132         | 23.7 | 84.2 | 2.19 | 3.90 | 90.4 | 9.5  | -57.66 |
| 9      | 20           | 70              | 72.0            | 30.8     | 59                       | 100                       | 131.3                    | 531.7               | 78198               | .119         | 22.1 | 80    | 2.3  | 4.07 | 87.5 | 10.8 | -55.35 |
| 10     | 40           | 80              | 83.0            | 34       | 62.1                      | 107.5                     | 157.9                    | 534.3               | 79595               | .133         | 22.9 | 74.1 | 2.37 | 4.57 | 88.9 | 14.8 | -62.51 |
| 11     | 40           | 90              | 86.9            | 34       | 62.1                      | 105.4                     | 140.7                    | 535.7               | 78384               | .133         | 21.9 | 76.9 | 2.36 | 4.68 | 89.8 | 11.3 | -60.09 |
| 12     | 40           | 100             | 89.3            | 34       | 64.2                      | 109.5                     | 144.8                    | 532.4               | 76290               | .128         | 21.2 | 78    | 2.35 | 4.35 | 85.2 | 13.2 | -60.71 |
| 13     | 30           | 80              | 75.9            | 34       | 60                       | 103.1                     | 136.3                    | 536.9               | 77792               | .130         | 21.4 | 74.4 | 2.34 | 4.39 | 88.5 | 14.0 | -60.07 |
| 14     | 30           | 90              | 76.5            | 34       | 62.1                      | 103.3                     | 136.7                    | 535.7               | 77832               | .125         | 23.3 | 79.0 | 2.35 | 4.39 | 87.7 | 11.2 | -62.67 |
| 15     | 30           | 80              | 69.8            | 90       | 124.4                     | 167.7                     | 213.8                    | 540.5               | 76225               | .122         | 21.9 | 74.8 | 2.37 | 4.52 | 86.1 | 13.5 | -61.58 |
| 16     | 30           | 70              | 67.5            | 40.1     | 68.2                      | 109.3                     | 140.4                    | 532.4               | 76057               | .139         | 23.9 | 81    | 2.30 | 4.11 | 87.2 | 10.8 | -60.98 |
| 17     | 30           | 90              | 76.9            | 55.9     | 81.8                      | 122.9                     | 156.2                    | 538.1               | 76707               | .126         | 22.7 | 82.2 | 2.20 | 3.93 | 85.9 | 8.7  | -61.18 |
| 18     | 30           | 70              | 73.3            | 57       | 65.1                      | 153.6                     | 270.8                    | 532                 | 76520               | .125         | 21    | 74.9 | 2.15 | 4.51 | 83.5 | 13.6 | -52.13 |
| 19     | 30           | 70              | 70.4            | 30.9     | 59                       | 141.2                     | 245.6                    | 531.9               | 76598               | .119         | 20.9 | 74.0 | 2.35 | 4.33 | 82.1 | 7.6  | -52.04 |
| 20     | 30           | 90              | 71.3            | 33.9     | 62                       | 103.2                     | 136.3                    | 537.9               | 78379               | .128         | 23    | 76    | 2.35 | 4.44 | 89.1 | 14.6 | -60.42 |
| 21     | 30           | 60              | 70.7            | 37       | 63                       | 106.3                     | 139.3                    | 539.8               | 76718               | .125         | 22    | 75.6 | 2.50 | 4.40 | 84.0 | 13.1 | -58.88 |

Coefficient of variability (SD/mean) for the test: 0.26%, 0.9%, 0.96%, 1.63%, 3.43%, 1.45%, 5.59%
<table>
<thead>
<tr>
<th>Function</th>
<th>% GM†</th>
<th>Cooling Water</th>
<th>Batch Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Estimate</td>
<td>SE**</td>
</tr>
<tr>
<td>Adjusted torque</td>
<td>275</td>
<td>5.93</td>
<td>4.17</td>
</tr>
<tr>
<td>Black Addition Time</td>
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<td>-1.808</td>
<td>1.247</td>
</tr>
<tr>
<td>Oil Addition Time</td>
<td>58.17</td>
<td>-5.982</td>
<td>2.195</td>
</tr>
<tr>
<td>Pigment Addition Time</td>
<td>115.7</td>
<td>-7.412</td>
<td>2.620</td>
</tr>
<tr>
<td>Total Mix Time</td>
<td>164.4</td>
<td>30.37</td>
<td>8.601</td>
</tr>
<tr>
<td>Total Energy</td>
<td>524.1</td>
<td>-33.26</td>
<td>-16.08</td>
</tr>
<tr>
<td>( n_0 )</td>
<td>77830*</td>
<td>445.2</td>
<td>288.3</td>
</tr>
<tr>
<td>( n )</td>
<td>127185</td>
<td>3.263E-03</td>
<td>2.3278E-03</td>
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<tr>
<td>Monsanto Rheometer ( T_{95} )</td>
<td>4.495</td>
<td>0.2976E-01</td>
<td>0.6277E-01</td>
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<tr>
<td>( M_{LL-4} )</td>
<td>87.25</td>
<td>.1883</td>
<td>.9871</td>
</tr>
<tr>
<td>Relaxation Time</td>
<td>12.18</td>
<td>.1081</td>
<td>.7173</td>
</tr>
<tr>
<td>Carbon Black Dispersion</td>
<td>-60.1653</td>
<td>.9604</td>
<td>1.2124</td>
</tr>
</tbody>
</table>

For 95% probability \( t = 2.120 \)

For 90% probability \( t = 1.746 \)

\[
\frac{t}{S.E.} = \frac{\text{Estimate}}{S.E.}
\]

*Coefficient of variation 1.01%
†Grand mean
‡Standard error
TABLE 5.25: Coefficients of Model for Various Responses for Unit Work

df = 15

<table>
<thead>
<tr>
<th>Function</th>
<th>% gm t</th>
<th>Cooling Water</th>
<th></th>
<th>Batch Temperature</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Estimate</td>
<td>SE††</td>
<td>Estimate</td>
<td>SE</td>
</tr>
<tr>
<td>Adjusted torque</td>
<td>266.5</td>
<td>-6.989</td>
<td>4.526</td>
<td>-3.040</td>
<td>5.318</td>
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<td>Black addition time</td>
<td>40.77</td>
<td>6.251</td>
<td>8.926</td>
<td>-6.207</td>
<td>10.49</td>
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<td>Oil addition time</td>
<td>70.46</td>
<td>5.702</td>
<td>9.751</td>
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<td>Pigment addition time</td>
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<td>4.019</td>
<td>12.01</td>
<td>-.5782</td>
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<td>Total mix time</td>
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<td>-2.797</td>
<td>14.13</td>
<td>10.14</td>
<td>16.59</td>
</tr>
<tr>
<td>( \eta_0 )</td>
<td>78410*</td>
<td>-475.6</td>
<td>806.5</td>
<td>737</td>
<td>947.4</td>
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<tr>
<td>n</td>
<td>.13015</td>
<td>-2.433E-03</td>
<td>1.536E-03</td>
<td>3.169E-03</td>
<td>1.664E-03</td>
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<td>Monsanto rheometer ( T_g )</td>
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<td>.1424</td>
<td>.1576</td>
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<td>1.523</td>
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<td>1.79</td>
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<td>Relaxation time</td>
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<td>1.290</td>
<td>.8741</td>
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<td>Carbon black dispersion</td>
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<td>2.83483</td>
<td>1.20555</td>
<td>-3.09351</td>
<td>1.30614</td>
</tr>
</tbody>
</table>

For 95% probability \( t = 2.131 \)
For 90% probability \( t = 1.753 \)

\*Coefficient of variation 1.45%
†Grand mean
‡‡Standard error
The adjusted torque, \( n_0 \), power law index, Monsanto rheometer data, Mooney torque, relaxation time and carbon black dispersion are all independent of cooling water and batch temperature for both types of mixing specifications. However it is interesting to note that for the batch condition control system the crosslinking system addition time, total mix time and total energy have a dependence on cooling water temperature and batch temperature. The crosslinking system addition time should be dependent on the two variables if an equivalent heat history is to be obtained. The dependence on cooling water temperature and batch temperature by the total mixing time, and total energy, indicates that the processing conditions have an influence on the effectiveness of the energy input into the mixed compound.

The mastication time shows a relationship to the temperature of the cooling water, but not good enough to be statistically significant.

The relationship of the oil addition time with the cooling water temperature is statistically significant. This is related to the rate of carbon black incorporation and dispersion and indicates that this process is affected by the cooling water temperature. The graphs which show the relationship of oil addition time, pigment addition time (crosslinking system) and total energy with the two process variables are given in Figures 5.16, 5.17 and 5.18.

Figure 5.16 shows the relationship of the oil addition time with cooling water temperature. The oil addition time decreases as the cooling temperature increases. Figure 5.17 shows the crosslinking system addition time decreases as the cooling water temperature increases which agrees with the faster mixing rate shown in Figure 5.16. However the crosslinking system addition time shows an initial decrease and then a very sharp increase as the batch temperature increases. This one would expect if heat equivalence is to be obtained. Figure 5.18 demonstrates the effect of cooling water temperature on total energy. At the batch temperatures of 70 and 75ºC the cooling water does not have a very pronounced effect. However above 75ºC, there is a reduction in the total energy as the cooling water temperature is increased. This suggests
FIGURE 5.16

BATCH CONDITION CONTROL

OIL ADDITION TIME VS BATH TARGET TEMPERATURE

OIL ADDITION TIME VS COOLING WATER TEMPERATURE
BATCH CONDITION CONTROL

PIGMENT ADDITION TIME VS BATCH TARGET TEMPERATURE

PIGMENT ADDITION TIME VS COOLING WATER TEMPERATURE

FIGURE 5.17
TOTAL ENERGY VS BATCH TARGET TEMPERATURE

BATCH TARGET TEMPERATURE degrees C

TOTAL ENERGY VS COOLING WATER TEMPERATURE

COOLING WATER TEMPERATURE degrees C

FIGURE 5.18
more efficient mixing at the higher cooling water temperatures and higher batch temperatures. The cooling water affects the slip or friction characteristics of the polymer with the chamber walls, and the batch temperature affects the flow characteristics and in particular the extensional flow characteristics of the polymer. Therefore the effects shown in Figure 5.18 are probably due to a reduction in the elasticity of the polymer as the temperature increases.

The other criteria to be reviewed is the rheological uniformity of the mixed rubber compound produced by the two control methods. Tables 5.26 and 5.27 contain the regression coefficients and residuals for the model relating $\eta_0$ to the two process variables. As seen, $\eta_0$ is independent of cooling water temperature and batch temperature.

It is therefore reasonable to statistically analyse the values of $\eta_0$ to obtain a mean and a coefficient of variation. If this is done the coefficient of variation for the batch condition control was 1.385% and 1.633% for the mixing cycle using unit work. Therefore there is an improvement in rheological uniformity of the mixed rubber compound when the batch condition control is used. The real significance of this is that mixing cycles using temperature or time as the cycle control criteria would be dependent on both cooling water temperature and batch temperature, and as such give less rheological uniformity than the cycle using unit work.

Statistically analysing the T95 value from the Monsanto rheometer one obtains a coefficient of variation of 5.43% for the mixing specification using unit work as the control criteria and 4.8% for the batch condition control. It is likely that in a factory environment where variables are not controlled as closely the improvement shown by the batch condition control over conventional methods would be much greater.

The arithmetic mean for the power law index is 0.1285 for unit work and 0.1276 for the process control system. The coefficient of variability being 3.72% and 6.66% respectively. The arithmetic mean for the relaxation time in seconds is 11.87 for the mix cycle based on unit work and 10.80 for the process control cycle. However with coefficients of variability of 16.5% and 22.3% respectively one cannot claim any significant difference in the two mix cycles.
TABLE 5.26: Regression Coefficients and Residuals for \( \eta_0 \) Using Batch Condition Control

<table>
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<th>Cycle</th>
<th>deviance</th>
<th>df</th>
<th>estimate</th>
<th>s.e.</th>
<th>Parameter</th>
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<table>
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<th>df</th>
<th>estimate</th>
<th>s.e.</th>
<th>Parameter</th>
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<td>445.2</td>
<td>288.3</td>
<td></td>
<td></td>
<td>CT</td>
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<td>3</td>
<td>69.17</td>
<td>296.0</td>
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<td>4</td>
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Scale parameter taken as 0.1020E+07

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<td>10</td>
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<td>-53.11</td>
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<td>1861</td>
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CT = Cooling Water Temperature
BT = Target Batch Temperature
TABLE 5.27: Regression Coefficients and Residuals for $\eta_0$ from Mixing Cycle Based on Unit Work

<table>
<thead>
<tr>
<th>Cycle</th>
<th>Deviance</th>
<th>df</th>
<th>Estimate</th>
<th>s.e.</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
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<td></td>
<td></td>
</tr>
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<td>2</td>
<td>0.7841E+05</td>
<td>672.6</td>
<td>% gm</td>
<td>0.7841E+05</td>
<td>CT</td>
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<tr>
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Scale parameter taken as 0.1815E+07

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CT = Cooling Water Temperature
BT = Target Batch Temperature
5.8.3 Conclusions
The batch condition control did produce mixed rubber compound with more uniform rheological and vulcanisation properties than a mixing specification using unit work as the mixing criteria.

5.9 SUMMARY OF ALL RESULTS
There have been four experiments presented in this chapter, the results of which may be summarised below:

a) A relationship between $\eta_0$ (viscosity at 1 sec$^{-1}$) and torque at a reference rotor speed and temperature has been established.

b) The batch condition control system has been shown to be capable of modifying the rheological properties of polymers, so that despite variations in rheological properties of the inputted polymer, mixed rubber compound which conforms to a desired rheological endpoint is obtained.

c) The batch condition control system has been shown to produce mixed rubber compound with more uniform rheological and vulcanisation properties than a mixing specification using unit work as the parameter for controlling the cycle.

d) The temperature control function successfully maintained a target batch temperature by adjusting the rotor speed.

e) The batch condition control system performed satisfactorily for both fixed speed and variable speed mixers.
References for Chapter 5

6.1 AREAS COVERED BY THE RESEARCH

The research has evaluated methods of controlling the batch internal mixing process for rubber compounds such that material with uniform rheological properties is produced each and every time. Models have been developed for fixed speed machines and variable speed machines and evaluated with natural rubber compounds. An evaluation of the effect of cooling water temperature and rotor speed on the mixing process was carried out.

6.2 SUMMARY OF RESULTS

6.2.1 Viscosity Model

A model for obtaining viscosity measurements during the mixing process was developed. The assumption was made that the internal mixer could be treated as two adjacent, concentric cylinder, rotational viscometers. The model was evaluated on a Farrel Bridge F40 Banbury and a BR Banbury. The model was found to have a dependence on rotor speed, which demonstrated that the assumptions made the model too simplistic. As a result of this the control program was developed relating torque at a reference rotor speed to viscosity.

6.2.2 Temperature Control Model

The concept was developed that a target batch temperature could be reached and then maintained by varying the rotor speed. The model used a proportional-integral-derivative control technique and proved successful in both a BR Banbury and an F40 Banbury.
6.2.3 Relationship of Adjusted Torque to Compound Viscosity

When the initial viscosity model proved unsuccessful it was decided to evaluate techniques which would not involve making assumptions. In theory the torque generated by a mixed rubber compound on the Banbury rotors at a given rotor speed and temperature should be proportional to the viscosity of that material. Experiments did confirm that the viscosity of a compound, as measured by the TMS rheometer, was proportional to the torque measured on the Banbury rotors at a reference rotor speed and batch temperature.

6.2.4 Batch Condition Control for an Internal Mixer with Fixed Rotor Speed

A batch condition control program was developed for internal mixers with a fixed rotor speed. The batch temperature cannot be controlled with a fixed speed machine and therefore it was necessary to adjust the torque to a reference temperature. Linear regression was then used to predict the energy input necessary to obtain the desired torque value. The model was evaluated successfully.

6.2.5 Batch Condition Control for an Internal Mixer with Variable Rotor Speed

Since the rotor speed can be varied it is possible to control the batch temperature at a reference level. The system therefore utilised a regression analysis to determine the energy required to obtain the target torque. When this was reached the rotor speed was set to the reference value and the torque obtained just prior to dumping. The system was evaluated and found to perform very well. In particular experiments were run with various blends of SMR10/SMR20 and SMR5CV/SMR20 natural rubber. The control system was able to adjust the mixing cycle to compensate for the varying viscosities of the polymers and produce final mixed compound with consistent rheological properties.
It was recognised that as well as uniform rheological properties, the control system must also produce uniform heat history for the vulcanisation system.

Since the endpoint was predicted, it was possible to calculate the point that the vulcanisation ingredients should be added to obtain equivalent heat history. However this did not prove very successful since the rotor speed was varied on a continuous basis and therefore the rate at which energy was inputted also varied. It was therefore difficult to obtain an equivalent heat history and more work is required.

6.2.6 Batch Condition Control Compared with a Mixing Specification Using Unit Work as the Control Criterion

The present mixing technique which gives the most uniform mixed rubber compound is that using unit work. Batches were mixed to a specification based on unit work and also using the batch condition control system. The result was that mixed rubber compound produced using the batch condition control program was more rheologically uniform than material produced using the specification based on unit work. The significance of this is that most mixing specifications in use today use time or temperature as the control criteria. Since these systems produce mixed rubber compound which has rheological properties less precise than that produced by unit work, the improvement in mixed rubber compound possible with the batch condition control program is very significant.
The basic concepts contained within the batch condition control system are really only the beginning and have opened doors into unexplored areas of processing technology. The system has been shown to be very effective with natural rubber, and has successfully modified the molecular structure of natural rubber blends to produce final mixed rubber compound to a rheological endpoint.

It is important to evaluate the system with other polymers and blends of polymers.

Only limited work was carried out using a fixed speed mixing cycle. It is clear that in this application the temperature adjustment of the torque is very important. The accuracy of the temperature adjustment over a range of ±10°C from the target temperature needs to be determined.

With the system for use with internal mixers with variable speed it is difficult to obtain satisfactory heat equivalence for the crosslinking system, due to the rotor speed constantly changing to control the batch temperature. This part of the system needs to be studied in isolation and a viable solution arrived at. Finally the system needs to be fully evaluated on full size internal mixers. Some work was carried out very satisfactorily on an F40 size Banbury mixer. This work however was limited and further studies on industrial size machines would be very beneficial.
In this appendix are the copies of the process control diagrams developed during this work. In Chapter 5 the flow diagrams for two batch condition control programs are given. The first is for a fixed rotor speed machine and the relevant program was called BRTORF. The second program was for variable rotor speed internal mixers and was called BRTORN. Both these programs used total energy to obtain the desired target torque. Two other control programs are also included in this appendix. They use time rather than energy in the prediction of the endpoint. The program for use with a fixed speed internal mixer was called BRTOTF and the one for use with a variable speed internal mixer was called BRTORR. It was felt that programs using time to predict the endpoint with a variable rotor speed Banbury could yield erroneous results, therefore energy was used instead.

The program used to obtain the setpoints is called BRSETF and is also included. The set-up runs were carried out by having an instantaneous power output connected to a chart recorder. The progress of the mixing cycle was followed on the chart recorder and as a desired setpoint was reached, an analogue input signal was sent to the computer to order for it to implement the next step in the mixing cycle.
189
1826 MOV CURSOR(3,25) \ DISPLAY MODEL='-BRIGHT' \ PRINT
1827 GOSUB 2410
1828 GOSUB 4100
1829 AOUT(0,,6) \ AOUT(-2045,,0) \ GO TO 3770
1830 GOSUB 2380 \ REM-----------------RAM UP
1831 GOSUB 2440 \ REM-----------------GATE OPEN
1832 GOSUB(0,,4)
1833 GOSUB 2410
1834 AOUT(,0,,3)
1835 AOUT(,2045,,0)
1836 HTEXT('R',F',22,25,'STORING DATA ON FLOPPY DISC.' )
1837 R5=(T8-Pl)*(Rl/60)
1838 MOVE CURSOR(-4,10) \ PRINT 'TORQUE AT DUMP='INT(T9)'TARGET TORQUE='T-4t25
1839 HTEXT('S,f',24,25,'STORING DATA ON FLOPPY DISC. ')
1840 FOR M=O TO (Ot1)/S
1841 FOR J=O TO 4
1842 P(H,J)=Z(U2)
1843 U2=U2t1
1844 NEXT J
1845 NEXT H
1846 CLOSE 12
1847 FOR M=O TO 2047,5
1848 FOR J=O TO 4
1849 PRINT USING .10, 'Data file name='Pt
1850 PRINT .1, 'PREDICTED DUMP ENERGY='X1'ACTUAL DUMP ENERGY=
1851 PRINT .1, 'REFERENCE TORQUE, 'T4tJ5'ACTUAL TORQUE=
1852 PRINT .1, 'REFERENCE TEMPERATURE='T7'ACTUAL TEMPERATURE='T5
1853 PRINT .1, 'TOTAL ENERGY K/J='W4'TOTAL WORK HJ/HJ='1.14/11.6607
1854 PRINT .1, 'ADJUSTED TORQUE='DS'PIGHENT ADDITION TIME='Pl
1855 PRINT .1, 'REGRESSION ENERGIES=·10~R(0,0)·='10~R(1,0)'='10~R(2,0)'.'10~R(3,0)'.'10~R(4,0)'.'10~R(5,0)
1856 PRINT .1, 'REGRESSION TORQUE='10~R(0,1)'='10~R(1,1)'='10~R(2,1)'.'10~R(3,1)'.'10~R(4,1)'='10~R(5,1)
1857 PRINT .1, 'ROTOR REVS AFTER PIGMENT ADDITION='RS'BLACK ADDITION TIME='P2
1858 PRINT .1, 'REGRESSION COEFFICIENT R6·OIL ADDITION TIME='PO
1859 PRINT .1, 'ROTOR SPEED='R1'AVERAGE ADJUSTED TORQUE='D9
1860 PRINT .1, 'REFERENCE REVS='R1'AVERAGE ADJUSTED TORQUE='D9
1861 PRINT .1, 'REFERENCE OIL ADDITION TIME='PO
1862 PRINT .1, 'REFERENCE TEMPERATURE='T7'ACTUAL TEMPERATURE='T5
1863 PRINT .1, 'REFERENCE TORQUE='T4'target torque='T-4t25
1864 PRINT .1, 'REFERENCE TEMPERATURE='T7'ACTUAL TEMPERATURE='T5
1865 PRINT .1, 'REFERENCE REVS='R1'AVERAGE ADJUSTED TORQUE='D9
1866 PRINT .1, 'REFERENCE OIL ADDITION TIME='PO
1867 PRINT .1, 'REFERENCE TEMPERATURE='T7'ACTUAL TEMPERATURE='T5
1868 CLOSE .1
1869 DISPLAY CLEAR
1870 GOSUB 2380
1871 GOSUB 2470
1872 FOR M=O TO (Ot1)/S
1873 FOR J=O TO 4
1874 PRINT USING .10, 'TIME BATCH POWER TORQUE TOTAL ENERGY
1875 NEXT J
1876 NEXT H
1877 PRINT .1, 'Another run using the same values '
1878 INPUT AS
1879 IF A$="Y" THEN P=7 GO TO 1050
1880 PRINT .1, 'Another run using different values '
1881 INPUT AS
1882 IF A$="Y" THEN 1050
1883 CLOSE
1884 STOP
1885 REM-----------------TEMP SUBROUTINE-----------------------------
1886 FOR I=0 TO 9
1887 AIN(x,,9)
1888 S=S+I
1889 NEXT Z
1890 U6=6/10
1891 S=O
1892 TS=W6/K
1893 RETURN
**FUNCTION TORGUE**

The subroutine computes angular velocity and motor voltage for a given angular velocity in degrees per second.

```plaintext
W = \frac{(RI/60)*6.2832}{R} \quad \text{(angular vel in rad/s)}
```

```plaintext
V = \frac{R*3.3 - 33}{R} \quad \text{(motor voltage)}
```

The subroutine processes data in two loops, calculating torque and energy. It also includes a regression analysis for torque values.

```plaintext
\text{IF} \ V = 1 \ \text{THEN} 3230
\text{IF} \ V = 3 \ \text{THEN} 3140
\text{GO TO} 3230
```

Regression analysis involves calculating the slope and performing a regression analysis on the data.
3660  R6=SOR((K0*(X2-Y3)/604)/(Y5-46/6))
3665  IF  R6<.B5  GO TO 3290
3670  MOVE_CURSOR(23,10)  PRINT  'REM  CALCULATED  ENERGY  TO  DUMP
3700  X8=(LOG10(T~)-CO)/HO  '
3710  X1=10**x8
3715  V1=1  T(1)=0  T(2)=0  T(3)=0  T2=0
3720  MOVE_CURSOR(6,20)  PRINT  'ENERGY  TO  DUMP='X1'  PRESENT  ENERGY='W4
3721  T(0)=0
3722  E4=((((1410/1.677*(R1/60)*2.2832))+((R1*6.08643)-98.64))/1000  '
3723  E7=E4/(R1/60)  '
3724  E9=X1-W4  '
3725  E5=(40*E7)  '
3728  S9=W4+(E9-E5)  '
3730  S1=(40*(R1/60))  '
3732  T2=0
3733  S4=S2-S3  '
3734  S2=S4/T1  '
3736  S5=S4/T1  '
3738  S6=S2*E6  '
3740  S9=S9+S6  '
3740  E5=(40*E7)  '
3748  IF  E8<E5  GO TO 1821
3754  IF  W4>59  GO TO 1821
3760  GO TO 3770
3770  GOSUB  4200
3775  IF  P=1  GO TO 3766
3778  X9=(T0-P1)  '
3780  IF  W4<X1  GO TO 3770
3785  D9=T9*(EXP(-B*(T5-T7)))
3790  D9=T3*(EXP(-B*(T5-T7)))
3790  GO TO 1830
4100  RETURN
4200  GOSUB  2530
4210  GOSUB  2770
4220  GOSUB  2940
4230  RETURN
4240  GOSUB  2940
4250  RETURN
REM 1000 REST THE PROGRAMME CALLED BRTORN
1010 REM STRIPPED DOWN VERSION OF BRTORN FOR FASTER SAMPLING RATE
1020 REM
1030 REM VARIABLE SPEED MIXING SPEC TO FIXED TORQUE USING CONVENTIONAL SPEC-MASTICATION IN.
1040 A=SYS(1)
1050 DISPLAY CLEAR
1060 WIDE LINE(-2,1)
1070 WIDE LINE(-2,3)
1080 PRINT ' PROGRAMME FOR MIXING TO PRESET TORQUE'
1090 PRINT 'CONTROL CONCEPTS BY R.R.HATTHEWS AND P.K.FREKLEY.'
1100 PAUSE(5)
1105 DISPLAY CLEAR
1110 Dim R(129), T(3)
1120 WIDE LINE(-2,15)
1125 WIDE LINE(-2,3)
1130 V=1
1135 B=3.1549E-03
1140 REM BATCH INFORMATION SUBROUTINE-------------------------
1150 PRINT 'INITIAL ROTOR SPEED RPM', INPUT 'R1'
1160 I=R1*1.5231
1170 REM MAXIMUM ROTOR SPEED RPM', INPUT 'R2'
1180 F=R2*1.5231
1190 REM MAX DUMP TEMPERATURE DEGREES C', INPUT 'T1'
1200 PRINT 'TARGET TORQUE FOR MASTICATION:', INPUT 'T2'
1210 PRINT 'ROTOR SPEED FOR BLACK INCORPORATION', INPUT 'R3'
1220 K2.025
1230 V7=T1*K
1240 K1=2500
1250 K2=150
1260 T6=2
1270 V9=(R1*3.3)-33
1280 T4=265
1290 REM TARGET TORQUE
1300 REM RAM DOWN
1310 GOSUB 2100
1320 IF T7>M2 GO TO 1310
1330 IF T7(14 GO TO 1310
1340 REM ENTER FILE NAME FOR DATA STORAGE!
1350 OPEN P. FOR OUTPUT AS 12
1360 R8=777
1370 REM START SIGNAL FROM BANBURY
1380 PRINT '9,F',24,25,'POLYMER MASTICATION.'
1390 PAUSE(5)
1400 AOUT(0,0)
1410 IF G=1 THEN 1450
1420 GO TO 1420
1430 AOUT(0,0)
1440 AOUT(2045,0)
1450 GET TIME(P2)
1460 AOUT(0,0)
1470 AOUT(2045,0)
1480 HTEXT('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.')
194
1760 HTXT('b,F'+24,25,'MIXING TO PREDETERMINED TORQUE.
')
1761 GSUB 4100
1762 GSUB 4200
1763 GSUB 2390
1764 GSUB 4100
1765 GSUB 2390
1770 GO TO 1770
1780 GSUB 2390
1800 MOVE_CURSOR(3+25) \ GET_TIME(P1) \ DISPLAY_MODE('BRIGHT')
1823 P=2 \ GSUB 4200
1824 GSUB 4200
1825 AOUT((0+0), \ AOUT(2045+6)
1826 MOVE_CURSOR(3+25) \ DISPLAY_MODE('BRIGHT') \ PRINT 
1827 GSUB 2410
1828 GSUB 4200
1829 AOUT((0+0), \ AOUT(2045+4)
1830 GO TO 1790
1831 AOUT((500+7) \ REM-----------------RAH
1835 R5=(TB-P1)*(R1/60)
1836 FOR M=0 TO (0+1)/6
1837 FOR J=0 TO 5
1838 PCH,J)=Z(U2)
1839 U2=U2+1
1840 NEXT J
1841 NEXT H
1842 CLOSE:12
1843 OPEN 'LP' FOR OUTPUT AS \ FILE '1
1844 PRINT 'Data file name='P
1845 PRINT 'PREDICTED DUMP ENERGY='X1\ACTUAL DUMP ENERGY = 'U4
1846 PRINT 
1847 PRINT 'REFERENCE TORQUE = 'T4\TARGET TORQUE = 'T9
1848 PRINT 
1849 PRINT 'TARGET TEMPERATURE='T5
1850 PRINT 
1851 TOTAL ENERGY K/J = 'U4\TOTAL WORK MWJ"3='U4/11.6607
1852 PRINT 
1853 PRINT 'PIGMENT ADDITION TIME='P1
1854 PRINT 
1855 PRINT 'adjusted torque='D8\PIGMENT ADDITION TIME='P1
1856 PRINT 
1857 PRINT 'R5=(TB-P1)*(R1/60)
1858 PRINT 'REGRESSION ENERGIES='10'R(0,0)='10'R(1,0)='10'R(2,0)='10'R(3,0)='10'R(4,0)='10'R(5,0)
1859 PRINT 'REGRESSION TORQUE='10'R(0,1)='10'R(1,1)='10'R(2,1)='10'R(3,1)='10'R(4,1)='10'R(5,1)
1860 PRINT 
1861 PRINT 'PIGMENT REVS AFTER PIgment ADDITION='R5\BLACK ADDITION TIME='P2
1862 PRINT 
1863 PRINT 'REGRESSION COEFFICIENT='R6\OIL ADDITION TIME='P0
1864 PRINT 
1865 CLOSE \1
1866 DISPLAY_CLEAR
1867 GSUB 2410
1868 GSUS 2380
1869 FOR J=0 TO 5
1870 FOR M=0 TO (0+1)/6
1871 FOR J=0 TO 5
1872 PRINT USING \'**\** *** **\Z(U)'
1873 U=U+1
1874 NEXT J
1875 NEXT M
1876 CLOSE \1
1877 DISPLAY_CLEAR
1878 GSUB 2380
1879 GSUB 2470
1880 AOUT((0+0), \ AOUT(0,0)
1881 FOR J=0 TO 5
1882 FOR M=0 TO (0+1)/6
1883 PRINT \PRINT 'press RETURN button to review data 
1884 INPUT AA
1885 DISPLAY_CLEAR
1886 MOVE_CURSOR(1,1)
1887 PRINT '******************************************************************************
1888 PRINT' TIME BATCH POWER SPEED TORQUE TOTAL 
1889 PRINT '******************************************************************************
1890 ROLL_AREA(4,24)
1891 U=0
1892 FOR M=0 TO (0+1)/6
1893 FOR J=0 TO 5
1894 PRINT USING \'**\** *** **\Z(U)'
1895 U=U+1
1896 FOR J=0 TO 5
1897 PRINT 
1898 PRINT 
1899 PRINT '******************************************************************************
1900 PRINT' TIME BATCH POWER SPEED TORQUE TOTAL 
1901 PRINT '******************************************************************************
1902 ROLL_AREA(4,24)
1903 U=0
1904 FOR M=0 TO (0+1)/6
1905 FOR J=0 TO 5
1906 PRINT USING \'**\** *** **\Z(U)'
1907 U=U+1
1908 FOR J=0 TO 5
1909 FOR M=0 TO (0+1)/6
1910 FOR J=0 TO 5
1911 FOR M=0 TO (0+1)/6
1912 PRINT USING \'**\** *** **\Z(U)'
1913 U=U+1
1914 NEXT J
1915 NEXT M
1916 CLOSE \1
1917 STOP
1918 FOR J=0 TO 5
1919 FOR M=0 TO (0+1)/6
1920 PRINT USING \'**\** *** **\Z(U)'
1921 U=U+1
1922 NEXT J
1923 NEXT M
1924 NEXT R
1925 CLOSE \1
1926 STOP
1927 REM-----------CHECK MIXER IS READY SUBROUTINE
1928 REM-----------CHECK MIXER IS READY SUBROUTINE
1929 REM-----------------RAM UP
1930 AOUT((2047+3) \ PAUSE(1) \ AOUT((0+0), \ AOUT(0,0)
1931 RETURN
1932 AOUT((2047+2) \ PAUSE(1) \ AOUT((0+0), \ AOUT(0,0)
1933 RETURN
1934 AOUT((2047+1) \ PAUSE(1) \ AOUT((0+0), \ AOUT(0,0)
1935 RETURN
1936 REM-----------------GATE SHUT
1937 AOUT((2047+5) \ PAUSE(1) \ AOUT((0+0), \ AOUT(0,0)
1938 RETURN
1939 REM-----------------GATE UP
1940 REM-----------------GATE SHUT
1941 REM-----------------GATE UP
1942 REM-----------------GATE SHUT
1943 REM-----------------GATE UP
1944 REM-----------------GATE SHUT
1945 REM-----------------GATE UP
1946 REM-----------------GATE SHUT
1947 REM-----------------GATE UP
FOR I=0 TO 9
2560 S=S+Y
2570 NEXT Z
2580 V6=V/10
2590 S=0
2600 T5=V6/K
2610 RETURN
2620 V7=TUK
2630 V8=V7-V6, REM----------VB IS ERROR VOLTAGE
2640 E=EtVB
2650 E1=(V8-1)
2660 IF E1<0 THEN E1=0
2670 IF E1<11 THEN E1=11
2680 AOUT("UL",11)
2690 L=V8
2700 I=0
2710 RETURN
2720 REM------------------TORGUE SUBROUTINE--------------------------
2730 Rl=Ul/13.5231
2740 WI=(Rl/60)-2.932
REH--------------ANGULAR
VEL IN RADS PER SEC
2750 V9=(Rl*J.3)-33 REM MOTOR VOLTAGE
2760 FOR X=0 TO 99
2770 AIN(,VS",11'
2780 07=07tV5
2790 NEXT X
2800 V'5=07/100
2810 IF V'5<T5 THEN V'5=T5
2820 IF V'5>10 THEN V'5=10
2830 A5=(VS*15.B87)-.79
2840 J9=AS*V9
2850 V7=(Rl*6.08643)-9B.64
2860 T9c(J9-J7)/(1.B7*Wl) REM J7 IS TORGUE FOR EMPTY 9/8,T9 IS TOTAL TORGUE
2870 W2=(J9*2.21)/I000 REM--ENERGY IN K/J
2880 W4=W2+W4
2890 RETURN
2900 REM----------DATA ACCUMULATION AND GRAPH SUBROUTINE---------------
2910 Z(0)=T8
2920 Z(0)=INT(TS)
2930 Z(0)=INT(J9)
2940 Z(0)=INT(W4)
2950 IF H=2 THEN 1700
2960 IF H=1 THEN 3290
2970 RETURN
2980 REM---------RUNNING AVERAGE OF TOROUE VALUES----------------------
2990 IF V'1 THEN 3230
3000 IF V=3 THEN 3140
3010 T(V)=T9
3020 T(V)=T(V+1)
3030 NEXT N
3040 V=2
3050 T(V)=T9 \ V=V+1
3060 RETURN
3070 REM----------------------REGRESSION ANALYSIS------------------------
3080 HTEXT("End",23,10,"PERFORMING REGRESSION ANALYSIS.
3090 H=J
3100 Rl=R7
3110 AOUT(,17",7)
3120 PAUSE(S)
3130 DIM R(S,1)
3140 FOR C=0 TO 5
3150 GOSUB 2540
3160 GOSUB 2790
3170 GOSUB 3100
3180 GOSUB 2950
3190 GOSUB(EXP(-B*(X-T5-T7)))
3200 R(C,0)=LOG10(W4)
3210 R(C,1)=LOG10(D0)
3220 NEXT C
3230 H=2 \ RETURN
3240 REM----------------------REGRESSION ANALYSIS------------------------
3415 NEXT C
3420 FOR C=0 TO 5
3425 Y1=R(C,1)
3430 Y2=Y*+Y1
3435 NEXT C
3440 Y3=Y2*X4
3445 FOR C=0 TO 5
3450 X5=R(C,0)*R(C,0)
3455 X6=X4*X5
3460 NEXT C
3465 X7=X*2
3470 FOR C=0 TO 5
3475 Y4=R(C,1)*R(C,1)
3480 Y5=Y5+Y4
3485 NEXT C
3490 HO=(6*Y3-Y3)/(6*X6)-X7
3495 IF HO>0 THEN H=0 \ GO TO 1770 \ REM TO ENSURE NEGATIVE SLOPE
3500 C0=(Y2-(HO*X4))/6
3505 Y6=Y2*2
3510 R6=SQR((H0*(X2-Y3)/6)/(Y5-Y6/6))
3515 MOVE_CURSOR(23,10) \ PRINT .
3520 XO=(LOG10(T4)-C0)/HO \ REM CALCULATED ENERGY TO DUMP
3525 X1=X0*X8
3530 Y1=10 \ T(1)=0 \ T(2)=0 \ T(3)=0 \ T2=0 \ H=3
3535 MOVE_CURSOR(6,20) \ PRINT 'ENERGY TO DUMP='X1' PRESENT ENERGY='W4
3540 P1=*
3545 E6=((T440)*(1.87*((R7/60)*6.2832))+(R7*6.08443-98.64))/1000 \ REM ENERGY IN KJ AT REFERENCE T
3550 E7=E6/(R7/60) \ REM ENERGY PER REV
3555 E8=E1-E4 \ REM-ENERGY REMAINING TO END OF CYCLE
3560 E9=E40*67 \ REM ENERGY FOR PIGMENT ADDITION
3570 S1=40*(R7/60) \ REM-SECS FROM PIGMENT ADDITION TO PREDICTED DUMP
3575 S2=S1+41 \ S3=S1+T5 \ REM-EQUIV HEAT HISTORY FOR STD AND ACTUAL CONDITIONS
3580 S4=S2-S3 \ REM DEVIATION IN HEAT HISTORY
3585 S5=S4/T1 \ REM DEVIATION IN SECS
3590 E6=S5*E6 \ REM-Necessary Correction in SECS.
3595 E9=E9+S6 \ REM CORRECTED PIGMENT ADDITION TIME.
3730 IF E9<0 GO TO 1821
3732 IF W4<0 GO TO 1821
3735 GO TO 3740
3740 GOSUB 4200
3775 IF P=1 GO TO 3732
3776 IF W4<0 GO TO 3740
3777 P4=(T8-P1)
3778 IF P420 GO TO 3740
3779 AQ1(U=17+,7) \ R1=R7
3790 PAUSE(3)
3791 GOSUB 2530
3792 GOSUB 2770
3793 GOSUB 2840
3795 DB=e9*(EXP(-B#(T5-T7)))
3798 GO TO 1030
4100 GOSUB 2530
4110 GOSUB 2770
4120 GOSUB 2940
4130 RETURN
4200 GOSUB 2530
4210 GOSUB 2420
4220 GOSUB 2760
4230 GOSUB 31.00
4240 GOSUB 2940
4250 RETURN

3785 D8+T9*(EXP(-B#(T5-T7)))
REM -- MIXING SPEC TO FIXED TORQUE USING CONVENTIONAL SPEC-MASTICATION.
1100 REM-X-FIXED SPEED MACHINE USING TIME
1110 A=S7(7,1)
1120 B7=3.15649E-03
1130 PRINT ' ROTOR SPEED RPM:'
1140 R1*13.5231
1150 PRINT ' VOLTS FOR INITIAL Rotor SPEED
1160 PRINT 'TEMPERATURE FOR HEAT EQUVALENCE ADJUSTMENT:
1170 INPUT T1
1180 PRINT 'TARGET TORQUE FOR MASTICATION:
1190 INPUT M2
1200 K=.025
1210 V9=(R1*3.3)-33
1220 T4=320
1230 T7=106
1240 PRINT 'ENTER FILE NAME FOR DATA STORAGE:
1250 OPEN Pt FOR OUTPUT AS FILE t2
1260 AOUT(0,0,0)
1270 GO TO 2330
1280 REM---------------~-------------START SIGNAL FROM BANBURY
1290 DISPLAY_CLEAR
1300 HTTEX('B,F',24,25,'AWAITING START SIGNAL.'
1310 AIN(0,0,4)
1320 IF D$ THEN 1450
1330 START_TIME('CHZ')
1340 GOSUB 2500
1350 HTTEX('B,F',24,25,'POLYMER MASTICATION.
1360 GOSUB 2410
1370 GOSUB 4100
1380 PAUSE(1)
1390 IF D>5 GO TO 1510
1400 IF T8<10 GO TO 1510
1410 HTTEX('B,F',24,25,'EACH ADDITION.
1420 A=1
1430 GOSUB 2380
1440 FOR W=0 TO 2
1450 GOSUB 4200
1460 NEXT W
1470 GOSUB 2410
1480 GOSUB 4000
1490 Q3=TB-P2
1500 GOSUB 4000
1510 AOUT(0,0,0)
1520 GET_TIME<P2)
1530 AOUT(0,0,6)
1540 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1550 GOSUB 4100
1560 GET_TIME<P2)
1570 AOUT(0,0,6)
1580 HTTEX('B,F',24,25,'ADD OIL.
1590 AOUT(0,0,0)
1600 GET_TIME<P2)
1610 AOUT(0,0,6)
1620 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1630 GOSUB 4100
1640 GET_TIME<P2)
1650 AOUT(0,0,6)
1660 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1670 GOSUB 4100
1680 GET_TIME<P2)
1690 AOUT(0,0,6)
1700 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1710 GOSUB 4100
1720 GET_TIME<P2)
1730 AOUT(0,0,6)
1740 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1750 GOSUB 4100
1760 GET_TIME<P2)
1770 AOUT(0,0,6)
1780 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1790 GOSUB 4100
1800 GET_TIME<P2)
1810 AOUT(0,0,6)
1820 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1830 GOSUB 4100
1840 GET_TIME<P2)
1850 AOUT(0,0,6)
1860 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1870 GOSUB 4100
1880 GET_TIME<P2)
1890 AOUT(0,0,6)
1900 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1910 GOSUB 4100
1920 GET_TIME<P2)
1930 AOUT(0,0,6)
1940 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1950 GOSUB 4100
1960 GET_TIME<P2)
1970 AOUT(0,0,6)
1980 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
1990 GOSUB 4100
2000 GET_TIME<P2)
2010 AOUT(0,0,6)
2020 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2030 GOSUB 4100
2040 GET_TIME<P2)
2050 AOUT(0,0,6)
2060 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2070 GOSUB 4100
2080 GET_TIME<P2)
2090 AOUT(0,0,6)
2100 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2110 GOSUB 4100
2120 GET_TIME<P2)
2130 AOUT(0,0,6)
2140 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2150 GOSUB 4100
2160 GET_TIME<P2)
2170 AOUT(0,0,6)
2180 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2190 GOSUB 4100
2200 GET_TIME<P2)
2210 AOUT(0,0,6)
2220 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2230 GOSUB 4100
2240 GET_TIME<P2)
2250 AOUT(0,0,6)
2260 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2270 GOSUB 4100
2280 GET_TIME<P2)
2290 AOUT(0,0,6)
2300 HTTEX('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.'
2310 GOSUB 4100
2320 GET_TIME<P2)
2330 AOUT(0,0,6)
1830 GOSUB 2380 \ REM INTO RAM UP
1840 GOSUB 2440 \ REM ---------------GATE OPEN
1860 HTEXT('BF',24,25,'BATCH BUFFERED.')
1865 R5=(CT/P)+R(R1/60)
1870 MOVE_CURSOR(4,10) \ PRINT 'TORQUE AT DUMP='INT(D9)'TARGET TORQUE='INT(T4)
1890 HTEXT('BF',24,25,'STORING DATA ON FLOPPY DISC.')
1900 FOR H=0 TO 0.1/5
1910 FOR J=0 TO 4
1920 P(H,J)=Z(U2)
1930 U2=U2+1
1940 NEXT J
1950 NEXT H
1960 FOR M=0 TO (OH)/5
1970 FOR N=0 TO 4
1980 PRINT USING...2700 AOUT(,2047",1)
1990 PRINT \ press RETURN button to review data
2000 FOR M=0 TO (OH)/5
2010 FOR N=0 TO 4
2020 PRINT \ print 'REFERENCE TORQUE = 'T4'ACTUAL TORQUE = 'D9
2030 NEXT N
2040 NEXT M
2050 PRINT \ TOTAL ENERGY K/J =U4/1000'TOTAL WORK MJ/MJ=U4/1160.7
2060 PRINT \ ADJUSTED TORQUE = 'D8'PIGMENT ADDITION TIME='Pl
2070 FOR M=0 TO (OH)/5
2080 FOR N=0 TO 4
2090 PRINT USING...2100 PRINT \ press RETURN button to review data
2110 FOR M=0 TO (OH)/5
2120 FOR N=0 TO 4
2130 PRINT \ print 'REFERENCE TORQUE = 'T4'ACTUAL TORQUE = 'D9
2140 NEXT N
2150 NEXT M
2160 FOR M=0 TO (OH)/5
2170 FOR N=0 TO 4
2180 PRINT USING...2200 PRINT \ press RETURN button to review data
2210 FOR M=0 TO (OH)/5
2220 FOR N=0 TO 4
2230 PRINT \ print 'REFERENCE TORQUE = 'T4'ACTUAL TORQUE = 'D9
2240 NEXT N
2250 NEXT M
2260 PRINT \ Another run using the same values i
2270 IF A6=** THEN 1330
2300 PRINT \ Another run using different values ii
2310 NEXT M
2320 STOP
2330 REM-----------------CHECK MIXER IS READY SUBROUTINE
2340 GOSUB 2470
2350 GOSUB 2380
2360 GO TO 1370
2370 REM--------------------------RAM UP
2390 AOUT((2047,,3) \ PAUSE(1) \ AOUT(0,,3)
2400 REM--------------------------RAM DOWN
2420 AOUT((2047,,2) \ PAUSE(1) \ AOUT(0,,2)
2430 REM--------------------------GATE OPEN
2450 AOUT((2047,,1) \ PAUSE(1) \ AOUT(0,,1)
2460 REM--------------------------GATE SHUT
2480 AOUT((2047,,5) \ PAUSE(1) \ AOUT(0,,5)
2490 REM--------------------------ROTATOR SPEED
2500 AOUT((1,,7)
2520 RETURN
2530 REM--------------------------TEMP SUBROUTINE-----------------------------
2540 FOR Z=0 TO 9
2550 AIN(z,,9)
2560 S=S+Y
2570 NEXT Z
2580 V6=V5/10
2590 S=0
2600 T5=V6/K
2610 RETURN
2730 REM
2750 REM
2770 REM
2790 REM
2810 REM
2830 REM
2850 REM
2870 REM
2890 REM
2910 REM
2930 REM
2950 REM
2970 REM
2990 REM
3010 REM
3030 REM
3050 REM
3070 REM
3090 REM
3110 REM
3130 REM
3150 REM
3170 REM
3190 REM
3210 REM
3230 REM
3250 REM
3270 REM
3290 REM
3310 REM
3330 REM
3350 REM
3370 REM
3390 REM
3410 REM
2830 VS=07100
2840 07=0
2850 AS=(V5$15.887)-.79
2860 J9=A5XV9
2870 J7=(R16.0643)-98.64
2880 T9=(J9-J7)/(1.87*W1)
2890 W2=J9*2.21
2900 W4=W2+W4
2910 RETURN
2920
2930 REM-------------------DATA ACCUMULATION AND GRAPH SUBROUTINE---------------
2940 GELTIMUla)
2950 Z(0)=T8 ,
2960 Z(0)=INHT5) ,
2970 Z(0)=INT(J9)
2980 Z(0)=INT(T2)
2990 IF H=2
3000 GOTO 1700
3010 IF H=3
3020 GO TO 3290
3030 RETURN
3040 
3050 REM--------RUNNING AVERAGE OF TORQUE VALUES-----------------------
3060 IF V=1 THEN
3070 IF V=3 THEN
3080 GO TO 3230
3090 HV=1/9
3100 TO=T(I)+T(2)+T(3)
3110 T2=TO/3
3120 IF H=1
3130 GO TO 3180
3140 IF T2(T3
3150 GO TO 3250
3160 T3=T2
3170 FOR 8=1 TO 2
3180 T=Hi)
3190 NEXT B
3200 V=2
3210 T<V)"*T9 , V=VH
3220 RETURN
3230 IF A=1 GO TO 3273
3240 IF A=2 __
3250 P5=(T8-PO)
3260 IF P5<2.5
3270 H=1
3280 RETURN
3290 HTEXT('B,F',23rlO,'PERFORMING REGRESSION ANALYSIS.
3300 H=3 " " .-. -.'
3310 DIH R('5,1)
3320 FOR c=0 TO '5
3330 GASUS 2540
3340 GOSUS 2770
3350 GOSUB 3100
3360 GOSUB 2950
3370 D6=T9*(EXP(-B7*(T9-T7»)
3380 R(C,0)=T8
3390 R(C,1)=LOG10(D8)
3400 NEXT C
3410 D8=0
3420 x=0 
3430 X2=0 
3440 X3=0 
3450 X4=O 
3460 Y1=O 
3470 Y2=0 
3480 Y3=0 
3490 Y4=0 
3500 Y5=0 
3510 Y6=0 
3520 FOR C=0 TO 5
3530 X5=R(C,0)*R(C,1)
3540 X6=R(C,0)*R(C,0)
3550 X7=0
3560 FOR C=0 TO 5
3570 X8=R(C,0)*R(C,0)
3580 X9=R(C,0)*R(C,1)
3590 X10=R(C,0)*R(C,0)
3600 X11=R(C,0)*R(C,0)
3610 Y5=Y5Y4
3620 RETURN
3630 REM-------------------REGRESSION ANALYSIS-----------------------------
3640 0£T_TIH£(T8)
3650 V=1
3660 T(1)=O
3670 T(2)=O
3680 T(3)=O,
3690 T(4)=O
3700 P=1
3710 S9"X1 '-9'.1
3720 R6=SOR('H0#(X2-Y3)/(4#X6)-X7)
3730 IF M0>0 THEN H=0 \ GO TO 1770 \ REM TO ENSURE NEGATIVE SLOPE
3740 CO=(Y2-(H0#X4))/6
3750 V=2
3760 R6=SOR('H0#(X2-Y3)/(4#X6)-X7)
3770 MOVE CURSOR(23,10) \ PRINT ' 
3780 X1=(L0D01(T4)-CO)/MO \ REM CALCULATED TIME TO DUMP
3790 GOTO 1900
3800 1912 \ T(1)=0 \ T(2)=0 \ T(3)=0 \ T2=0
3810 MOVE CURSOR(6,20) \ PRINT 'TIME TO DUMP=X1' PRESENT TIME=X0
3820 IF X=1
3830 3917 SRT(V)/(R1/60) \ REM----SECS TO OBTAIN 40 REV
3840 D10B SRT(V)/(R1/60) \ REM-----PENDANT ADDITION TIME
3850 REM-------TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3860 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3870 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3880 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3890 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3900 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3910 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3920 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3930 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3940 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3950 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3960 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3970 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3980 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
3990 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME
4000 REM---TIME PROGRAMMED HAVING THE "TIME TO DUMP" REM------TIME

REM --- SEC 5 TO OBTAIN 40 REV
3717 5I=40/(R1/60) \ REM --- SECS TO OBTAIN 40 REV
3718 59=X1-51 \ REM --- PIGMENT ADDITION TIME
3719 59=X1-51 \ REM --- PIGMENT ADDITION TIME
3720 S4=S2-S3 \ REM --- DEVIATION IN HEAT HISTORY
3721 S5=S4/T1 \ REM --- DEVIATION IN SECS
3722 R4=S4+S5 \ REM --- CORRECTED ADDITION TIME
3723 IF YB=R4 GO TO 1821
3725 GO TO 3740
3740 GOSUB 4000
3775 IF P=1 GO TO 3723
3780 IF YB<X1 GO TO 3740
3800 GOSUB 2870
3880 GOSUB 3850
3910 GOSUB 2950
3970 D9=T9
3980 D8=D9*(EXP(-B7*(TS-9.5)))
3990 IF D8<T4 GO TO 3940
3870 T(V)=T9
3880 T2=T0/3
3890 FOR D=1 TO 2
3900 T(D)=T(D+1)
3910 NEXT D
3920 U=2
3930 T(V)=T9 \ U=U+1
3940 RETURN
3940 P4=(YB-P1)
3950 IF P4<20 GO TO 3740
3950 GO TO 1830
4000 GOSUB 2530
4010 GO TO 4020
4020 GOSUB 2770
4030 GOSUB 3850
4040 GOSUB 2940
4050 RETURN
4100 GOSUB 2870
4110 GOSUB 2940
4120 RETURN
4130 RETURN
4200 GOSUB 2530
4210 GOSUB 2740
4220 GOSUB 3100
4230 GOSUB 2940
4240 RETURN
1010 REM * * * PROGRAMME CALLED MIXTOR** - V6 * * * * 
1020 REM ***MIXING SPEC TO FIXED TORQUE USING CONVENTIONAL SPEC-MASTICATION IN.
1030 REM-VARIABLE SPEED USING TEMPERATURE.
1040 A=SYS(7,1) 
1050 DISPLAY CLEAR \ MOVE_CURSOR(1,1) 
1055 WIDE_LINE('WIDE',2) 
1060 PRINT ' PROGRAMME FOR ' 
1090 PRINT ' MASTICATION ' 
1099 PRINT ' MIXING TO FIXED TORQUE.' 
1100 DIM t2,P(129,5) 
1110 DIM r7=80 
1190 V7=T1*K 
1120 WIDE_LINE('-WIDE',15) 
1130 U=1 
1140 REM-----BATCH INFORMATION SUBROUTINE----------------------------- 
1145 B=3.15649E-01 
1150 PRINT ' INITIAL ROTOR SPEED RPM ': \ INPUT R1 
1160 I=R1*13.5231 \ REM I IS VOLTS FOR INITIAL ROTOR SPEED 
1170 PRINT ' MAXIMUM ROTOR SPEED RPM ': \ INPUT R2 
1180 F=R2*13.5231 \ REM F IS VOLTS FOR MAX ROTOR SPEED 
1190 PRINT ' MAX DUMP TEMPERATURE DEGREES C T1 ': \ INPUT T1 
1200 PRINT ' TARGET TORQUE FOR MASTICATION': \ INPUT M2 
1210 PRINT ' ROTOR SPEED FOR BLACK INCORPORATION': \ INPUT R3 
1220 K=0.025 
1230 V7=T1*K \ REM-----------------VOLTAGE FOR MAX TEMP 
1240 V2=0.05 
1250 R1=3500 \ K2=40 \ REM---------CONTROL CONSTANTS 
1260 K2=40 \ T6=2 \ REM--T6=SCANNING RATE 
1280 V4=(R1+3.3)-32 \ REM ROTOR VOLTAGE 
1290 T4=320 \ REM-REF TEMPERATURE 
1300 T8<200 TO 1510 
1310 T8<30 GO TO 1650 
1320 T7<106 \ REM---REF TEMPERATURE 
1330 PRINT ' ENTER FILE NAME FOR DATA STORAGE:' \ INPUT P* 
1340 OPEN P* FOR OUTPUT AS FILE #2 
1350 GOSUB(2245,7) 
1360 GO TO 2330 
1370 REM-----------------START SIGNAL FROM BANDURY 
1371 DISPLAY CLEAR 
1410 HTXT('B,F',24,25,'WAITING START SIGNAL..') 
1420 AIN(0,0,4) 
1430 IF M1 THEN 1450 
1440 GO TO 1420 
1450 START TIME('CHZ') 
1460 I=R1*13.5231 
1465 GOSUB 2500 
1470 HTXT('B,F',24,25,'POLYMER MASTICATION.') 
1490 GOSUB 2410 \ REM RAM DOWN 
1510 GOSUB 2530 
1520 GOSUB 2770 
1530 GOSUB 2940 
1540 IF T7>T2 GO TO 1510 
1545 IF T8<T0 GO TO 1510 
1550 HTXT('B,F',24,25,'BLACK ADDITION.') 
1560 A=1 
1570 GOSUB 2380 \ REM RAM UP 
1580 I=R3*13.5231 \ R1=R3 
1581 FOR G=0 TO 1 
1590 GOSUB 2530 
1600 GOSUB 2770 
1610 GOSUB 2940 
1620 NEXT G 
1590 AOUT(+0,0,0) \ AOUT(2045,6) 
1595 GET TIME(PF) 
1600 AOUT(+1,7) 
1602 GOSUB 2530 
1604 GOSUB 2770 
1606 GOSUB 2940 
1610 GOSUB 2410 \ AOUT(+0,0,6) 
1611 GOSUB 2530 
1612 GOSUB 2770 
1616 GOSUB 2940 
1620 AOUT(+2045,0) 
1640 HTXT('B,F',24,25,'MIXING TO PREDETERMINED TORQUE.') 
1650 GOSUB 2530 \ REM TEMPERATURE CONTROL SUBROUTINE 
1660 GOSUB 2770 \ REM TEMPERATURE CONTROL SUBROUTINE 
1670 GOSUB 2940 \ REM DATA ACCUMULATION AND GRAPH 
1672 P3=P3-P2 
1675 IF P3<30 GO TO 1650 
1680 GOSUB 2530 \ REM TEMPERATURE CONTROL SUBROUTINE 
1685 GOSUB 2770 \ REM TEMPERATURE CONTROL SUBROUTINE 
1690 GOSUB 3100 
1695 GOSUB 2940 \ REM DATA ACCUMULATION AND GRAPH 
1698 GO TO 1680 
1700 GOSUB 2390 \ REM-----------------RAM UP 
1710 HTXT('B,F',24,25,'ADD OIL.') 
1720 AOUT(+2045,4) 
1725 GET TIME(PF) 
1730 A=2 \ U=1 \ T(1)=0 \ T(2)=0 \ T(3)=0 \ T(2)=0 \ T(3)=0 \ H=0 
1731 FOR U=0 TO 2 
1732 GOSUB 2530 
1733 GOSUB 2620 
1734 GOSUB 2740 
1735 GOSUB 3100 
1736 GOSUB 2940 
1737 }
2440 REM--------------------------------GATE OPEN
2450 AOUT(2047\+1) \ PAUSE(.1) \ AOUT(0\+1)
2460 RETURN
2470 REM--------------------------------GATE SHUT
2480 AOUT(2047\+5) \ PAUSE(.1) \ AOUT(0\+5)
2490 RETURN
2500 REM--------------------------------ROTOR SPEED
2510 AOUT(1\+7)
2520 RETURN
2530 REM-----------------------------------TEMP SUBROUTINE----------------------------------
2540 FOR Z=0 TO 9
2550 AIN(V5\+1) \ 0=0+1
2560 NEXT Z
2570 RETURN
2580 REM-----------------------------------ANGULAR VELO IN RADS PER SEC
2590 V7=15.897-0.79
2600 V9=(R1*6.08643)-9.64
2610 REM-----------------------------------DATA ACCUMULATION AND GRAPHS SUBROUTINE--------------
2620 GET_TIME(TB)
2630 Z(O)=INT(TB) \ 0=0+1
2640 Z(O)=INT(15) \ 0=0+1 \ REM-----------REM MATCH TEMP IN DEGREES C
2650 Z(O)=INT(J9) \ 0=0+1 \ REM-----------REM POWER IN WATTS
2660 Z(O)=INT(11) \ 0=0+1 \ REM-----------REM ROTOR SPEED IN RPM
2670 Z(O)=INT(19) \ 0=0+1 \ REM-----------REM PREVAILING TORQUE
2680 Z(O)=INT(12) \ 0=0+1 \ REM-----------REM RUNNING TORQUE AVE
2690 IF H=2 GO TO 1760
2700 IF H=1 GO TO 3180
2710 IF Z2<Z3 GO TO 3250
2720 Z3=Z2
2730 FOR B=1 TO 2
2740 T(B)=T(B+1)
2750 NEXT
2760 V=Z2
2770 T(V)=T9
2780 RETURN
2790 REM-----------------------------------RUNNING AVERAGE OF TORQUE VALUES------------------
2800 IF V=1 THEN 3230
2810 IF V=3 THEN 3140
2820 GO TO 3230
2830 T(V)=T(V+1)
2840 RETURN
2850 IF A=1 GO TO 3227
2860 IF A=2 GO TO 3265
2870 IF P5=(18-P0) GO TO 3265
2880 IF P5<25 THEN 3180
2890 H=1 \ RETURN
2900 H=2 \ RETURN
2910 REM->>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>>
2920 REM-----------------------------------PERFORMING REGRESSION ANALYSIS-------------------------
2930 NEXT('B\+F')=23+10;'PERFORMING REGRESSION ANALYSIS.'
2940 H=3
2950 R=17
2960 AOUT(17\+7)
2970 BIN R(5\+1)
2980 FOR C=0 TO 5
2990 GOSUB 2540
3000 GOSUB 2720
3010 GOSUB 3160
3020 GOSUB 2750
3030 GOSUB 3100
3040 GOSUB 2950
3350 GOSUB 2950
3360 R(C,0)=T8
3370 R(C,1)=LOG10(T9)
3380 NEXT C
3390 X=0 \ X2=0 \ X3=0 \ X4=0 \ Y1=0 \ Y2=0 \ Y3=0
3400 X5=0 \ X6=0 \ X7=0 \ Y4=0 \ Y5=0 \ Y6=0
3410 FOR C=0 TO 5
3420 X=R(C,0)*R(C,1)
3430 X2=X*4X2
3440 NEXT C
3450 FOR C=0 TO 5
3460 X3=R(C,0)
3470 X4=X4+X3
3480 NEXT C
3490 FOR C=0 TO 5
3500 X5=R(C,0)*R(C,0)
3510 X6=X6+X5
3520 NEXT C
3530 Y3=Y2*4X
3540 FOR C=0 TO 5
3550 X5=R(C,0)*R(C,1)
3560 X6=X6+X5
3570 NEXT C
3580 X7=X4+X2
3590 FOR C=0 TO 5
3600 Y4=R(C,1)*R(C,1)
3610 Y5=Y5+Y4
3620 NEXT C
3630 MO=((6*X2)-Y3)/((6*X6)-X7)
3635 IF MO>0 THEN H=0 \ GO TO 1770 \ REM TO ENCORE NEGATIVE SLOPE
3640 CO=(Y2-60*X4))/6
3650 Y6=Y2*2
3660 R6=SQR((MO*(X2-Y3))/Y5-Y6/6))
3670 MOVE CURSOR(25,10) \ PRINT 'TIME TO DUMP
3700 X1=(LOG10(T4)-CO)/MO \ REM CALACULATED TIME TO DUMP
3710 GET_TIME(T8)
3715 V1=1 \ T(1)=0 \ T(2)=0 \ T(3)=0 \ T2=0
3720 MOVE CURSOR(6,20) \ PRINT 'TIME TO DUMP=X1' \ PRESENT TIME=T8
3721 P=1
3722 R4=(X1-T8)*(R1/60)
3724 IF R4<=40 GO TO 1821
3725 GO TO 3740
3740 GOSUB 4000
3775 IF P=1 GO TO 3723
3780 IF TB<X1 GO TO 3740
3790 ADUT(17,17) \ I=17
3800 RI=R7
3805 GOSUB 2770
3806 GOSUB 2530
3808 GOSUB 3830
3810 GOSUB 2950
3820 D9=T9
3825 D0=D9*(EXP(-B*(T5-75))
3830 IF D8<T4 GO TO 3940
3840 GO TO 3740
3850 IF V=1 THEN 3910
3852 IF V=3 THEN 3970
3860 GO TO 3910
3870 T(V)=T9
3875 T0=T1+T(2)+T(3)
3880 T2=T0/3
3885 FOR D=1 TO 2
3890 T(D)=T(D+1)
3895 NEXT D
3900 V=2
3910 T(V)=T9 \ V=V+1
3920 RETURN
3940 P4=(T8-P1)
3941 IF P4<20 GO TO 3740
3950 GO TO 1830
4000 GOSUB 2620
4010 GOSUB 2620
4020 GOSUB 2760
4030 GOSUB 3050
4040 GOSUB 2940
4050 RETURN
1000 REM ****************************
1010 REM PROGRAMME CALLED RSETF** V1
1020 REM ****************************
1030 REM - MIXING SPEC TO FIXED TORQUE USING CONVENTIONAL SPEC-MASTICATION IN.
1035 REM-STRIPPED DOWN VERSION OF RTO4 FOR FASTER SAMPLING RATE
1040 A=SYS(7.1+)
1050 DISPLAY_CLEAR \ MOVE_CURSOR(1,1)
1055 PRINT '* PROGRAMME FOR
1060 PRINT " MIXING TO PRESET TORQUE"
1070 DIM A2,P(156,4)
1080 DIM Z(780),T(J)
1090 V=1
1100 B=-3.15649E-03
1110 IF P=7 GO TO 1330
1130 R1=75 \ GO TO 1160
1140 PRINT ' ROTOR SPEED RPM I" \ INPUT RI
1150 I=R1*13.5231
1160 K=.025 \ V2 c .05
1170 V7=T1*K
1180 V9=(R1*3.3)-33 \ REM MOTOR VOLTAGE
1190 IF (RI>3.3) GO TO 1130
1200 OPEN P$ FOR OUTPUT AS .2
1210 AOUT(,0,.,7)
1220 GO TO 2330
1230 REM----------------------_------START SIGNAL' FROM BANSURY
1240 DISPLAY-CLEAR
1245 HTEXT('B,F',24,25,'AWAITING START SIGNAL.')
1250 AIN(+0,+,4)
1260 IF I>3 THEN 1450
1270 GET TIME('CHZ')
1280 GOSUB 2500
1290 HTEXT('B,F'+24,25,'POLYMER MASTICATION.')
1300 GOSUB 2410 \ REM RAM DOWN
1310 GOSUB 4100
1320 IF T8<20 GO TO 1510
1330 HTEXT('s,F',24,25,'BLACK ADDITION.')
1340 A=1
1350 GOSUB 2380 \ REM RAM UP
1360 FOR G=0 TO 1
1365 GOSUB 4100
1370 NEXT G
1380 AOUT(0,0,0) \ PAUSE(.1) \ AOUT(2045,+,6)
1390 GET_TIME(P2)
1400 GOSUB 4100
1410 GOSUB 2410 \ AOUT(0,0,6)
1420 GOSUB 4100
1430 AOUT(+2045,+,0)
1440 HTEXT('B,F' +24,25,'MIXING TO PREDETERMINED TORQUE.')
1450 GOSUB 4100
1460 GOSUB 4500
1470 GOSUB 2380 \ REM-------------------RAM UP
1480 HTEXT('B,F' +24,25,'ADD OIL.')
1490 AOUT(+2045,+,4)
1500 GET_TIME(P0)
1510 A=2
1520 FOR W=0 TO 3
1525 GOSUB 4100
1530 NEXT W
1540 GOSUB 2410 \ REM RAM DOWN
1550 AOUT(0,0,4)
1560 GOSUB 4100
1570 HTEXT('B,F' +24,25,'MIXING TO PREDETERMINED TORQUE.')
1580 GOSUB 2300
1590 FOR W=0 TO 1
1600 GOSUB 4100
1610 NEXT W
1620 GOSUB 2410 \ AOUT(+2045,+,6)
1630 MOVE_CURSOR(3,25) \ GET_TIME(P1) \ DISPLAY_MODE('BRIGHT')
1640 A=3 \ GOSUB 4100
1645 AOUT(0,0,0) \ AOUT(+2045,+,6)
1650 MOVE_CURSOR(3,25) \ DISPLAY_MODE('BRIGHT') \ PRINT
1660 AOUT(+0,+,6) \ AOUT(+2045,+,0)
1670 GOSUB 4100
1680 GOSUB 4100
1690 AOUT(+0,+,6) \ AOUT(+2045,+,0)
1700 GOSUB 4100
1710 AOUT(+0,+,6) \ AOUT(+2045,+,0)
REM*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*..........................
2740 REM*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*-*..........................
2750 REM--------------TORQUE SUBROUTINE---------------------------------------------
2760 W1=(R1/60)*6.2832 \ REM------------ANGULAR VEL IN RADS PER SEC
2770 V9=(R1*3.3)-33 \ REM MOTOR VOLTAGE
2780 FOR X=0 TO 99
2790 2000 AIN([V5,]]
2810 2810 07=07+V5
2820 2820 NEXT X
2830 2830 V5=07/100
2840 Q7=0
2850 A5=(V5*15.887)-.79
2860 J7=A5*V9
2870 J7=(R1*6.0643)-98.64 \ REM J7 IS TORQUE FOR EMPTY B/B.T9 IS TOTAL TORQUE
2880 W2=(J9+2.21)/1000 \ REM--ENERGY IN K/J
2890 W4=W2/W4
2910 RETURN
2920 REMxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx
2930 REMxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx
2940 REM--DATA ACCUMULATION AND GRAPH SUBROUTINE-----------------
2950 GET_TIME(T8)
2960 Z(O)=TI) \ 0=0+1
2970 Z(O)=INT(T5) \ 0=0+1 \ REM--------REM BATCH TEMP IN DEGREES C
2980 Z(O)=INT(J9) \ 0=0+1 \ REM--------REM POWER IN WATTS
2990 Z(O)=INT(T9) \ 0=0+1 \ REM--------REM PREVAILING TORQUE
3000 Z(O)=INT(W4) \ 0=0+1 \ REM--------REM TOTAL ENERGY
3070 RETURN
3080 REMxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx
3090 REMxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxxx
4100 GOSUB 2540
4110 GOSUB 2770
4120 GOSUB 2950
4130 RETURN
4500 AIN(-6,+,4)
4510 IF Q<3 THEN RETURN
4520 IF A=1 GO TO 1700
4530 IF A=2 GO TO 1821
4540 IF A=3 GO TO 1835
APPENDIX B

In Chapter 5 an experiment was carried out which compared the rheological properties of final mixed batches, produced using the batch condition control system, with those produced using a unit work based mixing specification. The program for mixing to a unit work specification was called BRCONT and is contained within this appendix. It contained a mastication step, and was for a variable rotor speed machine and so contained the PID loop to control the batch temperature.
1000 REM ********************
1010 REM SUBROUTINE-****************
1025 REM TO MIX CONTROL BATCHES TO UNIT WORK FOR EXPERIMENT B
1030 REM ----------------- MIXING SPEC TO FIXED TORQUE USING CONVENTIONAL SPE-MASTICATION IN.
1040 A=SYS(7,1)
1050 DISPLAY_CLEAR \ MOVE_CURSOR(1,1)
1060 PRINT 'ROTOR SPEED FOR TARGET TORQUE: ' \ INPUT R1
1070 E=R1*3.15649E-03
1080 F=R2*3.15649E-03
1090 PRINT 'MAX DUMP TEMPERATURE DEGREES C: ' \ INPUT T1
1100 M2=250 \ GO TO 1160
1110 PRINT 'TARGET TORQUE FOR MASTICATION: ' \ INPUT M2
1120 R3=75 \ GO TO 1220
1130 PRINT 'ROTATOR SPEED FOR BLACK INCORPORATION: ' \ INPUT R3
1140 K=.025 \ VZ=0.5
1150 V7=118K \ REM V7=1200-V/A \ REM R7=100 \ REM CONTROL CONSTANTS
1160 K3=100 \ TV=2 \ REM T3=SCANNING RATE
1170 V9=KRI3.31-33 \ REM MOTOR VOLTAGE
1180 R7=75
1190 I7=R7*13.5231 \ REM VOLS FOR ROTOR SPEED AT TARGET TORQUE
1200 PRINT 'ENTER FILE NAME FOR DATA STORAGE: ' \ INPUT F*
1210 OPEN F* FOR OUTPUT AS FILE #2
1220 AOUT(,2045",0)
1245 AOUT(,0,,7)
1250 GO TO 2330
1260 REM-----------------------------START SIGNAL FROM DANBURY
1270 DISPLAY_CLEAR
1410 HTXT('B,F',24,25, 'AWAITING START SIGNAL.')
1420 AIN(,0,,4)
1430 IF O1 THEN 1450
1440 GO TO 1420
1450 START_TIME('CHZ')
1460 I=R1*13.5231
1470 GOSUB 2500
1470 HTXT('B,F',24,25, 'POLYMER MASTICATION.')
1480 PAUSE(5)
1490 GOSUB 2410 \ REM RAM DOWN
1510 GOSUB 4100
1535 PAUSE(1)
1540 IF W4<60 GO TO 1510
1550 HTXT('B,F',24,25, 'BLACK ADDITION.')
1570 GOSUB 2380 \ REM RAM UP
1580 I=R3*13.5231 \ R1=R3
1581 FOR 0=0 TO 1
1582 GOSUB 4100
1587 NEXT O
1590 AOUT(,0,,6) \ PAUSE(,1) \ AOUT(,2045,,6)
1595 GET_TIME(P2)
1600 AOUT(,I,,7)
1602 GOSUB 4100
1610 GOSUB 2410 \ REM (4,,6)
1612 GOSUB 4100
1620 AOUT(,2045,,6)
1640 HTXT('B,F',24,25, 'MIXING TO PREDETERMINED TORQUE.')
1650 GOSUB 4100
1655 IF W4<210 GO TO 1650
1670 GOSUB 2380 \ REM ----------------- RAM UP
1710 HTXT('B,F',24,25, 'ADD OIL.')
1720 AOUT(,2045,,4)
1725 GET_TIME(P0)
1731 FOR W=0 TO 2
1732 GOSUB 4200
1737 NEXT W
1740 GOSUB 2410 \ REM RAM DOWN
1750 AOUT(,0,,4)
1760 HTXT('B,F',24,25, 'MIXING TO PREDETERMINED TORQUE.')
1761 GOSUB 4200
1762 GOSUB 4200
1763 GOSUB 2390
1764 GOSUB 4200
1765 GOSUB 2420
1770 GOSUB 4200
1780 IF W4>400 GO TO 1770
1821 GOSUB 2330
1922 MOVE_CURSOR(3,25) \ GET_TIME(P1) \ DISPLAY_MODE('BRIGHT')
1923 GOSUB 4200
1924 GOSUB 4200
1925 AOUT(,0,,6) \ AOUT(,2045,,6)
1926 MOVE_CURSOR(3,25) \ DISPLAY_MODE('BRIGHT') \ PRINT'
1927 GOSUB 2410
1928 GOSUB 4200
1929 AOUT(,0,,6) \ AOUT(,2045,,6)
1930 GOSUB 4200
1932 IF W4<530 GO TO 1930
1934 GO TO 3779
1939 GOSUB 2380 \ REM ----------------- RAM UP
1940 GOSUB 2440 \ REM ----------------- DATE OPEN
1960 HTXT('B,F',24,25, 'BATCH DUMPED.')
1964 GOSUB 2410
1970 W4=0
1862 GOSUB 2410
1865 RS=(T8-P1)*K(R1/60)
1870 AOUT((-500,...) \ REM-----------------TORQUE TO INITIAL RPM
1880 HEXT("E"F)+24,25,"STORING DATA ON FLOPPY DISC.
1890 FOR M=0 TO (O11/6
1900 FOR J=0 TO S
1910 P(M,J)=Z(U2)
1920 1930 U2=U2+1
1940 NEXT J
1950 NEXT
1960 CLOSE 1
1970 OPEN "LP:" FOR OUTPUT AS FILE 11
1980 PRINT U,'Data file name _'p'
1990 PRINT .,'ACTUAL TORQUE='T9'ACTUAL DUMP ENERGY = 'W4
2000 PRINT 11,'TARGET TEMPERATURE='T1'ACTUAL TEMPERATURE='TS
2010 PRINT 11,'TOTAL ENERGY K/J = 'W4'TOTAL WORK MJ/H='W4/11.607
2015 PRINT 11,'ADJUSTED TORQUE='B8'PIGMENT ADDITION TIME='P1
2020 PRINT 11,'ROTOR REV'S AFTER PIGMENT ADDITION='R5'BLACK ADDITION TIME='P2
2025 PRINT 11,'OIL ADDITION TIME='PO
2030 PRINT 'PRINT press RETURN button to review data '
2035 INPUT AS
2040 DISPLAY_CLEAR
2045 GaSUB 2380
2050 GOSUB 2470
2055 AOUTC,O",7) , PAUSE(.1) , AOUT(,0",3)
2060 RETURN
2065 REM-----------------GATE OPEN
2070 AOUT(,R047",1) , PAUSE(.1) , AOUT(,0",1)
2080 RETURN
2085 REM-------------------------------GATE SUUT
2090 AOUT(,2047",5) , PAUSE(.1) , AOUT(,0",5)
2095 RETURN
2100 REM----------------TEMP SUBROUTINE-----------------------
2105 FOR Z=0 TO 9
2110 AIN(,Y",9)
2115 S=SfY
2120 NEXT Z
2125 V6=S/10
2130 T5="V6/K
2135 RETURN
2140 V7=V8
2145 I=Ul
2150 RETURN
2155 REM-------------------ROTOR SPEED
2160 AOUT(,1",7) 
2170 RETURN
2175 REM-------------------------------TEMP SUBROUTINE
2180 FOR Z=0 TO 9
2185 AIN(,Y",9)
2190 S=S+1Y
2195 NEXT Z
2200 U=U-58/10
2205 S=0
2210 T5=V6/K
2215 RETURN
2220 U7=T1*K
2225 VB=V7-V6 \ REM-----------V8 IS ERROR VOLTAGE
2230 E=E+VB
2235 E1=(VB-L)
2240 U1=(K1*VB)+(K2*E)+(K3*E1/T6)
2245 IF U1=F THEN U1=F
2250 U1=20415.5231
2255 IF U1=I THEN U1=I
2260 AOUT(U1,",?)
2265 U2=U8
2270 L=U8
2275 I=U1
2280 RETURN
2285 REM ---------TORQUE SUBROUTINE---------------------
2290 R1=U1/13.5231
2295 U1=(11/60)*UL*2037 \ REM-----------V8 IS VEL. IN RMS PER SEC
2299
2730 REM *****************************************************
2740 REM -----------------------------------TORQUE SUBROUTINE-----
2750 REM --------------------------------------------------
2760 R1=U1/13.5381
2770 U1=U1/60+2.032 \ REM --------------------------------------------------ANGULAR VEL IN RADS PER SEC
2780 U9=(R1*3.3)-33 \ REM MOTOR VOLTAGE
2790 FOR X=0 TO 99
2800 AIN(U5,+,11)
2810 07=07+V5
2820 NEXT X
2830 V5=07/100
2840 07=0
2850 A5=(U5*15.897)-.79
2860 J9=AS*U9
2870 J7=(U1.*08643)-93.64 \ REM J7 IS TORQUE FOR EMPTY B/B.T9 IS TOTAL TORQUE
2870 T9=(J9-J7)/(1.07641) \ REM J7 IS TORQUE FOR EMPTY B/B.T9 IS TOTAL TORQUE
2870 W2=(J9*2.21)/1000 \ REM--ENERGY IN K/J
2870 W4=W2+W4
2880 RETURN
2890 REM----------------------------------DATA ACCUMULATION AND GRAPH SUBROUTINE------------------------
2900 GET_TIME(T8)
2910 Z(0)=INT(T5) \ 0=0+1 \ REM------------------REM BATCH TEMP IN DEGREES C
2920 Z(0)=INT(J9) \ 0=0+1 \ REM------------------REM POWER IN WATTS
2930 Z(0)=INT(R1) \ 0=0+1 \ REM------------------REM ROTOR SPEED IN RPM
2940 Z(0)=INT(W4) \ 0=0+1 \ REM------------------REM TOTAL ENERGY
2950 RETURN
2960 REM----------------------------------
2970 R1=R7
2980 PAUSE(3)
2990 GOSUB 2620
3000 GOSUB 2760
3010 D8=T9*eEXP(-S*(T5-T7))
3020 GO TO 1538
3030 REM----------------------------------
3040 REM----------------------------------
3050 AQUIT(+17,+,7) \ R1=R7
3060 PAUSE(3)
3070 GOSUB 2530
3080 GOSUB 2770
3090 GOSUB 2940
3100 DB=T9*EEXP(-S*(T5-T7))
3110 GO TO 1038
3120 GOSUB 2530
3130 GOSUB 2770
3140 GOSUB 2940
3150 RETURN
3160 GOSUB 2530
3170 GOSUB 2620
3180 GOSUB 2760
3190 GOSUB 2940
3200 RETURN
REM**********CHECK BANDURY READY SUBROUTINE***************
2000 GOSUB 2100 \ REM----------RAISE RAM
2010 GOSUB 2200 \ REM----------CLOSE GATE
2020 RETURN
2070 REMXXXXXXXXXXXXX--BANDURY FUNCTION SUBROUTINES--XXXXXXXXXXXXXXXX
2100 REM----------RAISE RAM
2110 AOUT(2,2045++3) \ PAUSE(.1) \ AOUT(1,0++,3)
2120 RETURN
2130 REM------LOWER RAM
2140 AOUT(2,2045++2) \ PAUSE(.1) \ AOUT(1,0++,2)
2150 RETURN
2200 REM------DATE SHUT
2210 AOUT(2,2045++5) \ PAUSE(.1) \ AOUT(1,0++,5)
2220 RETURN
2230 REM------DATE OPEN
2240 AOUT(2,2045++1) \ PAUSE(.1) \ AOUT(1,0++,1)
2250 RETURN
2300 REM------SET ROTOR SPEED
2310 l=R1+13.5231
2320 AOUT(1,0++,7)
2330 RETURN
APPENDIX C

This appendix contains all the other miscellaneous computer programs which were developed and used in this work.

**BRTRI** - this program allowed the evaluation of the PID constants which are used in the temperature control loop. The calculated values were introduced into the equation and a mixing cycle followed. The program produced a split screen with a graph of rotor speed and a graph of batch temperature. The rate of temperature rise and the response of the rotor speed to it could be observed. At the bottom of the screen was printed the total error voltage plus the values for the proportional, integral and derivative terms. There the exact effect of each constant could be evaluated as the cycle progressed.

**BREMP** - it was necessary to determine the amount of energy used to actually mix the rubber compound. To do this one needed to know how much energy was used in the running of the internal mixer. Therefore the internal mixer needed to be run in an unloaded condition and power consumption accurately determined over a range of rotor speeds. This program automatically increases the rotor speed of the Banbury and measures the power used by the motor at each speed. The data so obtained was used to develop the equation used in the control programs for obtaining actual energy used in the mixing process.

**LUTTMS** - the viscosity data in this thesis has been generated on either the TMS rheometer at the Avon Rubber Company or the one at Loughborough University of Technology. In either case the shear stress was proportional to an output voltage. This program calculated the shear stress from the voltage and then performed a linear regression of log (shear stress) versus log (shear rate). The intercept is the apparent viscosity \( \eta_0 \) at 1 sec\(^{-1} \) and the slope is the power law index \( n \). The program prints
out both $n_0$ and $n$, as well as the regression coefficient so that the validity of the data points can be evaluated.
**Programme Called BRH**

---

**Target Temperature and Rotor Speed Evaluation Programme**

- **Target Temperature**
  
  \[ T_1 = 100 \text{ \degree C} \]

- **Initial Rotor Speed**
  
  \[ R_1 = 50 \]

- **Variable Calculations**
  
  \[ F = (K2 \times 13.52) / 21.7 \]

- **Rotor Response Calculation**
  
  \[ U_1 = (K1 \times V_8) + (K2 \times E) + (K3 \times E / T_6) \]

- **Ensure Rotor Speed Limit**
  
  \[ U_1 = \min(11, U_1) \]

---

**Visualisation and Output**

- Labels and Grids
  
  \[ \text{GRID(1,1)} \]

- Data Points
  
  \[ \text{POINT}(Z, R_8, 1) \]

- Data Movement
  
  \[ \text{MOVE_CURSOR}(23, 1) \]

- Console Output
  
  \[ \text{PRINT 'TOTAL ERROR: '}, E', 'ERROR VOLT: '}, V_8', 'ROTORSPEED: '}, R_8', 'BATCH TEMP: '}, T_5' \]

---

**Control Instructions**

- **Gate Shutter**
  
  \[ \text{IF } O < 1 \text{ THEN } 210 \]

- **Rotor Speed Adjustment**
  
  \[ \text{IF } O > 6 \text{ THEN } 210 \]

---

**Additional Notes**

- **Termination**
  
  \[ \text{STOP} \]
10 REM Program to obtain power values for empty bank
20 REM
30 REM
40 REM
50 DIM 42(40,4)
60 DIM R(60)
70 R=20
80 PRINT 'enter file name for data storage:' 
90 INPUT P$ 
100 FOR X=0 TO 99
110 AOUT(,X7) 
120 FOR X=O TO 99
130 AOUT(,X7) 
140 PRINT 'press Y when you are ready to proceed!' 
150 DISPLAY_CLEAR
160 IF R=120 GO TO 130
170 IF R=120 GO TO 130
180 PRINT 'TEST COMPLETE.' 
190 FOR M=0 TO 10
200 FOR J=0 TO 4
210 P(M,J)=Z(U2) 
220 U2=U2H 
230 NEXT J 
240 NEXT M 
250 CLOSE 
260 PRINT 'do you want to print out data:' 
270 INPUT W$ 
280 IF W$='N' GO TO 630 
290 OPEN P$ FOR INPUT AS FILE 4 
300 FOR M=0 TO 10
310 FOR J=0 TO 4
320 PRINT 'ROTOR SPEED=INT(R)'
330 NEXT J 
340 NEXT M 
350 STOP
REM-------------------PROGRAM CALLED LUTHS-----------------------------
20 REM--CALCULATES DATA FROM THE LUT TMS RHEOMETER----------------------
25 REM--SHEAR RATES 1,3,5,7,10,20
30 DISPLAY.CLEAR
40 PRINT 'INPUT TOTAL NUMBER OF DATA SETS:'; INPUT G
50 V=1-1
60 PRINT 'INPUT BATCH IDENTIFICATION: '; INPUT P#
70 REM---------------------REGRESSION ANALYSIS----------------------------
80 DIM R(5,1)
90 R(0,0)=0
100 R(1,0)=.477121
110 R(2,0)=.69897
120 R(3,0)=.845098 \ IF O=4 GO TO 150
130 R(4,0)=1 \ IF 0=5 GO TO 150
140 R(5,0)=1.30103
150 FOR I=0 TO 0-1
160 PRINT 'VOLTS: '; INPUT V9
165 S9=V9*3468.
170 R(I,1)=LOG10(S9)
180 NEXT I
190 DISPLAY.CLEAR
200 FOR I=0 TO 0-1
210 A=R(I,0)*R(I+1)
220 B=A/B
230 NEXT I
240 FOR I=0 TO 0-1
250 Z=R(I,0)
260 B=AZ
270 NEXT I
280 FOR I=0 TO 0-1
290 E=R(I,1)
300 F=F*E
310 NEXT I
320 G=F*F
330 FOR I=0 TO 0-1
340 H=R(I,0)*R(I,0)
350 J=J+H
360 NEXT I
370 K=G/2
380 FOR I=0 TO 0-1
390 L=R(I,1)*R(I,1)
400 M=M+L
410 NEXT I
420 M=-(M-J)/((G-K)-M)
430 C=(F-(M*0))/((N-P/G))
440 P=F/2
450 PRINT #1,'TMS DATA FROM L.U.T. MACHINE.'
460 OPEN #1 FOR OUTPUT AS FILE #1
470 PRINT #1, 'BATCH IDENTIFICATION:--P#
480 PRINT #1,'**************************'
490 PRINT #1,'REGRESSION COEFFICIENT=R6
500 PRINT #1,'NETA ZERO='10'C
510 PRINT #1,'**************************'
520 PRINT #1,'REH--SHEAR RATES 1,3,5,7,10,20
530 PRINT #1,'**************************'
540 PRINT #1,'**************************'
550 PRINT #1,'**************************'
560 PRINT #1,'**************************'
570 PRINT #1,'**************************'
APPENDIX D

This appendix contains two graphs. The first is a set of typical data from the TMS Rheometer at Loughborough. The TMS Rheometer was used to evaluate the rheological properties of the polymers and mixed compounds. In practice the data sets of shear rate and resulting shear stress were entered into a computer program which calculated the intercept at 1 sec\(^{-1}\) (\(n_0\)) and the slope (\(n\)) power law index.

The second graph is a plot of the data used to determine the temperature dependence of the natural rubber compound used in this work.
## TYPICAL TMS RHEOMETER DATA

<table>
<thead>
<tr>
<th>Shear Rate $\dot{\gamma}$ (sec$^{-1}$)</th>
<th>Shear Stress $\tau$ (Pa's)</th>
<th>$\log \dot{\gamma}$</th>
<th>$\log \tau$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>75778</td>
<td>0</td>
<td>4.8795</td>
</tr>
<tr>
<td>3</td>
<td>88090</td>
<td>.47712</td>
<td>4.9449</td>
</tr>
<tr>
<td>5</td>
<td>93639</td>
<td>.69897</td>
<td>4.9715</td>
</tr>
<tr>
<td>10</td>
<td>100228</td>
<td>1.00000</td>
<td>5.001</td>
</tr>
<tr>
<td>20</td>
<td>105083</td>
<td>1.30103</td>
<td>5.0215</td>
</tr>
</tbody>
</table>

**Slope** = Power Law Index $n$

**Intercept** $n_0$

**Power law equation:** $\tau = n_0 \dot{\gamma}^n$
DETERMINATION OF TEMPERATURE DEPENDENCE OF NATURAL RUBBER COMPOUND

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Mooney Torque</th>
</tr>
</thead>
<tbody>
<tr>
<td>90</td>
<td>116.4</td>
</tr>
<tr>
<td>95</td>
<td>117.0</td>
</tr>
<tr>
<td>100</td>
<td>115.5</td>
</tr>
<tr>
<td>105</td>
<td>111.9</td>
</tr>
<tr>
<td>110</td>
<td>111.8</td>
</tr>
<tr>
<td>115</td>
<td>108.6</td>
</tr>
<tr>
<td>120</td>
<td>109.7</td>
</tr>
<tr>
<td>125</td>
<td>106.5</td>
</tr>
<tr>
<td>130</td>
<td>102.0</td>
</tr>
</tbody>
</table>

Plot of log Mooney torque versus temperature