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Optimising the Continuous Final Mixing of Rubber Compounds

Simon Benedikt Wachter

Dublin Institute of Technology

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Optimising the continuous final mixing of rubber compounds

PhD Thesis

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February 2014

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Dr Niall Murphy

Dr. Olaf Skibba
I certify that this thesis which I now submit for examination for the award of PhD, is entirely my own work and has not been taken from the work of others, save and to the extent that such work has been cited and acknowledged within the text of my work.

This thesis was prepared according to the regulations for postgraduate study by research of the Dublin Institute of Technology and has not been submitted in whole or in part for another award in any other third level institution.

The work reported on in this thesis conforms to the principles and requirements of the DIT's guidelines for ethics in research.

Signature _______________________________ Date ________________

Candidate
Summary

The primary aim of this research project was to uniquely redesign, develop and validate the continuous final mixing of rubber compounds using single screw extruder gear pump combinations.

The thesis describes the introduction of novel methods and reconfiguration of final mixing as a continuous process in a single screw extruder. This led to the elimination of one of the mixing steps in an internal mixer for most compounds and the continuous mixing of curing agents or additional filler material into the base compound. The radical changes introduced have several advantages for the process allowing cost and energy savings during the production of rubber compounds.

The project started with evaluation of the current available technology and scientific background of rubber processing and mixing in single screw extruders. The combination of technologies, such as different mixing geometries in screws and barrels and the combination of these systems with a gear pump allowed a new method of final mixing to be proposed, introduced and evaluated.

Following initial basic feasibility trials, the initial concept was elevated to the design and build of an industrial scale system. Finally, the system was rigorously evaluated and the outcomes from testing proved that scaling up and the transition to the new method into a working industrial system was successful. The resulting production machine is capable of final mixing conventional rubber compounds on an industrial scale and will replace the batch to batch process currently used.
I. Acknowledgement

This work would not have been possible without the support and help of certain people and institutions.

Therefore, I would first like to thank Prof. Steve Jerrams and Dr. Olaf Skibba for their supervision and for all the support they have given me.

I am also grateful to the Dublin Institute of Technology and the Centre for Elastomer Research for giving me the opportunity to carry out this research project and the support they have provided for it.

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Finally I would like to thank Tina for her patience and great support during the research and especially the writing phase of this report.
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<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
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<tbody>
<tr>
<td>.csv</td>
<td>comma separated value file</td>
</tr>
<tr>
<td>cm³</td>
<td>cubic centimetre</td>
</tr>
<tr>
<td>dm³</td>
<td>cubic decimetre</td>
</tr>
<tr>
<td>EPDM</td>
<td>ethylene propylene diene monomer rubber</td>
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<tr>
<td>g</td>
<td>gram</td>
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<tr>
<td>h</td>
<td>hour</td>
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<tr>
<td>K</td>
<td>Kelvin</td>
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<tr>
<td>kg</td>
<td>kilogram</td>
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<tr>
<td>LIW</td>
<td>loss in weight</td>
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<td>m</td>
<td>meter</td>
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<tr>
<td>mesh</td>
<td>number of wires per inch for strainer meshes</td>
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<tr>
<td>mesh</td>
<td>wires per inch</td>
</tr>
<tr>
<td>mm</td>
<td>millimetre</td>
</tr>
<tr>
<td>MMI</td>
<td>man machine interface – touch screen and input / output devices</td>
</tr>
<tr>
<td>MU</td>
<td>Mooney units</td>
</tr>
<tr>
<td>NBR</td>
<td>Nitrile butadiene rubber</td>
</tr>
<tr>
<td>NR</td>
<td>natural rubber</td>
</tr>
<tr>
<td>p₁</td>
<td>inlet pressure gear pump / outlet pressure extruder</td>
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<tr>
<td>p₂</td>
<td>outlet pressure gear pump</td>
</tr>
<tr>
<td>PVC</td>
<td>Polyvinyl chloride</td>
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<tr>
<td>Term</td>
<td>Definition</td>
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<td>------------</td>
<td>---------------------------------------------------------------------------</td>
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<tr>
<td>PID controller</td>
<td>proportional–integral–derivative controller</td>
</tr>
<tr>
<td>PP S</td>
<td>Planetruder S – 30 mm planetary pump extruder</td>
</tr>
<tr>
<td>rev</td>
<td>revolution</td>
</tr>
<tr>
<td>rpm</td>
<td>revolutions per minute</td>
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<tr>
<td>s</td>
<td>seconds</td>
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<tr>
<td>S</td>
<td>torque</td>
</tr>
<tr>
<td>S’</td>
<td>1st derivative of torque</td>
</tr>
<tr>
<td>S”</td>
<td>2nd derivative of torque</td>
</tr>
<tr>
<td>SME</td>
<td>specific mechanical energy</td>
</tr>
<tr>
<td>T1</td>
<td>compound inlet temperature gear pump / outlet temperature extruder</td>
</tr>
<tr>
<td>T2</td>
<td>outlet temperature gear pump</td>
</tr>
<tr>
<td>TCU</td>
<td>temperature control unit</td>
</tr>
<tr>
<td>TPE</td>
<td>Thermoplastic elastomer</td>
</tr>
<tr>
<td>W</td>
<td>watt</td>
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Chapter 1

Introduction

Rubber is one of the most important materials today and is used in a wide variation of applications. In Europe, the existence of this material has only been known since the first Spanish reports of it from South America arrived, the history of rubber’s use dates back much further. The South American indigenes used rubber for thousands of years; archaeological findings confirm its use back to 1000 years B.C. Scientific research in Europe started in 1736 when the first rubber samples arrived at the French scientific society in Paris. The main issue, to make long-lasting and durable products, could not be resolved until as recently as one hundred and seventy years ago. The first successful rubber vulcanising technology was invented by in 1893 by Charles Goodyear and was significantly improved four years later by Thomas Hancock who also made the first rubber tyres for carriages. This technology led subsequently to a huge field of applications for rubber [1]

Rubber was initially only a natural product harvested from rubber trees in the wilderness and later in plantations. At the beginning of the last centenary, great efforts led to the successful formulation of rubber in laboratories. Still industrial production stayed low before world war two. The natural rubber supply situation forced the build up of increasing capacities for industrial production which has kept pace with new manufacturing processes until today. These industrially produced rubbers widen the range of available rubber types and formulations, however natural rubber is still an important material for numerous applications. [2]
In 2010 the worldwide production of natural rubber reached about ten million tons, this is roughly 40 % of the worldwide overall demand for rubber. Almost all rubber products are made from compounded rubber, consisting of a mixture of polymers, fillers and chemical additives in order to meet specific requirements of the different end products [1].

The mixing of these compounds has occurred in response to the technological possibilities arising since the time that manufacturing of rubber started. The first machines to process and mix rubber were invented and patented at the end of the 19th centenary. Open mills and Banbury type internal mixers were invented in this time allowing rubber compound mixing on an industrial scale. [3]

Since the end of the 19th centenary single screw extruders based on the Archimedes screw in closed barrels had their first applications. [3] Those machines were hot feed and a major step was achieved in the 1950s when the first cold fed single screw extruders became available. The last step in this development was made by the combination of single screw extruders and positive displacement gear pumps, allowing a good pressure building capability while maintaining a very constant output.

In the 1930s the first twin screw extruders were introduced in the rubber industry with limited reliability. The breakthrough came 30 years later by the introduction of much improved thrust bearings. These machines were then able to show mixing capabilities superior to single screw extruders. [4].

Based on these experiences, most research of continuous mixing has concentrated on twin screw extruders based on knowledge of their good mixing capabilities.

Notable research has been undertaken by Skibba [5] and followed up by Bogun [6] using twin screw extruders to mix rubber compounds. Both researchers had to base their
mixing on powdered rubber as this represented the only possibility of feeding a twin screw system. They showed the principal process to be a successful way to mix rubber compounds. Despite their good mixing results, the high process temperatures limit the efficiency of this process which together with the high price for powdered rubber rendered industrial implementation unfeasible.

Alternatively, the single screw extruder has been well-established throughout the rubber industry for many years. While it is widely used for processing and homogenisation, many different trials have also been undertaken to use it for mixing.

In particular research conducted by Meiertoberens and Michaeli at the Institut für Kunststofftechnik in Aachen [7] showed possibilities and limitations of the single screw extruder mixing capabilities using a special mixing zone design. The final limitation of the process is again the resulting high temperature as a result of mixing and pressure build-up in the extruder.

Despite the different approaches made to date, no actual process has successfully been transferred from research into industry.

Based on his results, Skibba suggested the investigation of extruder gear pump combinations as mixing devices. This allows combining the mixing capabilities of single screw extruders at low output pressures with the energy efficient pressure build-up and output metering capabilities of a gear pump [5].

A proposal to investigate this new approach formed the basis of the research programme set up between DIT and VMI to determine the continuous mixing capabilities of a single screw extruder gear pump combination.
1.1 Main research question

This thesis describes research carried out to develop a final mixing process from an initial beginning with small laboratory tests through a transformation into an industrial sized system.

The central question addressed in this research is ~

Can the continuous final mixing process for rubber in a single screw extruder gear pump combination be optimised to a level where it can be introduced effectively on an industrial scale?

1.1.1 Primary aim

Addressing the central question in this research prompted the following primary aim.

The primary aim of this research is to describe and further optimise the continuous final mixing of rubber compounds using single screw extruder gear pump combinations.

The validation of such a system in a real world industrial environment is necessary in order to allow mixing on an industrial scale

1.1.2 Objectives

The objectives given here emanated from the primary aim of the research:

a) To show that continuous mixing of metal flakes is possible in a single screw extruder resulting in a constant and reliable mixing quality.

As a consequence of a)
b) To develop the basic set-up for a single screw extruder gear pump combination to evolve the continuous mixing system via an R&D approach into a real industrial system

c) To carry out and validate an initial set of trials on an industrial scale under realistic production conditions.

The research question with its aims and objectives will be answered in this thesis in the following chapters.

Chapter 2 describes the development of procedures for mixing rubber, starting with a literature review of the process for the continuous mixing of elastomeric materials up to the current state-of-the-art in the discipline. It also describes the technology and processes used throughout this research programme.

Chapter 3 presents materials and methods for the full range of experimentation and development of the continuous mixing process.

Chapter 4 includes the results and discussions of extrusion trials and experimental validation of the previously discussed theoretical developments to improve the process set-up.

Chapter 5 presents the conclusions of the research programme and demonstrates that the primary aim of the research has been achieved by the introduction of novel technology in the discipline. Finally, suggestions for future research in this topic are offered and briefly discussed.
Chapter 2

The mixing of rubber – a review of procedures and relevant literature

2.1 Literature review

The literature is reviewed against the background of traditional rubber compounding and continuous mixing developments. Further, the current state of plastic mixing processes are compared with rubber compounding.

2.1.1 Traditional rubber compounding batch to batch

Rubber, as used for a wide variety of products, is actually a mixture of different basic raw materials where the base is a natural or synthetic rubber. To improve the properties of the end product, numerous other materials are mixed into this base compound. Such additives might be included for their mechanical properties such as abrasion resistance, or chemical properties such as UV stabilisation or hardness. Also processing aids may be included, allowing the material to run more smoothly in the machines. The most common addition to a rubber compound is carbon black (which is also responsible for the black colour of most rubber products) but silica, oil, curing agents, ageing inhibitors, UV-protection, softeners, foam builders and many more chemicals may also be used in a rubber compound recipe. [8]

Rubber compound mixing has been carried out since the invention of vulcanisation and the early use of rubber [8]. Several improvements in this technology have led to the two current main categories of mixing equipment; two roll mills and internal mixers.
2.1.2 Two roll mill

Two roll mills have been used since the beginning of rubber processing. The first mills were developed by the middle of the 19th century [8].

The principal set-up is simple; two rolls contra-rotate at different speeds (though sometimes at the same speed depending on the process). The gap between the rolls is adjustable and the temperature of each roll can be set independently using temperature controlled water.

Due to the speed difference in the gap, shear is applied to the material while it is moved through the rolls. Running at the same speed allows elongation mixing. This mixes, masticates and homogenises the material. The different mixing types are discussed in detail in section 2.3.1.

Mixing cycle times on a mill vary between 15 and 40 minutes. Furthermore, due to the high degree of manual labour, the operator and his experience influence the outcomes of the mixing process. Human errors involved in weighing of chemicals also accumulate. This was shown by Peter Uytterhaegen of Bekina during a presentation depicting the current state of his mixing system manufactured by VMI-AZ Extrusion and also explained in an interview [9]. If a two roll mill configuration alone is used, high labour costs result in relatively high mixing costs. If the mill is used in line with an internal mixer, the situation is greatly altered since the rubber is supplied at elevated temperature to the ‘batch off mill’, due to the resulting temperature built up during mixing. Depending on the compound, typical temperatures range between 100 °C and 160 °C. In this case, the mill removes a considerable amount of energy from the compound. The open mill provides a very high surface area between the rubber and the rolls as well as to surrounding air enabling the transfer of heat. This provides numerous interesting
research topics when used to homogenise and cool compounds after the batch is dumped out of the internal mixers [1].

2.1.3 Internal mixers

Banbury mixers have been increasingly used in the rubber industry since the 1920s, mainly because they offer the following advantages over two roll mills [10]:

- Complete closed mixing chambers without dust emission while dosing with chemicals [10].
- Significant shorter mixing times leading to higher output [10].
- Simpler operation and control using automatic control systems [1].

Another advantage is the increased user safety, as the mixing chamber is completely contained and hence the process is shielded from the outside.

The mixer consists of a mixing chamber, two rotors with parallel shafts in the chamber and a stamp to close the filling opening and to apply a controlled pressure to the compound during mixing. A typical layout of such a Banbury mixer is shown in Figure 1 [11].

Depending on the design of the mixer shafts they are either run tangentially to each other or are intermeshing. The intermeshing design allows faster mixing of carbon black, silica and other fillers based on a Calendering effect between the internal mixer rotors making this machine a very effective mixer. [12]
Despite its advantages, the main disadvantage of this mixing technology is the discontinuous process which results in batch to batch variation [13]. Each batch is mixed separately so all different recipe ingredients are added in portions. Much effort has been expended on reducing variations, for example automating weighting systems or pre-packing chemicals into bags [14]. Even with these improvements, effects like the “First Batch Effect” may occur and cause variations. Following a recipe change, the mixing chamber does not attain the required temperature. In some cases the quality of up to the first four mixes is unsatisfactory. This effect arises with the production of the first batches of one recipe due to a too low chamber temperature while starting the internal mixer and this leads to a mixture of lower quality [15].
2.1.3.1 Increase in compound temperature during mixing

The energy input during mixing using an internal mixer is very high which in turn leads to high temperatures in the compound, Luther measured 1.68 kWh/kg reaching temperatures of up to 140°C [15]. The mixing process is thus limited by the maximum compound temperature. In many cases the mixing process needs to be carried out in several steps and sensitive chemicals are added only during the last mixing step at low rotor speeds and temperatures [13]. The compound must be cooled down to ambient temperature between each mixing step.

2.1.3.2 Minimum production amounts

To gain the same physical compound properties, i.e. mixing quality for each batch, the degree of filling in the internal mixer should always be the same. Therefore, reducing the batch weight if less rubber is required, is not possible.

2.1.3.3 Batch handling and discontinuous production cycle

The finished compound is discharged after mixing into a two roll mill or conical counter-rotating twin screw extruder which is placed underneath the internal mixer. In the case of a two roll mill the contact surface between the hot rubber and the water-cooled roll is quite large. Due to the temperature difference between cold water and the rubber, a cooling effect in the rubber is achieved. After the mill, the rubber is forwarded as a sheet or strip to the batch off cooling unit. Air ventilators are used in the batch off unit to further cool down the rubber to avoid scorching in the compound. Despite cooling this equipment also stacks the compound onto pallets for further logistics.
2.1.3.4 Energy peak loads

During mixing with an internal mixer the whole rubber batch is usually dropped instantaneously into the mixer. This causes a high energy peak at the beginning of the mixing process, due to the high rubber viscosity when feeding rubber at ambient temperature. Limper was able to show up to 500 kW for a 45l mixer running a NR compound. [16]. This energy peak leads to very high maximum power requirements. [13] These power requirements have to be provided at high costs.

2.1.4 Continuous mixing developments

Many modifications have been carried out to develop a continuous mixing process for rubber. In some cases the target has been to mix the complete compound ingredients in just one mixing step using a co-rotating twin screw extruder [17]. Others used a master batch rubber mixture and attempted to continuously add small amounts of oil or other compound ingredients using a single screw extruder [7].

In most cases, major advantages have been found for continuous mixing when compared with batch mixing of the rubber compounds [18].

The main advantages of continuous mixing compared with batch mixing can be summarised as follows:

a. No energy peak loads during mixing

Banbury mixers show an energy peak load at the beginning of the mixing process when feeding high amounts of rubber at ambient temperature. Due to the high rubber viscosity at this low temperature the energy consumption is comparably high at the beginning of the mixing process reaching up to some hundreds of kW. [16]
A continuously operated system featuring the same output may consume about the same amount of specific mixing energy per rubber mass but at a mean and constant level. Therefore less investment is required to install the power supply system for a continuous mixing process.

**b. Reduced overall energy consumption**

Assuming that the compound can be mixed in one step using a continuous mixing process, cooling between the steps is avoided by comparison with multiple step batch mixing. Consequently the total energy input is reduced in the case of continuous mixing. This is shown with the following sample calculation:

A given heat capacity $c_p$ [J/(kgK)] [19] of natural rubber equals 1800 J/kgK [20]. Assuming a realistic temperature difference of 80 K between infeed and output gives $1800 \times 80 = 144000$ J/kg = 0.04 kWh/kg energy saving.

Additionally, further energy savings can be achieved because the rubber does not need to be transported and stored between the process steps in the case of one step mixing. Finally, extruding the product into its final shape is possible in many cases saving further energy.

**c. Continuous process and combination of process steps**

The continuous mixing process using an extrusion system can combine further processing steps that may be required for production of the final product, such as vacuum, straining or preform production. These additional processing steps can be integrated in the overall extrusion layout and thus further simplify handling and logistics. Such combinations have been set-up with the Shark 70 demonstration machine used in this research.
d. No batch-to-batch variation

Continuous mixing systems greatly eliminate batch-to-batch variations, which are common when using Banbury mixers. The variations caused by Banbury mixers can be reduced by blending different batches and feeding them at the same time. [9]

e. Ability to produce variable amounts of compound / Variable batch size

The batch size in a conventional Banbury mixer is dependent on its internal volume, which has to be filled to ensure that the mixer functions properly. Running continuous production with a single compound at constant output is therefore the best solution for these set-ups. A continuously running system is by its nature much more flexible. It allows more rapid switching between smaller batches. Also, there is a minimum batch size defined by the internal volume of the mixing machine, but thereafter any desired quantity can be produced, depending only on the time and speed of the running system.

Two different technologies have been developed and put into service with variable success.

2.1.4.1 Twin screw extruder for continuous mixing

Twin screw extrusion is very common for mixing purposes, especially in the plastics industries and for manufacturing of plastic / rubber composites.

Two basic designs are available for twin screw extruders. One is the co-rotating twin screw system and the other one is the counter-rotating twin screw system as illustrated in Figure 2.
In addition to these design principles a counter-rotating twin screw extruder can feature tangential or intermeshing screws as shown in Figure 3.

As a result, the influence on mixing- and transportability is analogous to the principle in Banbury mixers where intermeshing and tangential rotors are also used. [3]

The intermeshing screw design is self-cleaning and allows the avoidance of dead spots and therefore the risk of material degradation due to uncontrolled residence time in the screw [1].

Contrary to this a tangential twin screw extruder is not self-cleaning [4] and therefore is usually not considered for mixing rubber compounds. Such a system is described as being able to mix gel-like fluids [22].

The operating principle of counter rotating intermeshing twin screw extruders based on chamber conveying is the same as for volumetric operated gear pumps [3].

By comparison, the conveying principle of co-rotating intermeshing twin screw extruders is based on drag flow as is also the case for single screw extruders. The flow
can be modelled analogous to that in single screw extruders. These machines are
designed for high screw speeds up to 1500rpm [3].

Co-rotating intermeshing twin screw extruders are able to produce highly homogeneous
mixtures and allow efficient degassing of the compound [1]. Furthermore, their screw
design is modular. Sections for mixing, transporting, shearing or degassing can be
combined on splined shafts according to the process requirements of each product [4].
This enables processing of different compounds by simply modifying the modular
screw design but at a cost to internal screw cooling.

However, co-rotating intermeshing twin screw extruders suffer from several
disadvantages in the mixing of rubber which limit their application in the rubber
industry.

One major disadvantage is the insufficient ability for pressure build up. This can be
attributed to the drag flow operating principle of the system [23] which is opposite to a
positive displacement pump. If high die pressures are required the corresponding energy
input into the rubber compound is very high because the screw has to run at a relatively
high rotational speed. Therefore the output is often limited, due to the maximum rubber
temperature allowed to avoid vulcanization in the extruder. [13] Typically, industrial
compounds should not be processed at temperatures above 120 °C.

In addition, the good mixing performance of these extruders is based on the ability of
the design to provide excellent shearing of the compound. However, especially in
combination with active fillers as part of the rubber compound, this high shear input
causes high temperatures. This is another factor that may reduce the output caused by
temperature limitations.
Another major disadvantage is that a twin screw extruder cannot be directly fed by rubber strips as is the case for single screw extruders. An approach to solving this problem was the development of the use of powder rubber that can be fed directly into the feed zone of the extruder [5]. Another alternative would be to feed the twin screw extruder with a extruder gear pump combination [24]. This allows the use of standard feed strips but requires considerably higher investment in machines.

However, powder rubber was not successfully implemented in the industry, due to the increased cost compared with bale rubber. A further solution is the use of a single screw extruder to side feed the twin screw. This solution had already been described by Skibba in his PhD thesis [5]. The only disadvantages are the additional cost for the side feed extrusion system and its power requirements. But it allows the use of feed strips instead of granulated rubber making handling easier and manufacturing of this feeding material cheaper.

### 2.1.4.2 Single screw extrusion

The fundamental principle of a single screw extruder is based on an Archimedean screw. The screw rotates inside a cylinder and transports a compound between its flights, normal to the centre line of the cylinder.

Rauwendaal described the common flat projection model for extrusion as shown in Figure 4. The screw and barrel are projected flat. The model describes shear stream in a rectangular channel. The cylinder is turning in a relative motion around the screw in this model and the channel height is much smaller than the screw diameter. The flat projected cylinder is seen as a plate, which is moving with speed “V” through defined angle “θ” relative to the channel direction [4].
The compound adheres to the wall of the cylinder and to the screw surface, the speed difference between the surfaces causing shear stress. This is transformed by internal friction of the compound into heat. Parallel pressure is built up along the extruder starting from the feed zone [26]. Higher temperatures lower the viscosity of the compound [4]. This again reduces the heat input. In the downstream zones of the screw, the compound gets thermally and physically homogenised [3]. This can be observed by comparing the compound extruded using two screw lengths. The strip from the shorter length will have a significantly larger difference between core- and surface temperature than a strip from the longer screw.

To influence the behaviour of the compound and to remove excessive heat from the system, a cooling medium, most often water, is used in different zones to control the temperature. This system also allows limited control over the viscosity of the compound [1].
2.1.5 State of the art of continuous plastic mixing – Comparison with plastic processing

The basic set-up of rubber and plastic extruders are similar, the basic technology is almost the same. Operating temperatures for plastics are in most cases significantly higher dependent on the melting point of the extrudate.

One of the biggest differences between rubber and plastic extruders is the drive power required as the torques necessary to process rubber compounds are much higher. To withstand these higher loads, parts must be more robust. Also it is one of the basic specifications for rubber extruders that they are always self-cleaning [4]. This is a result of the crosslinking nature of most rubber compounds that require a machine to empty completely when stopping for prolonged periods. Otherwise starting up may not be possible due to cross-linked rubber in the screw. This automatically rules out the use of most of the widely used mixing elements for plastic extrusion.

In the plastic industry single screw extruders are widely used for mixing applications. In these applications all components are mostly dosed into the feed hopper. [10]. The feeding material is in general free flowing or dosed with the aid of feeding systems like vibration devices to avoid clogging up. [12]

Despite the differences between plastics and rubber extrusion, some of the basic ideas have been transferred from the former to the latter as the basic mechanics of transport, flow and mixing are the same.

2.2 General machine set-up for mixing

The extrusion system used for the experimental part of this study is described here followed by an evaluation of the auxiliary equipment and the possible set-ups that can
influence the process. The compound and chemical system are discussed followed by the analysis of systems and procedure.

2.2.1 Single screw extruder gear pump system

Firstly, a description of an extruder gear pump system provides an overview of the technology employed followed by a description of the advantages that a gear pump adds in the continuous mixing of rubber.

2.2.1.1 Basic extruder layout

Single screw extruder gear pump systems are the basis for all the research in this thesis. Generally these machines are characterised by the screw diameter and specific pump volume. Figure 5 shows the set-up and principle of such a system and is reproduced from the Shark system sales publications distributed by VMI-AZ Extrusion. The machine basically consists of two independent units. The extruder carries out mastication (i.e. mixing). The machines are also equipped with mixing zones to improve the efficiency of the mixing. Either a multicut transfermix zone, a pin barrel, (short for a barrel equipped with pins which are sitting in slots in the flights of the screw) or both of these systems can be used. Section 2.2.2 describes this in more detail. The gear pump is fed by the extruder at a variable inlet pressure (p1). The gear pump builds up the required outlet pressure (p2) which is determined by the head and die shape in front of the extruder.
Figure 5: Extruder gear pump set-up (courtesy VMI-AZ Extrusion)

Figure 6 illustrates the design of a typical rubber compound gear pump and the flow of the compound through it.

Figure 6: Basic rubber gear pump layout [27]

2.2.1.2 Advantages of a single screw extruder gear pump system

A volumetric gear pump allows an efficient high-pressure build up with little energy input into the compound. This leads to a reduced energy input into the compound and low temperature increase compared to single screw extruders especially for p2 pressures exceeding 150 bar. [28]
The volumetric operating principle of the gear pump ensures a steady and constant output compared with a conventional extruder. Equi-sized volumes of material are transported along the gear pump housing and displaced into the outlet by meshing of mating gear teeth. Given the complete filling of the gear teeth cavities at the inlet, pressure fluctuations are thereby greatly reduced. [29]

These two characteristics are based on the principle of the positive displacement pump where flow is almost independent of pressure build up. The latter is provided through the torque of the drive system while the flow is dependent on the rotation speed. [30]

Results exhibiting operating consistency were achieved in trials by Köhler et al in 2004. They showed measured output pressure fluctuations by merely +/- 1 bar on a 150 mm extruder equipped with a gear pump. [31]

The output consistency provided by a gear pump is also clearly shown in Figure 7 and 8. In each, the blue line is the pressure plot of the extruder (p1) measured at the pump inlet, while the red dotted line is the p2 pressure measured at the outlet of the gear pump.
Pressure fluctuations at the pump inlet $P_1$ caused by screw flights and feed strip variations are greatly reduced at the outlet of the pump if the pump is always completely filled with rubber. This situation applies in both of the cases depicted in Figure 7 and Figure 8: Pressure fluctuation of a gear pump compared with an extruder, $p_1 < p_2$ and $p_1 > p_2$. 
Figure 8; when either $p_1$ is smaller than $p_2$ or in some mixing applications when $p_1$ is larger than $p_2$. These findings have not previously been published as it was not thought to be of interest to those using extruder gear pump systems. The aim during extrusion is normally to reduce the temperature instead of increasing it by adding more energy into the compound. All data provided in Figure 7 and Figure 8 was collected from tests on a 120mm extruder used in conjunction with a gear pump. The set point for $p_1$ was 25 bar for the first test and 70 bar for the second. A sampling interval of 1 second was used for the pressure data collection with a total of 145 samples obtained. The compound was EM06, a synthetic rubber tread compound supplied by the Kraiburg company.

For each compound, a minimum $p_1$ inlet pressure value is necessary for filling the gear pump completely [31]. Additionally, it may be advantageous to increase the $p_1$ pressure to achieve a higher energy input.

Figure 9 shows a cross sectional schematic of the extruder gear pump system and the positions of $p_1$ and $p_2$ pressure measurement. A typical pressure profile is also given in Figure 8. The inlet pressure ($p_1$) is adjustable by increasing or decreasing screw speed. Inlet pressures above minimum fill pressure, required to fill the gear pump, result in an increased energy input into the compound.
p1 is limited by the connection between extruder and gear pump. Usually this connection is limited to a maximum p1 of 350bar in accordance with internal design specifications stipulated by VMI.

Another limitation is the increasing rubber back flow which occurs in the screw when increasing p1. If p1 rises, rubber regurgitation into the feed zone will also increase. A high amount of regurgitation in the extruder feed section can cause variations in feeding or a miss feed and the process collapses. The maximum possible p1 pressure is different for each compound and mainly attributed to the viscosity of the compound.

The extruder drive energy is transferred via shear stress into the compound. An increased energy input (increased p1 pressure with faster screw speed) usually has a direct effect on the mixing quality. The inlet pressure (p1) is kept constant by a
proportional–integral–derivative controller (PID). This common type of feedback controller is widely used in the industry for such tasks.

When increasing the inlet pressure, the gear pump acts like a throttle so that the screw conveying efficiency is reduced and rubber backflow in the screw increases. The mean residence in the screw remains constant when increasing p1 but the residence time distribution in the screw increases, due to the increased rubber back flow.

Control of the total output of the extruder gear pump system is very important for characterising the continuous mixing process. This enables the system to be fed by regular feed strips without control of the input and without the necessity of steady feeding by rubber strips. Section 3.1.1 provides an overview of the technology necessary to determine this total input and Section 3.1.1 explains in more detail how the output of the gear pump is calculated.

2.2.2 Screw and barrel design for mixing

The design of the screw and barrel directly influence the possibility of creating shear, distributive and dispersive mixing, throughput, feeding behaviour and the self-cleaning properties of the extruder. Both the barrel and the screw design play a significant role in increasing or decreasing mixing efficiency, especially for different mixing zones. Lawal et al showed in experimental and theoretical studies a limited mixing ability in single screw extruders with simple screw design when no mechanisms for reorientation of the compound are present [32].

The aim of each extrusion process is to achieve a homogenously mixed compound at the outlet of the system. In most cases, especially for cold fed tyre compounds this
cannot be achieved if a standard conveying screw and barrel are solely used. This led in the nineteen-seventies to the development of pin and transfermix extruders. [33]

Pin barrel extruders are widely used in the rubber industry since their invention in the 1970s by Harms and Menges [34]. The mixing section is characterised by pins which are placed in several rows circumferentially and radially around the screw. The distance between the tip of the pin and the screw inner diameter is between approximately 0.2 and 0.5mm. For each pin row, the screw features a notch in the screw flight to allow injection of the pin into the screw turning area without damaging the screw. [3]

The arrangement of the pins in the barrel divides the material being transported in the rotary laminar flow along the screw flights [35] as shown in Figure 10.
Figure 10: Mixing improves as more pins interact with the compound (compound is extruded from left to right) [36]

The shear input of a pin section is relatively low avoiding great heat input. The notches in the screw flight are also found to have influence, increasing the mixing capabilities of this system [37]. Mixing in the pin barrel is quite efficient because of the frequent flow splitting and reorientation of the material. It undergoes flow diversion and shifting with only a low shear rate providing mixing and homogenising without excessive thermal overstressing. [35]

Improved heat transfer and relatively low shear rates keep the material temperature low. Nijman was also able to show that this lower temperature was accompanied by a higher output per screw revolution. [38] The pin barrel also allows a flexible set-up where the number of installed pins influence the homogenising character directly [34]. In his study, Camesasca compared a pin extruder to a barrier screw and a normal conveying...
screw and clearly showed the high mixing efficiency of the pin extruder based on Shannon entropy, wherein a rigorous measure of mixing, that has been widely applied across different disciplines, is used to quantify laminar mixing in an extruder and calculations of simulated two-colour mixing [39]. The homogenisation abilities of pin extruders vary considerably from compound to compound [34]. Pin zones provide elongation flow which is favourable for dispersive mixing and distribution [37].

Due to this effect and also the relatively high shear necessary for mixing chemicals into the compound, the pin extruder needs in some cases to be combined with an additional mixing element such as a transfermix section.

Transfermix extruders were suggested by Meyer as alternatives to pin extruders and other plastication set-ups employed to mix and homogenize compounds in the tyre industry [40].

Fischer showed if intensive homogenization and mastication was required, a transfermix extruder could be used in front of the gear pump. The transfermix extruder has become established as an effective mixing, homogenizing and plasticising system for rubber extrusion for a wide range of rubber compounds. This can be attributed to its unique barrel and screw configuration. Extruders of this type have a geometry which, by means of reducing and increasing flight depths in screw and barrel, transports the compound out of the rotating screw into the stationary barrel having concentric opposite-handed threads and back into the pressure build-up section of the screw [41]. The receding flights of the screw interact with the increasing channels of the barrel in such a manner that the extrudate is partially forced out of the screw into the barrel and hence sheared normal to the flow already transferred into the barrel [42]. Figure 11 represents a diagrammatic sketch of such a transfermix section.
Furthermore, due to their design, the compound is forced to delay and gets parted and reassembled into a new order. One screw flight is feeding more barrel flights at the same time. Furthermore the number of flights in the barrel can increase along its length. [43] Common designs have full flights which are running throughout the whole section and half flights which start at the middle of the section.

This means the stream is cut into small strips which are thereafter reassembled in a new configuration.

![Figure 11: Schematic multicut transfermix zone sketch [43]](image)

This can be further substantiated by consideration of a theoretical division factor $K$ that describes the number of theoretically possible divisions of material, making up a defined volume per kg of compound in the transfermix extruder [7]. The dividing factor $K$ is derived from the extruder throughput $\dot{m} = \frac{dm}{dt}$, screw speed $n$ and a Transfermix
specific constant $C$, in turn derived from the number of flow channels in the particular screw and barrel configuration of the Transfermix zone according to Equation (1)

$$K = \frac{C \times n}{\dot{m}} \left[ \frac{rpm \times h}{kg} \right]$$

(1) [44]

Where $C = machine\ constant$

$n = screw\ speed\ [rpm]$

$\dot{m} = throughput\ [kg/h]$

The factor $K$ defines the theoretical number of partial elements or partial volumes per kg of compound situated in the transfermix zone is divided into by shear activity when flowing through the zone. By increasing the screw speed $n$ whilst maintaining a constant throughput $\dot{m}$ it can thus be seen that the divisional factor increases and hence also the shear rate. This leads to improved mixing/homogenization in the extruder [44].

This configuration enables effective mixing of the compound in a very short section (approximately 2 L/D) of the extruder compared with conventional single screw extruders, allowing for a very compact overall L/D ratio of between 10 to 12. If the transfermix extruder is used in combination with a gear pump, the length of the extruder can be reduced to an L/D ratio of approximately 9 [44].

Another important feature which can be observed when running such extruders is the self-cleaning ability of this type of mixing zone for most compounds.

Pin and MCT extruders are the most commonly used. There are some special designs for niche purposes available. It is relatively difficult to make a general recommendation of which system to use. The best solution should be considered using information from
pre-tests which allow technical comparisons. Furthermore, it is essential to be guided by the available literature.

2.3 Mixing model used to set-up tests

Following the first trials and review of the results, additional research was undertaken into the flow behaviour described in Chapter 2.1.4.2. In particular, the flow behaviour of a single screw extruder and the possibilities of controlling mixing were reviewed. Based on the flow behaviour, different mixing concepts were compared. The aim was to reach effective mixing with the minimum energy input required to keep productivity high.

2.3.1 Categorising the different mixing types

In general three types of mixing are distinguished. Distributive mixing is mostly referred to mixing of compounds into each other [23]. The influence of shear is not required in all cases [45]. However, in general the extrusion process involves shear due to the transport mechanisms discussed in section 2.1.4.2.

Dispersive mixing is necessary to break up agglomerations of particles and to disperse them to the necessary degree in the master batch [37].

Laminar or shear mixing is the basic type of mixing resulting from the transport mechanism between the screw and barrel.

Camesasca explained that the two essential aspects of mixing in polymer processing can be differentiated into dispersive and distributive mixing. Dispersive mixing deals with the reduction in size of a cohesive minor component (agglomerates of solid particles or
droplets of an immiscible fluid), whereas distributive mixing accounts for the overall spatial distribution of the minor components in the polymer matrix. [39]

Figure 12 shows the difference between the different mixing types.

![Figure 12: Different mixing types [45]](image)

Rauwendaal defined the main aims of distributive mixing:

- The polymer should be subjected to significant shear strain
- The fluid should be split frequently with reorientation of the fluid elements

Conversely, he further defined the specification for dispersive mixing sections:

- The mixing section should have a high stress region where material is subjected to high stress
- This high stress should only occur for a short time.

All fluid elements have to experience the same high level of stress for the same time in order to reach uniform mixing [37].
All the mixing types are based on the flow dynamics in a single screw extruder. The following sections give a more detailed review of flow dynamics and how the materials get transferred into mixing zones and elements for continuous mixing.

2.3.2 Flow dynamics in the extruder for Newtonian and power law fluid models

Dick [36] plotted the flow of a particle in a single screw extruder. Figure 13 depicts a situation where particles can only move along the wall areas with considerable velocity. At about 1/3 of the channel height there is a point where particles have zero velocity. The particles of the compound are not moving linearly following the screw channel but in a spiral movement. This may include going in a reverse direction relative to the transport direction.
Figure 13: Flow in a single screw extruder [36]

According to Stevens et al [46] a description of the flow behaviour and resulting path of particles moving through the extruder is dependent on the output and the pressure gradient in the extruder as shown in Figure 14 and Figure 15. The velocity profiles depicted are a result of the combination of drag flow due to screw rotation and pressure flow caused by the p1 pressure built up in front of the extrusion die.
In the figures, $Q$ is the actual volumetric output, $Wbh$ is the theoretically possible volumetric output of the screw based on its geometry and the screw speed $W$. In reality
a ratio of $\frac{1}{3}$ for $\frac{Q}{Wbh}$ is currently considered to comply with good screw design. The relation $\frac{dp}{dz}$ shows the pressure gradient in the screw [46].

The first image “a” in the figures 14 and 15 show a situation where there is no output. The material is moved together with the moving wall of the barrel at 90° to the output flow direction. The backflow is in the lower third of the screw channel with lower speeds. In the next image “b” some flow is possible for a high extruder output pressure $p_1$.

Situation “d” in Figure 14 equals “c” in Figure 15 and complies with a situation where $p_1$ is almost at zero. No backflow occurs, but as previously mentioned this situation will not occur in reality.

Following the velocity profiles, shear rates in the lower part of the channel are negative while they are positive in the upper region. This means that the mixing occurring in the lower part is counteracting relative to the mixing in the upper part of the channel (Figure 16). This causes demixing effects in the outer recirculating flow [4].
Figure 16: Shearing in the outer and inner recirculating flow [37]

Thus different flow patterns can now be used to influence mixing in the machine. Meiertoberens et al used a throttle to influence the backflow in the machine [7]. This has the major disadvantage of reducing the output of the machine, or, alternatively, a die requiring a certain pressure set will not allow the machine to operate at pressures below this level.

An extruder gear pump combination allows control of the backflow by setting a controlled $p_1$ pressure in-between the extruder and gear pump. This pressure is completely independent of the die pressure. By increasing $p_1$, the backflow increases and a part of the material is forced to flow a greater distance through the screw and therefore acquires more shear energy. Also the mean residence time distribution widens leading to improved homogeneity while mean residence time remains constant.
Polymer melts typically exhibit behaviour that is strongly non-Newtonian, exhibiting shear-thinning and viscoelastic effects. [37]

In a Newtonian fluid, the viscosity (defined as the ratio of the stress to the velocity gradient of the fluid) is constant. In many practical situations the fluid in a single-screw extruder will exhibit non-Newtonian behaviour. Non-Newtonian fluids have a viscosity that is not constant, for example the viscosity can vary with shear, pressure or time. [47] Introducing these dependencies complicates the analysis and renders analytical treatments intractable, even with the assumption of isothermal flow. Because of the nonlinearity of the governing equation for non-Newtonian fluids, a simple superposition of drag and pressure flow is not a valid procedure. [37]

Such calculation have been published by Braun et al [25] and Buick. The latter calculated a comparison of velocity and shear for different power-law indices (n) simulating different compound behaviour. Newtonian fluid has an n value of 1 while typical polymers have indices n < 1 [47]. For greater clarity n = 0.5 and n = 1 are respectively marked in red and green in Figure 17.
Figure 17: The normalised x-component of the velocity and the normalised shear as a function of the normalised y-position [47]

Some conclusions can be drawn in respect of the influence of power law fluids on the models used here.

By calculating the flow velocity distribution in screw flights, Rauwendaal also showed that the shear rate in areas with small speed differences is almost zero as it depends on the speed difference in flow. As a result, no mixing will take place in these regions. [48]
When taking the power law fluid behaviour of the processed polymers into consideration, this effect is increased. The velocity profile for a Newtonian fluid is parabolic at \( n=1.0 \), but for smaller values of power law index, this parabolic curve becomes flatter for the mixing of rubber compounds. The low shear region expands and consequently the region exhibiting poor mixing is larger. [48]

As a result, the core flow experiences less shear and will stay at a lower temperature, resulting in temperature inhomogeneity in the flow channels [4].

This results in agglomerates in this area experiencing no external forces and remaining in an unchanged state. Rauwendaal stated that shear flow was not very efficient in achieving dispersive mixing because particles in the fluid were not only sheared but were also rotated (Refer to Figure 18).

In elongational flow, particles undergo a type of stretching deformation without any rotation; called “irrotational flow”, (Figure 18). Since there is no rotation in pure elongational flow, the deformation of the fluid is effectively transferred to clusters in the fluid. This results in tensile forces acting to pull apart the clusters into smaller clusters. [49]
This break-up of agglomerations can be approximated with another model used firstly to describe the breakup of drops in Newtonian fluids for liquid dispersive mixing. It also allows the determination of efficiency of mixing zones.

In a lot of cases for rubber compounds, dispersive mixing can be seen as a breakup of agglomerations or drops reducing their size and distributing them in the surrounding matrix. Conversely, distributive mixing may require some breakup or splitting of the material added to the mix in order to produce the desired particle size from the original supply form [4].

Grace [50] carried out a very extensive study on the breakup behaviour of drops in liquids. Observing the breakup behaviour of liquids mixed together in a Couette device and a four-roll device. The coquette device consists of two concentric cylinders turning.
in opposite directions (contra-rotating). The observed liquid is sheared in the space between the cylinder walls.

*Figure 19* Couette device as described by Grace [50]

The results allowed him to determine the critical Weber $E_B$ number for breakup in relation to the viscosity ratio for the two mixed fluids. (Refer to Figure 20).

*Figure 20: Critical Capillary (Weber) number versus the viscosity ratio by Grace [50]*

Depending on the viscosity ratio, a minimum Weber number is necessary to break up drops in the matrix. He also found that this critical capillary number $E_B$ was lower in elongation flow than in shear flow [50].
\[ E_B = \frac{G_B r_d \mu_c f(p)}{\gamma} \]  

(2) [50]

\[ G_B = \text{shear rate} \left[ \frac{1}{s} \right] \]
\[ r_d = \text{drop radius} \left[ m \right] \]
\[ \mu_c = \text{continuous phase viscosity} \left[ Pa \ s \right] \]
\[ f(p) = \text{function over viscosity ratio} \ p = \frac{\mu_D}{\mu_c} \ f(p) = \frac{19p + 16}{16p + 16} \]
\[ \gamma = \text{interfacial tension} \left[ \frac{N}{m} \right] \]

Although these measurements were made with Newtonian fluids, the results can be used as a basis for categorising and explaining mixing behaviour in a single screw extruder. This also allows the right set-up of different mixing equipment to be chosen in line with the selection previously explained.

In Newtonian fluids the Weber number is commonly used to analyse fluid flows interfacing two different fluids. The main difference in polymer mixing applications is that in a non-Newtonian fluid, the Weber number changes during the mixing process. The shear rate \( G_B \) is dependent on the actual viscosity. If the drop radius \( r_d \) is reduced, a minimum drop radius will be reached at which mixing will end. Also the viscosity relationship between the two mixed compounds can change during the process.

Rauwendaal explained that different studies have shown that the critical Weber numbers did not change significantly when viscoelasticity was introduced. The effect of viscoelasticity will mainly induce rapid deformations in short time scales [4].
Based on this consideration, Figure 21 shows the four basic situations occurring in mixing in an extruder.

\[ \text{Figure 21: Critical Capillary (Weber) number versus viscosity ratio for different mixing situations [50]} \]

Situation A represents the common shear flow in an extruder screw flight. Shear mixing allows for certain agglomerates or liquid drops to break up and get mixed in. It also shows that shear flow is not suitable for mixing if the viscosity relation of the two compounds is above 3.5 when the critical Weber number tends to infinity as in situation
B [4]. Hence, highly viscous parts cannot be dissolved in compounds of low viscosity. Drops or agglomerates are likely to rotate in the flow instead of breaking up as shown previously in the upper image of Figure 18. If the same mixture was to be run on a mixing device able to provide elongation flow, it can be mixed as shown in situation C. Agglomerates or drops would get stretched until they break apart [4]. This also allows the mixing of materials with higher viscosity ratios in elongational fields when shear flow is no longer able to provide sufficient mixing.

Situation D depicts when either the radius of the drops results in a lower Weber number (the shear rate is insufficient) or the viscosity of the compound is altered due to the pressure and temperature in the process. In this situation, no mixing is possible.

Comparing situations A and C, if all the variables in the Weber number are fixed except the shear rate, it is observed that elongation flow is also a more efficient way of mixing. Elongation flow requires smaller shear rates compared with shear flow, resulting in lower energy input. Translated into a real extrusion system, this allows running at the same compound temperature for a higher output.

Consequently, an appraisal of the models suggests elongation flow is the most desirable form of flow. There are different ways of achieving elongation flow. One of the most common solutions is the pin barrel extruder, referred to in section 2.2.2. Harms et al first described the device in 1974. The pins that protrude from the barrel into the inside of the rotating screw flight allow the flow to diverge and remix but also force the material to flow around the pins causing elongational flow. [51] Depending on the number of pins and the length of the screw there is the possibility that some particles could pass through the pin zone without experiencing very much elongation flow.
In order to achieve homogeneous mixing, all particles should be forced to pass the same mixing zone at least once under comparable conditions. Such a situation can be reached by using a barrier flight extruder screw invented in 1959 by Mailleffer. This development represented a great step forward in extruder technology. All barrier screws have two channels in their barrel section. A secondary flight is usually situated at the beginning of the transition zone. This creates two different channels. One is filled by the still solid and unplasticised material. By reducing the channel crosscut area in this way, the melted material is forced into the other channel. This increases in volume as shown in Figure 22. Beyond the Maillefer section all the material is plasticised [12].

Figure 22: Maillefer screw design [4]

Rauwendaal states that compared with other mixing zones there are no significant advantages from using barrier screws. In some conditions unplasticised material building up in the wrong flight can even result in their blockage [4].

The design was used in rubber extrusion and allowed more uniform extrusion but only when used with relatively high temperatures. [34] This influence can be limited by lowering the barrier. This leads to a reduced homogeneity, so here a balance has to be found for each process. The best results are gained if the screw is used with low pressure p1 at the screw tip. [38]

Rauwendaal has suggested a so called “CRD” mixer to achieve optimised elongation flow for continuous mixing in single screw extruders. In the proposed mixer, the flights
are lower and have a tapered edge and improved land. A wedge channel between barrel and flight allows the compound to enter and experience higher shear, but elongation flow occurs at the same time as shown in Figure 18.

The flights are cut at regular distances to build a throttle providing only elongation flow between the walls that are fixed relative to each other. The mixer is recommended for use in all type of polymer compounds and especially for mixing colour granules into the main compound. [52]. For self-cleaning in rubber applications, one of the screw flights retains its original configuration (without the wedge) and at the same height as the conveying flights. [53]

Another approach to improving mixing capability was suggested by Skibba for improving the shear and elongation flow in the transfermix section described previously. The conventional transfermix has very narrow gaps between the screw and barrel flights, cutting the compound and rearranging the layers of the compound. (Refer to Figure 11). Compared with the configuration in Figure 11, Skibba advocated that the gap between the flights should be widened. The flight was widened and, relative to its counter flight, had a tapered profile. The resulting wedge shaped gap allowed a much higher amount of compound to enter the area between the flights resulting in a high shear and elongation flow. As the complete volume of the screw is transferred into the barrel, the core stream of the flow is broken up and the temperature homogeneity is improved. In Figure 23 this concept is shown in a crosscut through the centre of the section showing a four-flighted screw and an eight-flighted barrel. The round edges and the angled land forming the wedge profile are also clearly shown. [54] Lawar et al found the main mixing in single screw extruders happened in the nip region between barrel and screw flight [32]. However in a conventional screw only a small amount of
the compound can pass through this small gap due to small tolerances. The widened gap in this design allows most of the material to pass.

Another advantage of this design is its flexibility. The barrel section is usually a short insert allowing it to be changed simply. Different designs with varying numbers of flights can be used based on pretesting results.

This design has been introduced for some machines and will be the subject of further research.

As stated earlier, the energy input influences mixing quality. There is a temperature limit for the compound restricting this input to avoid the commencement of crosslinking. An improved heat transfer, reducing the temperature would allow more mixing energy input. Heat transfer from the compound to the wall is limited so the only possibility to attain this is to increase the surface in contact with the compound. A
possible technology allowing this could be planetary roller mixing sections as shown in Figure 24.

Figure 24: Planetary roller section used as mixing element. [4]

These sections use a centre gear-screw. In gear terminology this is referred to as a sun wheel and is part of the full screw mechanism that includes rotating planetary screws turning in mesh with an annular gear screw. This is in principle a combination of a single screw extruder and a multiple screw extruder. There are several advantages arising from this configuration. Firstly no pressure loss occurs in the planetary roller mixing zone; in fact it is even capable of building up pressure. The main advantage of the set-up is very good heat transfer properties that are enhanced by a factor of 2 to 3 compared with a standard extruder. This enables the systems to attain good dispersive and distributive mixing. The systems are self-cleaning. Also, depending on the design, the following disadvantage has to be considered. A consequence of the set-up is that back mixing in front of the planetary zone is set to a fixed process value irrespective of the machine control. Such systems are commonly used for Polyvinyl chloride (PVC) extrusion but also for compounding of Thermoplastic elastomers (TPE). [37]

A flexible design layout enables the fitting of the machine design to the process requirements for different mixing tasks or compounds. [55].
Such a system is not only able to mix but, depending on its design and position in the machine, also to build up pressure. This planetary gear pump can be used instead of a conventional gear pump [56]. Research carried out on planetary roller extruders by Luther et al showed the ability of this equipment to offer good heat transfer characteristics. The equipment used also showed good mixing results, however direct comparisons cannot be made as their research covered the full mixing process [15].
Chapter 3

Materials and methods

3.1 Process window for the continuous mixing system

Designing a machine for continuous mixing requires the set-up of some envelope parameters before testing can be undertaken. The possibility of controlling the overall output of the machine by using a gear pump is critical to the process. Further, the design of the screw and barrel determines the basic mixing ability of the machine. This ability can be influenced during the process by varying the inlet pressure $p_1$ of the gear pump. Finally, process temperature settings for the different zones allows further tuning of the overall process.

3.1.1 Output controlled by the pump

One of the main advantages of the continuous mixing system described here is the possibility of feeding standard strips which are widely used in industry. These strips vary in width and thickness and are pulled into the machine by the use of a feed roll that is either driven by the screw or independently by a separate drive.

The input value for the control system necessary to calculate the amount of base compound pulled into the machine is the actual throughput of the gear pump. This volume minus the volume dosed by the dosing systems equals the input volume, which is the basis for all calculations carried out in respect of machine control. This continuous mixing system requires a pump having a high efficiency over a wide range of pressures.
To show the constant output accuracy of a gear pump, a simple test was set-up on a 70 mm laboratory extruder gear pump system equipped with an Extrex 70-5 RV pump. While running the pump at constant speed, samples at 36 second intervals were cut out of the extruded strip at the die. The weight of each sample was recorded and is shown in Table 1. This was repeated for 3 different speeds. The samples had to be cut by hand introducing a small variation of ±0.5 seconds which is also shown. Taking this variation into consideration the output is still seen to be constant to within ± 0.5 % of the mean for the worst case.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Speed #1</th>
<th>Speed #2</th>
<th>Speed #3</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>12 rpm</td>
<td>16</td>
<td>37 rpm</td>
</tr>
</tbody>
</table>

| Mean output [kg/h] | 23.173 | 29.576 | 71.081 |

| Variation output 0.5 s [kg/h] | 0.322 | 0.411 | 0.987 |

| Calculated output [kg/h] | 23.5 | 30.2 | 72.7 |

*Table 1: Sample weight and mean output gear of pump*

As previously mentioned, the overall output of the machine is one of the input values of the machine control system. To be able to calculate the output \( \dot{m} \) of the pump, the following gear pump output Equation (3) can be used.

\[
Q = \alpha \cdot N - Q_l \left[ \frac{kg}{h} \right]
\]  

(3) [57]

where
\( \alpha = \text{volumetric displacement per revolution} \hspace{1mm} [\text{kg/rpm}] \)

\( N = \text{frequency of rotation} \hspace{1mm} [\text{rpm}] \)

\( Q_l = \text{leakage flow} \hspace{1mm} \left[ \frac{\text{kg}}{\text{h}} \right] \)

If typical input values are used as in eqn (4)

\[
\dot{m} = \frac{V_{\text{spec}} \cdot Sg \cdot \text{rpm} \cdot \eta \cdot 60}{1000} \left[ \frac{\text{kg}}{\text{h}} \right]
\]  

(4)

where

\( \dot{m} = \text{output per time} \hspace{1mm} \left[ \frac{\text{kg}}{\text{h}} \right] \)

\( V_{\text{spec}} = \text{specific gear pump volume per revolution} \hspace{1mm} \left[ \frac{\text{cm}^3}{\text{rev}} \right] \)

\( Sg = \text{compound specific gravity} \hspace{1mm} \left[ \frac{\text{g}}{\text{cm}^3} \right] \)

\( \text{rpm} = \text{operational speed of the gear pump} \hspace{1mm} [\text{rpm}] \)

\( \eta = \text{gear pump conveying efficiency (usually between 90 and 96\%)} \)

\( 60 \text{ and } 1000 \text{ are the necessary factors to achieve outcomes in kg/h} \)

Then, given the specific volume of the gear pump at 176 ccm/rev [58], the theoretical output of the machine can be calculated. The results are shown in Table 1.

The theoretically determined value of pump output is higher than the real output. This is as a result of the leakage flow \( Q_l \) in the pump. The relation is expressed as the pump efficiency in Equation (5):
\[ \varepsilon_v = 1 - \frac{Q_l}{\alpha v N} \] (5) [57]

The leakage flow is mainly dependent on differential pressure \(\Delta p\) across the pump and viscosity of the pumped medium [57].

It can be split up in three main flows; leakage over the tips of the gears, through the bearings and through all other mechanical unavoidable gaps. Experimental research and calculations by Limper et al resulted in efficiency reductions between 2% and 10% depending on counter pressure and rotation speed. The results of their research are shown in Figure 25. The pump they used is the same type as used for the trials made on the Shark 70 system described in this thesis [28].

![Graph showing gear pump leakage volume streams at different set-ups](image)

*Figure 25: Gear pump leakage volume streams at different set-ups [28]*

This study also claims a positive transporting effect between the tips of the gear pump and the housing caused by drag flow bigger than the pressure flow in the opposite
direction. This effect is influenced by the gap between the moving surfaces and therefore also dependent on mechanical wear of the gear pump parts [28].

The difference between theoretical and real output is also shown in Table 1. This difference has to be taken in consideration to correct the calculation on output and further data processing for the machine. Due to the dependence on viscosity and wear, the efficiency should be monitored on a regular basis when operating a machine. To include decreasing pump efficiency with increasing differential pressure, the control system of the machine included a differential pressure dependent efficiency factor.

3.1.2 P1 window

P1 can be set within certain limits as described in Section 2.2.1.2, in a window between minimum pressure for filling the gear pump and the greatest pressure to maximise the mixing effect. The mixing energy gets converted into heat and is limited by the maximum temperature allowed for the compound. This window differs for different compounds, but can be used to control mixing in the process.

As an example of how mixing can be influenced by p1 pressure, Figure 26 shows a black EPDM compound having light yellow granules mixed into the EPDM base rubber. This trial was carried out at the facility of Excel Polymers in the United States in August 2010. The company supplied the machine, compound and chemicals for this trial. Other than p1 pressure, all other process variables were kept constant during the whole trial. The machine was not equipped with pins nor was it equipped with a mixing zone in the screw. The chemical granules were dosed into the main feed hopper by a vibratory loss in weight feeder providing constant feed during the test. Cutting longitudinally through the centre of the strip exiting the machine allows a clear visual
inspection of the distributive mixing. As long as traces of the constitutive chemicals are visible, it can be assumed that mixing is inadequate.

![Image showing granules with red arrows indicating unmixed granules]

Figure 26: $p_1$ variation and mixing effect; the red arrows point to unmixed granules

When running the machine with a $p_1$ pressure of 20 bar, the granules were almost unchanged in size or form after extrusion. By raising the $p_1$ pressure in steps of 15 bar, fewer granules were traceable and shear could be observed in the stretched shape of the chemical granules. At 65 bar $p_1$ pressure, no traces of yellow granules were visible and it can be assumed that they were completely mixed into the rubber compound matrix. The temperature, measured at the die with a penetration probe, remained at 10 K below the maximum temperature that the specification allowed, leaving some latitude for using a higher $p_1$ if required.

Adjusting the $p_1$ inlet pressure of the gear pump offers a process window that can be used to optimize mixing efficiency for each individual compound.
A further parameter influencing mixing efficiency of the extruder is the design of the mixing zone. Based on trials with the compound to be used later in production on a test (laboratory based) machine, the type and shape of the mixing zone can be determined.

### 3.1.3 Process temperature settings

A further opportunity to improve the mixing process can be achieved via the temperature control system of the machine. Temperature control units are connected to different zones and parts of the machine in order to keep them at pre-defined temperatures. Changing settings will allow the viscosity of the compound to be influenced locally. [1]

Zones that can be subject to re-setting are: screw, feed zone, barrel, gear pump and extrusion head. Influence on the flow behaviour is indirect. By changing the temperature in a zone, only a thin cross-sectional area of the compound is influenced directly due to the low heat transfer in the compound. The viscosity of a typical rubber compound is dependent on temperature which means that flow behaviour will alter as a result of changing temperature. A hot surface leads to a “film” with relatively low viscosity, enabling the material in the channel to glide as it achieves relatively low shear in the main flow. A cold surface has the opposite effect; the shear rate is increased. So in general, a cold barrel in combination with a relatively hot screw increases pumping efficiency, while the inverse situation decreases it. For detailed set-ups of the different zones, reference to relevant literature such as Stevens and Covas should be made, as a full evaluation of the large number of possible variations here would introduce a large amount of information that is of limited relevance to the research described. [46]
3.2 Dosing systems for chemicals

Dosing of chemicals into a continuous mixer requires equipment which is capable of supplying the necessary amount of chemicals continuously with the required accuracy. Depending on the delivery form of such chemicals (powder; polymer pellets or liquid) there are different feeding systems which are already used in research and in the plastics industry. The systems described in this section act as loss-in-weight feeders measuring the weight loss continuously during the production process. Knowing the weight loss is for example necessary to provide inputs to a control system for setting the speed or frequency of a screw feeder [59]. The significant advantage of such systems is that they are easy to calibrate and can be adapted for a wide variation of different physical properties of dosed materials. Section 3.5 gives a full explanation of the materials. In this work, the costs of the systems are not compared; the focus is only on their process advantages and disadvantages. Nevertheless, the different feeder systems feeding in the main hopper described in the following Section are considerably less expensive as dosing extruders are more complex systems.

3.2.1 Single screw loss-in-weight-feeder

These dosing units are based on the principle of a single Archimedean screw used to transport the material. A silo contains the material and feeds it via an inverted cone base into the screw. Due to the rotating screw, transportation of the chemicals is provided into an outlet barrel. The screw speed is the controlled variable in order to set-up the dosed amount.

Dosing units used for this research are provided by the German manufacturer Brabender Technologie. These units are equipped with flexible walls to avoid the build-up of
bridges with powder or any other blockage that might occur above the dosing screw. Furthermore, different screw designs are available to suit different materials such as powder or granules. An example of such a unit is shown in Figure 27.

Figure 27: Brabender flexwall single screw feeder [59]

The exchangeable screw and barrel of the single screw LIW feeder allow for easy set-up of different materials to be dosed if necessary. Additionally, the unit features a very compact footprint, 500 by 500 mm and 1000 mm high, and all parts that may require service can be reached from the outside. [59]

3.2.2 Twin screw loss-in-weight-feeder

Twin-screw feeders work on the same basic principle as that described for single screw feeders. Two rotating inter meshing or counter meshing screws are responsible for the discharge of the material to be fed. In particular, powders which are not easy to transport and powders behaving like fluids during transportation can be handled with
this type of feeder. Another advantage is their ability to dose very small amounts of powder down to small values (dm$^3$/h) [59].

### 3.2.3 Belt feeder

This type of dosing equipment uses a conveyer that is placed underneath the silo to transport and dose material. Limited by the height of the opening in the silo, the amount of material transported is already fairly consistent at a constant belt speed. By controlling the speed of the conveyer belt, the dosing rate is set and also maintained stable in respect of small speed changes. This general design is simple and robust. During transportation of the material, no shear is induced, which can be considered an advantage for shear sensitive materials [59].

Usually, belt feeders feature a bigger footprint by comparison with other units. Furthermore, increased effort is required to clean them and they are not ideal feeds for powder due to the design of the belt and belt drive.

### 3.2.4 Vibratory feeders

Another type of dosing unit uses a U-shaped rail, which vibrates to transport the material. The transportation rate can be set depending on the frequency of vibration. This technology avoids shearing of the material that can occur with the other three systems previously described. These units work by vibration alone and consist of no rotating parts. When switching between different material types such as powder and granules of different sizes, no parts need to be exchanged.

These feeders are especially used for dosing free flowing bulk materials like granules or small grained materials.
During trials carried out in this research, a negative effect occurred with powder feeding using vibration feeders. The powder formed agglomerates and consequently irregular feeding resulted. As a result, the accuracy was variable and the trials had to be aborted. After switching to granules the agglomeration effect was no longer present.

### 3.3 Gear pump systems for volumetric dosing

As an alternative to weight scale feeding systems, chemicals or compounds can be dosed into the main extruder by using volumetric operating gear pump systems. These systems are often used for liquids or highly viscous compounds. In this case, pressure is required to transfer the material into the main machine and gear pump systems have been found to be effective for this purpose. An example of a gear pump system from VMI-AZ is shown in Figure 28. The Planetruder design is based on patent DE 130 48 985, utilising a planetary gear pump driven directly by the extruder screw [60]. To increase the sealing length and improve the transported volume per revolution an enhanced version with two parallel stages is suggested in another patent, EP1801418A1. [56]
Figure 28: Planetary pump principle set-up with sun wheel (12,58) and planetary wheels (20-24) [56]

The volume of the gear pump is fixed and its pump efficiency can be measured simply by measuring the output at defined set points of counter pressure. This data allows a plot of the efficiency to be extrapolated over the whole envelope for which the unit is used. An example is shown in Figure 29.

Figure 29: Gear pump efficiency plot [27]
3.4 Basic machine system for using continuous mixing

Based on transfermix single screw technology used in conjunction with a gear pump, two prototype machines for final mixing were designed and manufactured at VMI-AZ Extrusion. The process schematic of these machines is depicted in Figure 30. One machine consists of a 70mm screw diameter while the other machine features a screw diameter of 120mm.

Both machines are equipped with a loss-in-weight (LIW) screw feeder type DDW40plus made by the Brabender company, Germany. The basic design of the LIW feeder chosen allows precise feeding of chemicals, powder and granules. The loss-in-weight-feeder is placed above the feed hopper so that the chemicals are able to fall directly into the extruder feed opening. Feeding of the rubber master batch is also provided through the extruder feed hopper using rubber strips (Figure 30).

*Figure 30: Continuous mixing with transfermix single screw extruder and gear pump (courtesy VMI-AZ Extrusion)*

Both test machines are equipped with optional pin zones as shown in Figure 31. The screw/barrel/pin combination gives great flexibility due to very good mixing and dispersive actions.
Furthermore, both machines are equipped with the option of using different strainer meshes and are able to cut preforms using a servo driven cutting knife. The extrusion head can be assembled with different die geometries.

Figure 32 depicts the 70 mm machine used for most of the experimentation described in this text. This machine can deliver a maximum output of 350 kg/h and is considered by VMI to be the smallest size that would be used in an industrial environment.

The whole machine is set-up quite flexibly with an exchangeable transfermix barrel allowing the use of different mixing zones. Other parts of the equipment, for example the screw, can also be replaced with minimal effort.
The complete system, including the dosing unit, is controlled via the man-machine interface (MMI) employing a touch screen. Additional to the process data, which is shown in real time on the MMI, all data can also be read and logged in real-time by a standard laptop via Ethernet. The data is stored as a “.csv”-file allowing its processing with common software such as Microsoft Excel.
To analyse the vulcanisation characteristics of processed compounds, a Rheometer type MDR 2000 manufactured by MonTech in Germany was used. The Vulcameter will be described more in detail in Section 3.6.2.

3.5 Compound and chemical systems

This research concentrates on VMI’s approach to continuous final mixing. Most of the compound ingredients are already in the base compound and only the crosslinking system is mixed continuously as a final processing step to finish mixing. Hence, the following description concentrates on chemicals and the master batch necessary for final mixing.

3.5.1 Rubber in sheets / strips

The rubber master batch is supplied as feed strips or sheets, depending on the machine size. The master batch is mixed in a conventional first step on a Banbury mixer. Without the presence of a curing agent, the master batch is less sensitive to high mixing temperatures and does not have to be transported under air conditioning. Also the set-up of the machines with self-feeding of strips allows the feeding of multiple strips simultaneously to blend different charges, leading to reduced batch to batch variation. Handling of the feed strips or sheets is also not an issue as this is the standard procedure in each rubber processing company. Pallets and other supply chain equipment for transporting and storing rubber compounds also remain unchanged.

3.5.2 Curing agents

Curing agents are necessary to transform the rubber compound from viscoelastic to elastic after it is formed into the shape of the final product. This is done by cross-linking
the molecular polymer chains with curing agents. The trials for this research were conducted with peroxide and sulphur linked compounds.

### 3.5.3 Sulphur

Sulphur has been used since the very beginning of commercial rubber manufacture and is especially used for natural rubber as the common curing agent. The sulphur reacts together with the polymer to build mono, di- and polysulfide structures. The specific fraction of sulphur required varies depending on curing time and temperature. The resulting rubber matrix has good mechanical properties, although resistance to high temperatures is limited, as the rubber will ultimately be subject to thermal degradation [1]. Sulphur is either available as powder or bound with a polymer in granular form. With Ethylene-Propylene-Diene-Monomer rubber (EPDM), Figure 33, it is used most often as a binder. Storage and handling in the machine is simple and harmless provided personal safety equipment is used.

![EPDM structure](image)

*Figure 33: EPDM structure[1]*

### 3.5.4 Peroxide

Peroxide is used as a curing agent particularly for EPDM compounds, but also for other compounds required for products to be used in higher temperature environments. Compounds cross-linked with Peroxide usually have inferior mechanical properties including lower fatigue resistance. [1]
Peroxide is sometimes available as powder, but most often is delivered in the form of small pressed pallets. Due to its chemical characteristics, it has to be handled with care to avoid fire risks during processing in machines. It should not be stored in bright sunlight or at high temperature. It might also cause skin irritations and personal safety equipment must be used. For the trials in this research PEROXAN BIB-40 MB supplied by Pergan GmbH was used [61]. Its structure is shown in Figure 34.

![Figure 34 Structure formula CAS RN 25155-25-3](image)

**3.5.5 Powder or granules**

There are basically two delivery conditions for these curing agents. The simplest form of supply is powder. Powders are available almost everywhere and from numerous suppliers (e.g. Rhein Chemie), and are the cheapest form of these chemicals. Also millroom departments are used to handling different powders. However, especially in rubber processing companies, there are some disadvantages if the machine is set-up in a normal production line. Using and handling these powders requires specific preparations and special equipment is necessary. Workers have to be protected due to health risks when handling these potentially hazardous chemicals; explosion risk has to be minimised by employing special equipment and set-up of the machines and the powder must be contained in the entirety of the equipment handling it to avoid spreading powder throughout the building.
To simplify handling, eliminate the explosion risk and have the possibility of combining some premixed chemicals, a polymer is used for binding. The resulting granules can be simply processed with dosing units. Often different colours are used for easy determination of the chemicals. Another important advantage is the ability to combine a set of different chemicals in one granule. This allows a custom made system for each different compound that a customer may wish to process.

In the manufacturing process, polymer and chemicals are mixed together. As a result the chemicals are already pre-dispersed in the polymer matrix. This avoids agglomerations of powder and supports the distribution and mixing in the extruder.

Different granules can be easily premixed with a simple concrete mixer, especially when ingredients would already react together while being transported and stored. This is avoided by being separated into different granules that are premixed just before use. A picture of premixed granules is presented in Figure 35. Using such premixes for final mixing provided acceptable results during the trials. The compound reached the same required quality in respect of its properties compared with the traditional process. Typically, the granules are of a cylindrical shape with diameters between 6 and 8mm and lengths ranging from 4 to 8mm.
Variation of the binder allows the melting point and the Mooney viscosity to be set, influencing the material behaviour during the mixing process. Also, the handling characteristics outside of the machine are important to avoid jamming and to allow a good flow and transportation in the dosing unit without deformation.

### 3.6 Analysis systems and methods for ensuring compound quality

Like most extruders used for production, a single screw mixing extruder requires some process quality control to ensure compound quality and this is especially so for larger machines to avoid the production of large amounts of scrap. Unfortunately online systems still have limitations in what can actually be measured, whereas offline systems to monitor quality are quite common and for some tests are the only available option.

#### 3.6.1 Offline analysis

Offline analysis systems and procedures are not an integrated part of the production line and are unable to process and show quality measuring data in real-time, though they
may have only a small time delay which allows the use of this data to influence the control of the machine.

3.6.1.1 Dimension and weight checks

Depending on the product, there are different systems used in front of the die and extruder to check the product dimensions. This only makes sense for final products or as a backup system for measuring the output. The output can be calculated together with the pull-off speed and the specific gravity. However, due to die swell and (in this case) elastic behaviour, the measurement has limited accuracy and therefore is only useful for checking the abrasion and wear of the gear pump.

Meter weight scales are also used in the industry but they are mainly to oversee equipment necessary for extruders running without gear pumps.

3.6.1.2 Visual inspection

A first impression of mixing quality can be obtained from a visual inspection. This is only possible when the two mixed materials are different to each other either by colour or reflectivity or in different phases. For example a white powder will be visible in the surrounding black compound while black powder in the same coloured compound is hardly noticeable.

In the first case, quality can be assumed to be good if powder is no longer visible. This would provide fairly good visual evidence.

In the second case, the only features that can be observed are agglomerations which are characterised by a considerable amount of powder at one spot. The feedback gives only an imprecise measure of quality.
But even with no visible trace of the chemical additive, the results in many cases are insufficient until further analyses such as those from rheometer tests show the same result.

Figure 26 shows such a situation. If two colours are mixed together, this effect can be used to provide evidence on mixing quality and flow behaviour. Chapter 5.1.4 details the results of such a test with results based on visual inspection of the extruded strip.

This method allows a very rapid determination of the mixing quality. The distribution of the mixed compound is possible on very small scales. The visual check is either limited by the resolution of the eye and/or the vision aids available.

In contrast, samples tested in for example a vulcameter require a minimum volume of about 5 cm$^3$ and the results are therefore always quoted for a mean value of this volume. Referring again to section 5.1.4, the inhomogeneities would be too small to be detected in a rheometer on most occasions.

### 3.6.2 Rheometer

Two different rheometers, one Vulcameter and one Mooney Viscosimeter were used in this research.

#### 3.6.2.1 Vulcameter

The vulcameter is one of the most common and important devices for testing physical properties of the final mixed compound. The measuring principle is standardised in ISO 6502, ASTM D5289 and DIN 53529.

Operation:
A rubber sample is placed into a chamber, between a top and bottom plate. The rubber sample has a defined volume. The top plate in the chamber is oscillated by a motor through small angles while a torque sensor is connected to the bottom plate. The bottom plate is also able to turn, being driven by the oscillations transferred through the compound. The whole chamber is heated to the required vulcanisation temperature. The transfer of frequency and torque is plotted and defined points are measured. The graph and the data allow characterisation of the compound. Figure 36 shows the rheometer used during the research.

Figure 36: MDR 2000 Vulcameter die configuration [63]

For a final mixed compound including curing chemicals, its elastic part increases while the viscous element tends to zero with increasing test time. If the amounts of curing chemicals are incorrectly added or they are not well distributed, the value reached at the end of testing will be different. If final mixing is carried out successfully, each compound complies with a characteristic graph and each sample in the Rheometer can be compared to a master graph.[64]
Figure 37 shows the set-up conditions for the Rheometer test (e.g. temperature and time). The table beneath the graph represents the date and measured data as previously described.

Where:

- $S$ is the torque measured between the two rotors
- $S'$ is the first derivative of $S$ and represents the elastic part of the compound
- $S''$ is the second derivative of $S$ and represents the viscous behaviour of the compound
3.6.2.2 Mooney Viscometer

The behaviour of different extrudates can be described by their Mooney values. Using a Mooney Viscometer is quick and simple; a sample is placed in a chamber which has an internal rotor. Following a defined preheating phase, the rotor starts to turn slowly at 2 rpm. The torque is measured and quoted in Mooney Units (MU). A graph depicting MU versus testing time is plotted [65].

There are two rotors available, L for “large” with a 38.1 mm diameter as standard, S for “small” with a 30.48 mm diameter for higher viscosity compounds that have reached the maximum torque on the L rotor during testing. 100 MU is defined as equal to a torque of 8.3 Nm. [1].

The measurement following the basic set-up is defined by the following code: ML(1+4) 100°C, where:

- Rotor
- Preheating time
- Testing time
- Testing temperature

This definition allows a comparison of different measurements. [66]

3.6.3 Chemical analysis

In most cases a chemical inspection was not possible at the site of the research. Only a minority of VMI’s customers were equipped with an adequate laboratory to enable such research. Also for confidentiality, most customers did not share their full results as they
would reveal too much information about their compound recipes. Therefore the following trials do not refer to chemical analysis results.

3.6.4 General remarks in respect of the quality of data

The tests in this research have mostly been carried out in industrial environments. This has some influence on the way trials are performed and the resulting quality of the data. However, these industrial environments created the opportunity for trials that were much closer to real production when compared with a laboratory set-up.

Also the investments in expensive equipment and the sheer size of this equipment would be prohibitive for laboratory trials.

However, some points should not be overlooked; there are a number of uncertainties in the process that have to be borne in mind during extrusion tests.

To minimize the risk of measurement errors it was necessary to carry out calibration checks on machines and equipment before the tests. This included speed checks on main motors and other sensor equipment. Most of the equipment was not continually monitored and especially demonstration machines tended to get adapted to new set-ups when required for demonstrations.

Many of these trials could not have been undertaken without the support of additional people working on and around the machine. The support was necessary to cope with the output, sorting and storing of samples and also on feeding the machine with the compound and refilling dosing units. While everyone normally carried out these tasks with great care, sometimes unforeseen mistakes happened and in very rare cases these were not detected before the trial were completed.
An example of such operating errors is the measurement of output temperature by hand which can give rise to different readings when different people undertake the same operation with the same equipment. This was observed several times during numerous trials.

This necessitated a final careful check of all results and datasheets before accepting data for analysis.

An indirect influence was the set-up of the machines for trials. While in most cases the aim was to adapt the machine optimally for the research task, in some cases a different approach had to be used. Hence, in each case it was not possible to adapt an existing machine that had already been sold to a VMI customer to the exact task and consequently the machine had to be used in a semi-optimised state.

3.6.5 Manual data logging

Data logging is the collection of data during an extrusion test. There are different ways of doing this, often depending on the test conditions and the experiment.

Most tests in this research were made on demonstration machines in the VMI workshop while some tests had to be run at VMI customer facilities.

In each case settings, results and set-points compared with actual values had to be recorded. The normal method employed was to write the data during the trial directly into a spreadsheet. Care had to be taken in order to input data to the correct column and avoid any transposed digits as discussed in previous sections. Most data was displayed on the MMI of the machine; some additional data had to be measured by hand during the run (e.g. compound temperature using a thermometer, output rates).
Weight samples cut by hand from the extrudate are usually measured at 36 second intervals. This allows an easy conversation by dividing the measured weight in grams by 10 providing the output in kilograms per hour as shown in Equation (6)

\[
\dot{m} \left[ \frac{kg}{h} \right] = \frac{1000g}{3600s} = \frac{10g}{36s}
\]  

(6)

The cut was normally made directly at the die to maintain a constant position and the time was recorded by a stopwatch. To reduce errors, numerous samples were taken for each datapoint reducing the individual error in time-sampling and cutting. By keeping the procedure the same for all measurements the errors are maintained at a constant level.

### 3.6.6 Automatic data logging

For some of the trials conducted during this research it was possible to connect a Laptop via an interface to the control system of a machine. This allowed reading and storing the process data available in the CPU to be automated. Nevertheless some data had to be written down manually as either there was no sensor in the machine or no connection to external modules. This occurred particularly in experimental set-ups configured for a small number of tests.

The big advantage of automated data logging was that sampling speeds were only limited by cycle times of the CPU. A second advantage was that all data was unquestionably recorded and no fields were overlooked.

Normally the logger was set to record all available data so it allowed the later consideration of data initially not regarded as interesting.
The superior resolution allowed control system responses to disturbances to be reviewed in order to optimise controllers in the machine control system. It also allowed tracing of long-term effects such as temperature changes in waveform caused by two point controllers in TCUs.

3.6.7 Online systems

Running production in the traditional way, using two-stage Banbury mixers, does not allow complete online quality control. There is enough time to process samples taken directly after mixing before the compound is actually cooled down and fed to the extrusion lines.

Nevertheless a so-called fingerprint of the mixing process is recorded. It includes the motor power, temperature and position of the ram. Additionally, the integrated motor power giving the total energy input is normally included. [67]

However, switching to continuous mixing creates the necessity to know the quality of the product immediately at the machine. This can be illustrated by the following example; running a 150 mm extruder at 1500 kg/h with an estimated 15 minutes for the rheometer check to run before there is an outcome, would create 375 kg of scrap material.

3.6.7.1 Rheinchemie online system

The German company Rheinchemie is one of the key suppliers for tailor made chemical systems and is currently developing an online quality screening system. This system is currently only presented as a black box to the user and the precise measuring and sensor equipment is confidential. Nevertheless, it has already been used in some trials and showed its potential for determining the concentration of chemicals in the rubber mix.
Furthermore, small chemical concentration variations appear to be visible in the data produced by this online quality control system. To date, this system has not been released. In the future it will potentially offer a very interesting way of providing the continuous mixer with online quality feedback and thus may influence process parameters. As soon as this system is released it has the potential to be a very helpful tool in the industrialisation of the continuous final mixing process.

### 3.7 Sample production

Samples for inspection of the mixing quality were made in two ways in this research.

In most cases strips were cut in front of the die and marked with a test number or a time stamp to connect them to the settings for each test point. The samples were then collected and inspected and compared at the conclusion of the test. Thereafter, some were stored to provide the possibility of comparing them with samples from future set-ups.

Alternatively, a rotating knife cutter was used which was installed on some of the test machines. In this situation samples with equal volumes could be collected. This was primarily used when mixing in metal flakes as described in section 5.1.5.2.

### 3.8 Experimentation set up

The experimentation has two main focal points. The first set of experiments focuses on dispersive sulphur mixing while the second set concentrates on distributive mixing.

Dispersive mixing started with powder feeding on a 120mm extruder gear pump system and additional follow up trials set on a smaller 70mm system. Comparing experiments using pre-dispersed chemicals in granule form followed.
The second focal point was centred on distributive mixing. Starting with two different coloured compounds, a visual inspection of mixing results allowed comparison of influencing factors based on literature and models discussed in Section 2 with experimental results. P1 variations, dosing methods and elongation mixing were tested on a 70 mm extruder gear pump combination.

While two colour testing is quite common to visualize mixing, a novel approach was made by mixing brass flakes into the compound. Firstly again a 70mm extruder gear pump was set up in a laboratory environment for the production of sample compounds. A visual inspection allowed a first examination before a second round of experiments was conducted on a 90 mm extruder gear pump system in an industrial environment allowing validation of mixing on an industrial scale. Instead of visual inspection, the density of the final mixed compound was measured to allow the determination of the mixing quality.
Chapter 4

Results and discussion – Dispersive sulphur mixing

The theories described in previous chapters have outlined the differences between distributive and dispersive mixing.

The following ‘results and discussion’ is therefore split in two parts to reflect each form of mixing. Firstly dispersive mixing of powder sulphur will be reviewed. The sulphur was mixed in the main compound as a curing agent and therefore had to be dispersed into the polymer matrix to allow the crosslinking reaction in the final compound. To provide consistent material properties, after dispersion the chemicals needed to be well distributed in the rubber matrix.

4.1 Powder feeding

Sulphur is available in two forms allowing mixing into the masterbatch. The most simple is powder, which is the cheapest solution but by its nature introduces some handling issues.

Several trials were carried out running master batch compounds together with curing chemicals in powder form. This was achieved with differing results as explained in the following sections.

4.1.1 120 mm systems

A sulphur-based curing agent and its base compound were mixed in combination with a vibratory feeder at the plant of Excel Polymers in Jonesborough, Tennessee. Excel polymers is a leading global supplier of performance elastomer solutions, offering a comprehensive portfolio of compounded polymer materials, performance additives and
value added services to rubber parts manufacturers. It is headquartered in Solon, Ohio, USA and employs approximately 800 people in the US, Mexico, UK and China [68].

The aim of the test was to observe the mixing of a quantity of about 4% of curing agent into the master batch.

The machine used for the trials was a standard VMI Shark 120 extruder-gear pump combination equipped with a transfermix zone and a conveying screw without a mixing zone. From the onset of the trial, the vibratory feeder did not perform as intended. The powder tended to stick to the vibrating rail instead of being transported. Large sections of powder agglomerates broke off. This resulted in inconsistent dosing of the chemicals. Changing the feeder parameter settings could not solve this problem.

Consequently, the decision was taken to change the feeding unit and use a single screw loss in weight feeder. With this change of unit, a precise feed rate could be achieved for the chemicals.

When running the system within the temperature limit of the compound it was not possible to attain the necessary mixing effect in the screw. Rubber samples taken by cutting along the extrudate centreline still showed spots of undispersed curing chemicals.

Additionally, the powder chemicals covered all surfaces of the screw and the feed roll. This reduced the friction drastically and the rubber feed strips regularly started to break, particularly when p1 was set at higher levels to achieve more mixing. An increase of p1 inlet pressure setpoint resulted from the absence of the transfermix component in the screw.
Another serious issue was the previously mentioned disadvantage of feeding powder. In Figure 38 dust is observed to have covered almost every part of the machine, but is especially noticeable under the feed roll. This can obviously not be tolerated in normal production environments due to economic, health, safety and environmental considerations.

From various cases of oral poisoning with Sulphur, it was determined that doses of 15 g upwards for an adult human can cause toxic effects (nausea, vomiting, diarrhoea, abdominal pain and headache, vertigo, depression and collapse). Lower doses (< 1 g) mostly have caused only diarrhoea (to a degree dependent on predisposition) [69].

Figure 38: Sulphur dust covering the machine and its environment after running for about one hour

To prevent this dust spreading above the feed hopper a large vacuum device had already been installed. Nevertheless a visible dust layer built up in about one hour when the
machine was running in this configuration. Traces of dust could be found all around the vicinity of where the machine was set-up.

In consequence, the trials with powder chemicals were stopped and polymer-bound chemicals were used for further testing. The available granules contained chemicals but no curing agents so they had no influence on the vulcanisation process. Therefore only visual inspections by cutting the extruded strip were used to give feedback on the mixing process. As shown in Figure 26, traces of granules could be observed by eye up to a certain pressure. Above 60 bar $p_1$ pressure, no traces could be observed in cuts of the extrusion strip. The mixing ability of the machine was demonstrated in this way though no more trials could be carried out on the machine. The results gave a rudimentary indication of the direction the research should follow, but the analysis was unsatisfactory and required improvement for further trials.

4.1.2 Powder mixing with 70 mm Shark system

Based on the results from the 120 mm extruder, a comparable set-up was evaluated on the 70 mm laboratory extruder gear pump combination.

As a result of the previous powder mixing tests, the feeding was accomplished with a single screw-loss-in-weight feeder. The feeding consistency improved as it had before with the 120 mm screw. However, again a complete dispersion of all the powder was not possible; the temperature limit of the compound was reached before complete dispersion was achieved.

4.1.3 Mixing quality distributive mixing – Results

The desired quality of continuous mixing could not be reached with the dispersive mixing setups that have been described. Accompanying issues with powder
contaminating the environment, including health risks for the operators led subsequently to no longer focusing on dispersive mixing and alternatives were sought.
Chapter 5

Results and discussion – Distributive mixing

The main focus of this research centred on distributive mixing of crosslinking systems, filler and colours into the main compound. First trials with polymer bound predisposed granules were conducted and energy consumption of the continuous process measured. To prepare and accompany the trials with brass flakes, additional yellow – red coloured compound mixing was conducted. The colour mixing allowed a review of the models previously discussed at a good resolution since small colour differences indicate fine mixing quality. This also helped in the checking of the performance of a partially new machine design in respect of mixing brass flakes.

5.1 Granule feeding

The following sections show results of mixing trials made using granules containing the curing agent bound in rubber.

5.1.1 70 mm Shark system

Dosing chemicals in granular form immediately proved some of the advantages to be gained from continuous mixing. However, handling in the LIW feeder and behaviour of the machine caused some unexpected machine stops at the very beginning of the tests. As with peroxide, some polymer bound sulphur granules showed a tendency to melt in the extruder feed zone. Compared with the peroxide, the percentage dosage of the total output was higher because of the binder. Usually the chemical concentration in the polymer bound granules is between 60 to 80% pre-dispersed in the polymer binder.
In order to allow dispersion at low energy input, the melting point was set to a low level. The comparably high quantity of chemicals being fed in with the mix melted in the extruder feed zone and acted as a lubricant. This effect caused breakage of the master batch feed strip.

The low viscosity also caused locking up of the LIW dosing system when an accumulation of granules became blocked in the small conveying screw and in the funnel guiding the material to the screw. After increasing the viscosity and using an improved conveying screw, these two issues were resolved.

5.1.2 NBR compound experiments

Another common approach to visualize flow and mixing capability in extruders is to use two coloured compounds. In this research a green based compound and additionally a yellow compound bound sulphur were chosen. The base compound was a nitrile butadiene rubber (NBR) supplied and used by ContiTech Northeim to manufacture printing blankets. Its density is 1,2 g/ml and the viscosity is 60 Mooney (ML1+4 100°C). The homogeneity of the cross cut section provided a visual indication of the mixing quality.

To first compare the results for the current process, samples of the same compound mixed on an internal mixer were tested with the vulcameter set at 190 °C and for three minutes testing time. The results are shown in Figure 39.
The machine control was set to the parameters given in Table 2 during the trials. The machine was started and filled with parameter set #1 until pressure $p_1$ was reached at 100 bar. After this, conditions as per test #2 were used with $p_1$ set at 30 bar. At this point $p_2$ displayed 0 bar because the die was not mounted at the outlet of the gear pump.
<table>
<thead>
<tr>
<th>Test</th>
<th>Compound</th>
<th>Time</th>
<th>Current Pump</th>
<th>Current Screw</th>
<th>Speed Pump</th>
<th>Speed Screw</th>
<th>Pressure p1</th>
<th>Pressure p2</th>
<th>Output</th>
</tr>
</thead>
<tbody>
<tr>
<td>#1</td>
<td>C_NBR</td>
<td>X</td>
<td>4</td>
<td>15</td>
<td>100</td>
<td>no die (0)</td>
<td>62</td>
<td></td>
<td></td>
</tr>
<tr>
<td>#2</td>
<td>C_NBR</td>
<td>X</td>
<td>4</td>
<td>12</td>
<td>30</td>
<td>no die (0)</td>
<td>62</td>
<td></td>
<td></td>
</tr>
<tr>
<td>#3</td>
<td>C_NBR</td>
<td>X</td>
<td>21</td>
<td>69</td>
<td>4</td>
<td>9,8</td>
<td>20</td>
<td>167</td>
<td>62</td>
</tr>
</tbody>
</table>

Table 2: Test parameters for NBR tests

As soon as the machine was filled with rubber it was paused to close the head at the pump outlet. The machine was now started in automatic mode with the parameters shown in #3. Firstly, a 2100 mm strip was extruded with parameter set #2 without the extrusion head in front of the gear pump.

Figure 40 shows the first 1700 mm of extrudate, test setup #2. The surface displayed ribs caused by emptying the cavities of the gears when running with p2 at zero pressure. At 150 mm, the colour of the compound changed from yellow to green, indicating that the yellow cleaning compound was replaced by the green compound in the machine. Together with the first green compound, the first chemicals arrived as the dosing started; as soon as the extruder caught the green strip. The colour change itself lasted approximately 150 mm and from this point on the colour remained constant.
The sample is shown in greater detail in Figure 41. It also shows crosscut sections taken at 100 mm intervals to check the mixing quality visually and provide samples for the vulcameter. These cross cuts which are shown in Figure 42 did not reveal any visual traces of the granules or the chemicals; there were no layers or colour spots. The entire surface of each appeared to be homogeneous exhibiting a consistent green colour.
There were some yellow traces visible along the surface of the extruded strip. They were as a result of some yellow compound trapped on the side of the flow channel in the gear pump and so did not influence the results.

Subsequent crosscuts are not depicted but they were identical to those shown.
Figure 43: Vulcameter graphs of the trials without a die attached, setup #2

Although there was no visible trace of the chemicals, which shows good distributive mixing, there was still some variation in the dispersive mixing. Figure 43 shows the vulcameter curves of all samples coded with the position at which they were taken out.
of the strip and displayed in different colours. Clearly, there is a strong variation between the curves.

To visualize the change relative to the position at which each sample was taken out of the strip, a three-dimensional graph, Figure 44, was produced.

![Vulcameter graphs sample #1-#15 for the trials without a die, setup #2](image)

The Z-axis has its origin at ‘1’ on the right, up to sample ‘15’ on the left. The X-axis depicts the measuring time from 0 to 3 minutes. The temperature for all the tests was 190°C. The torque derivative $S'$ is shown on the Y-axis.

$S'_{min}$ remained constant indicating that there was a constant base compound viscosity. $S'_{max}$ and the line between it and $S'_{min}$ start from a low value due to there being insufficient curing agent dosed for the first three samples. The graph shows a rise in $S'$
with $S'_{max}$ almost reaching the reference value 15 in the “900 mm sample”. The value of $S'_{max}$ fell away beyond this point and did not recover. This instability during the machine start-up obviously lasted longer than expected, not only initially when the screw was incompletely filled, but also on the first occasion that pressure $p1$ was built up in front of the gear pump.

Similar results were achieved in the second trial with the die mounted. Again samples were cut out of the strip, this time every 250 mm from a starting point of 100 mm, as shown in Figure 45. No traces of chemicals were visible on the cut surfaces, showing again good distributive mixing. Also no inclusions or flaws appeared and there were no other signs of insufficient distributive mixing on the whole surface of the strip.

![Figure 45: Start-up strip 3600 mm Shark70 with die, setup #3](image)

The Vulcameter graph is shown in Figure 46 indicating some variation but also some matching results with the reference samples shown in Figure 39. $S'_{min}$ is reasonably constant, again indicating a stable base compound viscosity.
Figure 46: Vulcameter graphs for trials with the die attached, setup #3

To visualise the change of S’max in Figure 47, all graphs are shown in parallel in the order of extrusion. Results were quite similar to those in the first trials without the die attached and the first samples did not match the reference sample. Starting at 2300 mm, the 3D graph shows less variation from sample 9 (2350 mm) indicating stabilisation in the mixing process and also showing good dispersive mixing.
Figure 47: Vulcameter graphs shown in parallel for trials conducted with the die attached, setup #3

The samples depicted from 2350 mm to 3600 mm in Figure 48 show good mixing results. It can also be seen in this figure that, compared with the reference samples, the continuously mixed samples displayed slightly higher (shifted parallel) values.
This indicates that the dose of chemicals was slightly too high in proportion to the base compound. This was not caused by the dosing equipment itself as it was run within very close tolerances to the set point. In Table 2 the pressure $p_2$ was recorded at 167 bar and this pressure caused a low leakage flow between the wall of the pump and the rotors in the gear pump, thereby reducing the efficiency. This difference between the theoretical...
pump volume per revolution used in the control software of the machine and the real output volume may have led to overdosing.

5.1.3 Energy savings offered by continuous mixing

Earlier in this work, the possibilities of energy saving by running continuous final mixing were discussed. Most of the potential for energy savings is based on different handling or simply different process set-ups. To compare the actual energy consumption of continuous final mixing with internal mixing, all the essential data was recorded for trials carried out in July 2011. The test compound was an NBR master batch mixed with sulphur based granules. The density of this compound was 1.2 kg/m³ and the viscosity around 60 MU. The mixing was achieved on a 120 mm extruder gear pump combination equipped with a transfer mixing zone.

To compare the energy input of different processes, the specific energy input in kWh/kg was used. This amount of energy per unit weight can be calculated for most processes by comparing the energy input into the system with the mass per time in the system. Refer to Equation (7).

\[ SME = \frac{P}{\dot{m}} \left[ \frac{kWh}{kg} \right] \]  \hspace{1cm} (7) [33]

During his research, Bogun calculated a value of 0.22 kWh/kg for a compound that was similar to the test compound. This was based on mixing tests he made using an internal mixer [6].

Luther et al published data based on a silica compound mixed on a Banbury mixer. Calculating the SME for the final mixing stage with only curing agent mixed in the compounds gave 0.42 kWh/kg [15].
16 data sets for different output settings were collected during the tests on the 120 mm extruder gear pump system. These included the energy consumption of the screw and pump drive. The data were each divided by the mass flow which was also measured, leading to the specific mechanical energy values shown in Figure 49.

**Figure 49: Specific mechanical energy input for continuous mixing**

The main component for specific energy was transferred via the screw into the compound. This energy is necessary for plasticization and mixing. The energy for the gear pump is primarily necessary for building up the pressure before the strainer mesh and the die of the machine.

Averaging the 16 data samples gave a specific mechanical energy input of 0.08 kWh/kg. This is approximately one third of the energy Bogun calculated in his research [6] and one fifth of the value that Luther [15] showed. The mixing quality cannot be compared directly but both trials delivered good mixing quality.
A comparison with measurements made by Nijman showed that these figures are in a realistic range for a single screw extruder [38].

These data also show the necessity to design the mixing elements so that they are as effective as possible, or to be able to cool the compound efficiently. Cross-linked compounds are all characterised by a temperature limit. Mixing based on all forms of shear will result in temperature increase of the compound. Based on the model of Grace, elongation mixing should be preferential, as indicated by the lower necessary critical Weber number over a wider field of viscosity ratios, as it allows mixing with less energy input.

Based on Equation (7) the maximum energy input is reached when the largest possible delta T of the compound is set in the following equation in terms of $Q_{comp \, max}$ (8):

$$SME_{max} = \frac{Q_{comp \, max} - Q_{cooling}}{\dot{m}} \cdot \frac{1}{1000 \times 3600 \, s} \left[ \frac{\text{kWh}}{\text{kg}} \right]$$

(8)

$Q_{comp \, max}$ is therefore a compound dependent constant. A linear connection between $SME_{max}$ and $Q_{cooling}$ is solely dependent on the heat dissipating properties of the extruder.

The heat dissipation ability of a single screw extruder is limited by the residence time of the compound and the low heat transfer in the compound itself. Only layers directly in contact with the surface of the barrel or screw are able to dissipate heat.

Alternatively, the heat history of the compound is changed. Data from Luther indicates better properties in some fields of the final compound mixed with reduced heat [15].
5.1.4 Two colour mixing—yellow and red

In order to compare the theoretical models reviewed earlier, following the first mixing trials and actual mixing results, a series of tests was set-up with a yellow EPDM base compound and a red compound (each defined in Table 3) mixed into it. This setup allowed visual checking of the mixing quality and a relative comparison of the results.

<table>
<thead>
<tr>
<th>Colour</th>
<th>Supplier</th>
<th>Supplier compound name</th>
<th>Viscosity [MU]</th>
<th>Base compound</th>
<th>Delivery form</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yellow</td>
<td>Bekina</td>
<td>VMI yellow</td>
<td>80</td>
<td>EPDM</td>
<td>Strip</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>ML 100 1+4</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1,7 g/ml</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>70x8 mm</td>
</tr>
<tr>
<td>Red</td>
<td>Rhein</td>
<td>Fe-Red -70</td>
<td>55-65</td>
<td>EPDM</td>
<td>Granule</td>
</tr>
<tr>
<td></td>
<td>Chemie</td>
<td></td>
<td></td>
<td></td>
<td>ML 50 1+4</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2 g/ml</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>6mm dia x 5,5 mm long</td>
</tr>
</tbody>
</table>

Table 3: Compound specification for two colour mixing trials

This test was conducted on the VMI Shark 70 demonstration machine; the screw diameter was 70 mm and the pump an Extrex 70-5 RV with a volume of 176 cm³/rev. The barrel was equipped with a full set of pins and a multicut transfermix zone. This zone is positioned about 1 l/d in front of the gear pump inlet. Herringbone gears, emptying to the centre, were installed in the gear pump. The basic layout is shown in Figure 50; the actual machine is shown without side feeding extruder in Figure 32.
Figure 50: Layout Shark 70 demo machine with side feeding and LIW options (picture curtsey VMI-AZ extrusion)

The red compound was fed into the machine by a second extruder gear pump combination. This extruder was a 30 mm planetary extruder where the pump was driven directly by the screw. It was able to feed into the main barrel via one of the pin holes. For the trials it was connected to the second pin row in the extrusion direction.

As an alternative a loss in weight feeder FW40 from Brabender was installed on the machine above the main feed hopper to allow the dosing of granules directly into the feed zone.

5.1.4.1 p1 variation influence – backmixing

The first sets of test was run on June 26th 2012 with the objective of firstly determining the influence of backmixing on this machine set-up and, in a second set-up, the influence on mixing quality of the two different dosing systems.
The output was set at 74 kg/h with the main compound fed through the feed hopper. For the first setting, 5% red compound was side fed into the main machine via a Planetruder S. p1 was set to two different settings, 20 and 60 bar. Further data of the trials is shown in Table 4.

<table>
<thead>
<tr>
<th></th>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>#1.1</td>
<td>19.8</td>
<td>PP S</td>
<td>65.8</td>
<td>21.0</td>
<td>5.7</td>
<td>17.9</td>
<td>16.4</td>
</tr>
<tr>
<td>#1.2</td>
<td>60.3</td>
<td>PP S</td>
<td>65.7</td>
<td>20.6</td>
<td>5.7</td>
<td>18.2</td>
<td>18.5</td>
</tr>
</tbody>
</table>

*Table 4: Yellow - red mixing set-up for p1 variation*

The machine was started on the set-ups and allowed to run for a short period of time to allow the process to stabilize. When stable, samples were cut out of the extruded strip and marked to connect them to the recorded machine data.

The cross cut area was examined visually for uniformity and traces of unmixed material. Typically an insufficient mixing quality produces concentric layers around the centre of the flow. The gears in the pump produce a shape in form of an 8 around the centre. If the cross-cut surface has a uniform colour and all shades of the original colours are no longer traceable, good mixing is attained.

To improve the contrast for the images in Figure 47 they were converted to black and white and the contrast setting heightened. Otherwise the colour shade variation would not be easily seen. The flow and therefore the results are very symmetric for a cross-
section allowing the samples to be cut in half to be set opposite each other for comparing.

The sample in Figure 51 on the right side is the sample with the low inlet pressure $p_1$ set to 20 bar.

![Comparison of samples with different inlet pressures](image)

**Figure 51:** Sample #1.1 and #1.2, black/white image yellow/red mixing compared for two $p_1$ settings, no strainer plate

Different shades were in evidence particularly around the circumferential centre layers. Light grey areas depict the more yellow areas; the darker spots depict a more red compound. Increasing the inlet pressure $p_1$ to 60 bar gave the result shown in Figure 51
on the left side. The entire crosscut area was consistently shaded indicating good distributive mixing.

Some small marks remained only around the centre indicating incomplete mixing of the material. This was displaced material that remained for long periods in the tip of the double herringbone gears. Experience gained throughout the research substantiated the long running time needed to clean these gears when changing compounds or particularly changing colours.

It is clearly shown that increasing p1 pressure improved the mixing result. It was also shown that the shear caused by the transport mechanics in the screw was insufficient to allow mixing. Also the installed multicut transfermix section was unable to provide sufficient distributive mixing at these settings. Increasing the p1 pressure forced a part of the compound to remain for longer in the screw, widening the residence time distribution and thus helping to improve mixing.

**5.1.4.2 Influence of the dosing methods**

The second part of this test set-up focused on a comparison of the influence of the dosing methods on mixing process and quality. Therefore the same settings were run as in the previous trial while i) dosing the second material via the side feeder and ii) for the second set-up, using a loss in weight feeder above the feed hopper.

Adding two more set-ups with the LIW dosing system gave the outcomes shown in Table 5.
Table 5: Yellow - red mixing set-up for dosing system variation

As before samples were cut out of the extruded strips and the cross cut areas visually inspected for uniformity.

The first setting at 20 bar for p1 revealed a significant difference in mixing quality. The side feeding samples were at the low quality level already shown; the loss in weight feeder samples showed an increase in the quality of mixing but still insufficient mixing resulted as shown in Figure 52. There were circumferential layers visible around the centre of both samples and the colour differences were higher for the side feeding samples.
Figure 52: Samples #1.1 and #1.3 yellow/red mixing at $p_1 = 20$ bar, comparison between side feeding and dosing into the main hopper, no strainer plate.

Raising the inlet pressure $p_1$ to 60 bar improved the mixing quality for both compounds as shown in Figure 53. The mixing quality results are reversed by comparison with the previous set.
Figure 53: Samples #1.2 and #1.4, yellow/red mixing at p1 60 bar, comparison between side feeding and dosing into the main hopper, no strainer plate

The quality of the side fed samples is sufficient as previously demonstrated while the loss in weight dosing samples still showed the circumferential layers around the centre.

Considering these results in conjunction with the theoretical models lead to the following conclusions:

For the situation of dosing with the LIW feeder, the whole length of the extruder actively mixed the added compound into the main matrix. The cumulative maximum shear stress of the machine possible was applied to both the main compound and dosing chemicals. The residence time was maximised due to the dosing position.
Because of the machine design, the dosing port for the side feeder was significantly further forward at about 1/3 of the screw length. Residence time in the main machine was reduced and the cumulative shear caused less mixing.

In both cases the low inlet pressure $p_1$ of 20 bar was sufficient to fill the cavities of the gear pump but did not cause backflow in the screw. The multicut transfer mix section rearranged the layers of the compound but was not able to provide a distribution on the small scale necessary for the two coloured compounds to blend into each other.

At a higher inlet pressure $p_1$ the quality of the side feeding mix was improved. In Figure 53 circumferential layers can still be seen on the right side. As discussed following the first trials, most mixing takes place in the front of the screw where backflow leads to a widened residence time distribution and results in better distribution of the red particles. Backflow caused by the increased $p_1$ pressure widened the residence time distribution and forced material to pass the multicut transfer mix zone more than once.

This improved the mixing to create the necessary fine distribution of the two compounds to reach a uniform mixture. These results are in compliance with the predictions gained from the theories about shear stress and backflow mixing.

### 5.1.4.3 Elongation mixing trials

To determine the influence of elongation mixing, the same machine set-up from the above two trials as used on 23rd October 2012 was employed again. The main and additional compounds were also unaltered. For dosing the side feeder was used.
### Table 6: No strainer / strainer mixing set-up

A strainer plate is normally used in order to hold mesh layers that are supposed to filter solid objects from the compound. To support the meshes, the strainer plate is a solid steel plate with as much of its surface removed as is practicable by drilling multiple holes into it. The remaining structure has to be sufficient to support the mesh and the resulting forces. In most cases it is placed in front of the gear pump.

The strainer plate and its holes should act as a form of static elongational mixer. The compound is parted and forced through the holes as shown in Figure 54.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>#2.1</td>
<td>20.7</td>
<td>PP S</td>
<td>93.9</td>
<td>21.5</td>
<td>9.7</td>
<td>19.6</td>
<td>29.4</td>
</tr>
<tr>
<td>#2.2</td>
<td>19.7</td>
<td>PP S</td>
<td>153.5</td>
<td>22.5</td>
<td>9.7</td>
<td>19.4</td>
<td>30.1</td>
</tr>
</tbody>
</table>
While a calculation of the exact flow profile was not possible without the help of numerical solutions, the mean elongation $\varepsilon$ can be calculated according to the following Equation (9)

$$\varepsilon = \frac{\Delta l}{l_0}$$  \hspace{1cm} (9) \hspace{1cm} [70]$$

Assuming the compound is incompressible, volume $V$ of the material under observation is constant [37]. In this situation, the change of diameter between the flow channel and the multiple holes of the strainer plate force the material to elongate. The flow channel is a single cylinder with a radius of $r_1$, the strainer plate consists of $x$ holes of radius $r_2$ mm. Therefore:

$$V = const = r_1^2 \pi l_0 = x \cdot r_2^2 \pi (l_0 + \Delta l)$$  \hspace{1cm} (10)$$

(10) which if solved for $\Delta l$ gives:
\[ \Delta l = \frac{r_1^2}{x r_2^2} l - l \]  

(11) and if substituted in (9) delivers the equation:

\[ \varepsilon = \frac{r_1^2}{x r_2^2} - 1 \]  

(12)

Introducing the values \( r_1 = 60 \), \( r_2 = 4.5 \) and \( x = 80 \) [71] provides the mean elongation \( \varepsilon = 1.22 \).

Figure 55 and Figure 56 show the influence of using a strainer plate between the gear pump and die. The first shows that the basic set-up provided insufficient mixing.

Figure 55: Sample #2.1, black/white image yellow/red mixing at \( p_1 = 20 \) bar, no strainer plate

Without changing the machine settings a strainer plate was installed and the process was re-run. The result is shown in Figure 56; where the mixing quality is seen to be significantly improved.
Small circular regions with lighter shading were still visible but the overall uniformity of the sample was greatly increased. Also the mixing quality was better with no traces of completely unmixed colours evident. The pattern of the strainer plate was visible, showing the traces of elongation flow.

At the point of reaching the strainer plate, the flow channel area was reduced to about 45% of its cross cut area forcing the flow to accelerate and diverge into all the small holes [71]. An additional effect was that the flow was faster in the centre of the holes by comparison with the material in contact with the walls as was described in the pressure flow prediction. The relatively small hole of the strainer plate built up an elongation flow profile with a high shear rate and the mixing quality was significantly improved. Exiting the strainer plate, the single flows joined together in a new configuration. The residence time distribution widened, parts of the compound came into contact with parts fed into the machine before or after them, mixing them in a revised order. These
theoretical predictions were achieved in the outcome of the tests using the experimental set-up.

A follow up trial was run on 26\textsuperscript{th} October 2012 to determine the difference made to the mixing result by using a mesh in front of the strainer plate compared with the effect of the strainer plate alone. A comparison of the two situations is shown in Table 6.

<table>
<thead>
<tr>
<th></th>
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<th></th>
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<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>#3.1</td>
<td>40</td>
<td>PP S</td>
<td>78</td>
<td>20.9</td>
<td>9.8</td>
<td>19.0</td>
<td>30.3</td>
</tr>
<tr>
<td>#3.2</td>
<td>40</td>
<td>PP S</td>
<td>180</td>
<td>22.7</td>
<td>9.8</td>
<td>19.0</td>
<td>28.6</td>
</tr>
</tbody>
</table>

*Table 7: No mesh / mesh set-up*

For sample #3.1 the strainer plate was used alone. The strainer plate was employed to produce sample #3.2 with two layers consisting of a 40 mesh support and a finer 80 mesh layer.

Mesh is used for screens using a system based on the number of wires per inch. Together with a conversation table the dimension of the opening between the single wires was determined to be 0.177 mm [72].

The results are shown in Figure 57. In the right-hand sample, the flow marks caused by the strainer plate are clearly visible. On the left side these marks are hardly visible.

Based on the datasheet for the screen used with mesh 80, a free area of 55\% was left for the flow. The mesh was flattened against the screen by the p2 pressure so it was
assumed that only the free area of the strainer plate allowed flow through the mesh. Applying this to Equation (12) gives:

$$\epsilon = \frac{r_1^2}{0,55 \times r_2^2} - 1$$  \hspace{1cm} (13)

producing a mean elongation $\epsilon = 2.99$. This is more than twice the outcome of the results obtained with the strainer plate alone. Therefore a much-improved result was expected. The results in Figure 57 comply with this expectation.
Figure 57: Comparison of strainer plate results for samples #3.1, only strainer plate and #3.2, strainer plate and mesh 80

The mesh acted in the same way as an elongation mixer as did the strainer plate but on a smaller scale. Also the flow was divided into numerous small flows which re-joined in changed order shortly afterwards.

Compared to the free flow cross cut area of 45% [71] the mesh with 55% caused about the same mean elongation. The much improved mixing was caused by the combination of strainer plate and the mesh.

A major disadvantage of this static mixing arrangement is its lack of flexibility. It did not allow different settings unless parts were changed when the machine was stopped. This is opposite to an example where backmixing in the screw can be influenced via
screw speed. Energy input in mixing sections located in the front part of the screw can also be controlled within certain limits by the same effect.

However, the testing set-up showed clearly the advantages of elongation flow in mixing compared with simple shear flow. The aim of further developments is to include such elongation mixing in screw and mixing zone concepts to exploit these advantages.

5.1.5 Metal flake distribution

Contrary to the afore mentioned mixing of chemicals into compounds, this section concentrates on a new and novel approach of mixing metal flakes, in this case brass, into the compound. The purpose of the flakes was to improve the wear resistance of the final product in its application. This made it necessary to develop a new process which required proving for later industrial application. Additionally, the introduction of brass flakes allowed the quality of mixing to be assessed.

Damage or excessive wear to modern gear pumps is very unlikely unless the filling grade of soft metal fillers exceeds 40% volume [73].

5.1.5.1 Brass flake experiments undertaken at Runding

The trials described in this section were carried out on a Shark 70 machine. The machine was a 70 mm transfermix extruder, equipped with an 8 flighted multicut transfermix section D (transfermix) zone and a 14 L/D twin flighted screw and also included 4 flights in the transfermix section. An Extrex 70 RV gear pump equipped with herringbone gears was positioned in front of the extruder. The internal volume of the gear pump was 176 cm$^3$/rev.

The material was a Neoprene compound (Garlock 38803) with a density of 1.76 g/cm$^3$ having the Mooney values ML(1+4)100°C - 77MU and ML(1+4)130°C – 47 MU in
accordance with the specification of Garlock. The flakes properties complied with the following Table 8.

<table>
<thead>
<tr>
<th>Property (Meshes to the Inch)</th>
<th>Micron Size</th>
<th>Typical percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>+18</td>
<td>850</td>
<td>Less than 10 %</td>
</tr>
<tr>
<td>+36</td>
<td>425</td>
<td>Less than 50 %</td>
</tr>
<tr>
<td>+100</td>
<td>150</td>
<td>Less than 100 %</td>
</tr>
<tr>
<td>-100</td>
<td>150</td>
<td>Less than 20 %</td>
</tr>
</tbody>
</table>

*Table 8: Brass flake raw material specification [74]*

The brass flakes were constantly dosed into the main feed hopper of the machine by a Brabender LIW feeder unit. One test point was carried out with constant conditions for the extrusion system and the LIW feeder. Consequently, the quality of distribution during start-up of the machine and continuous operation could be analysed. The machine was set to the parameters shown in Table 9.

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>#</td>
<td>G 38803</td>
<td>21</td>
<td>84</td>
<td>5</td>
<td>14</td>
<td>49</td>
<td>108</td>
</tr>
</tbody>
</table>

*Table 9: Machine set-up for brass flake experiments*

The extruder was equipped with a breaker plate in the head and a slot die of 100 mm width by 8 mm in height.
The trials started with filling the empty machine by feeding base compound strips and the brass flakes simultaneously. Filling the machine took about 3 minutes. The samples shown in Figure 58 were cut directly in front of the die and also were marked with a test time allowing them to be correlated to the test start time of 0930.

![Overview photographs of brass flake experiment samples](image)

**Figure 58: Overview photographs of brass flake experiment samples**

A thin silver line was used to show where the cut was made in order to count the number of brass flakes in each sample. Some of the cut areas are shown in Figure 59 and 57. Again, the sample area, 25 mm wide for the whole thickness of the strip, was marked with a silver pen. The knife used to cut the samples was treated with a silicon spray to avoid any sticking when the samples were prepared. This kept damage to the
cut edges and surfaces to a minimum. However the aligned brass flakes were not cut but were bent and appeared angled at 90° to the flow direction.

Figure 59: Detail photograph showing cut surface of brass flake sample 1.1

Figure 60 Detail photograph showing cut surface of brass flake sample 2.1

Figure 61: Detail photograph showing cut surface of brass flake sample 5.1
Following the cut, the 25 mm wide sample areas were remarked again with a silver pen ensuring the area was constant for all samples. The brass flakes contrasted well with the surrounding rubber matrix. Counting flakes was aided by the use of a magnifying glass. Results are shown in Table 10. Due to the flow in the die, the flakes were all found to be oriented in the direction of the flow.

<table>
<thead>
<tr>
<th>Sample number</th>
<th>Sample area number</th>
<th>Number of brass flakes per area</th>
<th>Average value for sample strip</th>
<th>Time stamp sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.1</td>
<td>10</td>
<td></td>
<td>933</td>
</tr>
<tr>
<td>2</td>
<td>1.2</td>
<td>7</td>
<td>8.5</td>
<td>933</td>
</tr>
<tr>
<td>3</td>
<td>2.1</td>
<td>87</td>
<td></td>
<td>935</td>
</tr>
<tr>
<td>4</td>
<td>2.2</td>
<td>77</td>
<td>82</td>
<td>935</td>
</tr>
<tr>
<td>5</td>
<td>3.1</td>
<td>80</td>
<td></td>
<td>938</td>
</tr>
<tr>
<td>6</td>
<td>3.2</td>
<td>82</td>
<td>81</td>
<td>938</td>
</tr>
<tr>
<td>7</td>
<td>4.1</td>
<td>85</td>
<td></td>
<td>941</td>
</tr>
<tr>
<td>8</td>
<td>4.1</td>
<td>79</td>
<td>82</td>
<td>941</td>
</tr>
<tr>
<td>9</td>
<td>5.1</td>
<td>77</td>
<td></td>
<td>942</td>
</tr>
<tr>
<td>10</td>
<td>5.2</td>
<td>72</td>
<td>74.5</td>
<td>942</td>
</tr>
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<td>11</td>
<td>6.1</td>
<td>82</td>
<td></td>
<td>946</td>
</tr>
<tr>
<td>12</td>
<td>6.2</td>
<td>86</td>
<td>84</td>
<td>946</td>
</tr>
</tbody>
</table>

Table 10: Brass flake experiment data sheet
Figure 62 is a graphical depiction of Table 10. To decrease counting errors, averaging each value counted for each of the two surfaces was necessary. It was possible for some flakes to be counted twice or two flakes to be mistaken for one since they were in the same location. The average values are shown in dark blue and square markers while the raw data is depicted in green and round markers. The first two samples show almost no traces of brass, but after only two minutes the number rose and remained constant at approximately 82 flakes per sample area for the next ten minutes of the test. A drop was observed at around 7 minutes after stabilisation, possibly caused by inconsistent feeding of brass due to the small sheets of rubber that were fed into the hopper.

![Brass flake experiment graph](image)

**Figure 62: Brass flake experiment; number of flakes per area, (■ denotes average value per section, • denotes raw data)**

However, these results show the distribution became constant very rapidly and therefore the distributive mixing ability of the equipment at this set-up was excellent. As a follow
up, further trials with more compounds using continuous strips to improve feeding consistency are scheduled on a larger scale.

5.1.5.2 Distributive mixing trials with metal flakes on a production scale

The approach described using brass flakes as an indicator proved to be simple and effective when analysing distributive mixing. However the analysis needs to be improved.

As a first step, a different machine set-up will be tested with the inclusion of brass flakes. The new machine will be built for the purpose of mixing brass and other free flowing material on an industrial scale. Also defined sample volumes and measurement of their density will replace the current procedure of counting flakes by hand. A constant density allows feedback on the accuracy of mixing. The density of the batch containing flakes compared with the master batch density will relate to the absolute amount of flakes mixed regardless of their shape.

5.1.5.3 Set-up 2 – Shark 90

The trials on this machine were carried out in two locations. The first trials were run on December 18\(^{th}\) and 19\(^{th}\), 2012 at the VMI plant in Runding, Germany. A second test was conducted on June 27\(^{th}\) 2013 at the plant of Garlock in Palmyra, New York.

The machine used was a Shark 90 extruder gear pump combination based on an extruder with a 90 mm screw diameter and 16 L/D process length. An Extrex 90-5 RB gear pump with 218 cm\(^3/\)rev internal volume was installed on this machine. It was equipped with 12-6-flighted transfermix zone (12 half flights, 6 full flights) in the barrel. The screw was also equipped with a mixing zone with 8 flights. An integrated vacuum zone with an adjustable throttle to allow devolatilisation of the compound was
present but not in use, the vacuum suction port was closed with a plug to avoid compound output. The throttle was kept fully open during the trials.

The dosing system was a Brabender Flexwall 40 loss in weight screw feeding system. For the trials, a screw of 20 mm diameter, 18 mm pitch and a barrel with 32 mm inner diameter was installed to obtain initial outputs. Table 11 shows the results of the dosing unit test and calibration run prior to the mixing tests. The unit performed as anticipated and the essential quantities calculated for the mixing test were achieved within the feeding limits of the unit. This setting allowed dosing rates up to 200 kg/h free flowing brass flakes. The second option; a barrel with a 35 mm diameter and a screw with 28 mm diameter and 33 mm pitch was unnecessary. The estimated output would have been much higher than necessary and the unit could not have delivered the smaller amounts necessary for the start-up of the process.
<table>
<thead>
<tr>
<th>Set-point output [kg/h]</th>
<th>Measured weight [kg/h]</th>
<th>Dosing unit screw speed [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>25.25</td>
<td>10</td>
</tr>
<tr>
<td>25</td>
<td>25.2</td>
<td>10</td>
</tr>
<tr>
<td>50</td>
<td>49.99</td>
<td>22</td>
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<tr>
<td>50</td>
<td>49.88</td>
<td>22</td>
</tr>
<tr>
<td>75</td>
<td>74.94</td>
<td>32</td>
</tr>
<tr>
<td>75</td>
<td>73.4</td>
<td>32</td>
</tr>
</tbody>
</table>

*Table 11: Dosing unit test and calibration*

The dosing unit fed into the machine via a hose guiding the brass flakes into the feed hopper directly above the in-feed zone. The flakes fell in-between the wall of the hopper and the feed strip and were dragged into the barrel.

The compound and flakes first passed the mixing zone and from there went through the throttle section. A pressure build up zone followed, before feeding the material into the gear pump. An extrusion head of 200mm inner diameter is attached to the gear pump. For the trials, it was equipped with a throughput ring without any strainer mesh and die for the product. The preforms were hollow cylinders open on one side as for the samples shown in Figure 63.
Figure 63: Preforms produced during brass mixing trials

Cutting was done with a rotating knife cutter. The knife cuts were made in a position directly at the die which was determined by a calculation of the output based on the internal volume of the gear pump. This system allowed very accurate and constant cutting results with very uniform preform weight. Downstream from the cut the preforms were cooled in a water tank and conveyed to boxes where they were stored manually for drying off. Each box held about 40 samples. Each sample was later weighed on a scale and the result recorded.

During the trials, the machine data was collected in a spread sheet holding all relevant set-points and actual values derived from the machine. The samples were made to the actual specification of the customer preforms to keep realistic settings for the whole process.

In the current process, the mixing of this compound is carried out on a two-roll mill batch by batch with the manual addition of the brass. The preforms were shaped and cut
later in a step carried out on a ram extruder as set out by Garlock staff during the trials. Later the preforms were put into heated presses for final shaping and vulcanisation.

Again the compound was a Neoprene formula with a density of 1.76 g/cm\(^2\) and the Mooney values ML(1+4)100°C - 77MU and ML(1+4)130°C – 47 MU in accordance with the Garlock specification. The feed strip was endless and 150 x 10 mm in cross-section. The machine was able to pull this strip with the support of a hydraulically driven feed-roll into the feed hopper. The flakes were again as specified in Table 8.

Theoretical consideration of this mixing process led to the conclusion that a good mixing quality was achieved when an even distribution of the brass flakes in the surrounding compound matrix was reached. The weight per volume should therefore also become more uniform as the quality of mixing improves. The preform cutter and the control of this machine were built to cut as precisely as possible with preforms produced at a constant volume. Measuring and comparing the weight distribution of the preforms, with and without brass flakes mixed in, would allow determination of the distribution uniformity.

The basic test planning is shown in Table 12. First a base line of unfilled samples was produced and measured. In consequence, two extrusion speeds were chosen. The machine was running with identical p1 values for both speeds. Different speeds did not influence the output and the cutter accuracy. These two set-ups allowed determination of the level of accuracy of the machine to be ascertained.

Thereafter, a set of tests with 50 % of the final dosing rate was planned. Two outputs were to be run, each on three p1 settings.

The second set was to be with the full amount of dosing as to the product specification planned again with two output set-ups at three sets of p1 each.
Due to a shortage of the compound and brass flakes during the trial, the second set of high output trials had to be cancelled at short notice at site.

<table>
<thead>
<tr>
<th>Compound number</th>
<th>Dosing material</th>
<th>Dosing rate</th>
<th>Pump speed</th>
<th>p1 set</th>
</tr>
</thead>
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<td>#0.1</td>
<td>Base compound 38803</td>
<td>0</td>
<td>21</td>
<td>30</td>
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<td>Base compound 38803</td>
<td>0</td>
<td>32</td>
<td>30</td>
</tr>
<tr>
<td>#1.1</td>
<td>Base compound 38803</td>
<td>brass</td>
<td>8.705</td>
<td>21</td>
</tr>
<tr>
<td>#1.2</td>
<td>Base compound 38803</td>
<td>brass</td>
<td>8.705</td>
<td>32</td>
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</tr>
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<td>Base compound 38803</td>
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<td>17.41</td>
<td>21</td>
</tr>
</tbody>
</table>

*Table 12: Test set-up for Garlock mixing trials*
5.1.6 Mixing quality dispersive mixing - Results

Firstly, the measured samples were checked and any unrepresentative spurious measurements were marked and eliminated from the results. There were three reasons for these unrepresentative results:

a) There was a weight correction function on the machine. Preforms were cut and put onto a scale. The weight of a preform was either satisfactory or outside of the specification. In this case the actual weight was inputted to the machine control and the set-points updated accordingly. The preforms that followed were of the correct weight. Prior to correction, an operator had obviously caught some of the samples in the sample baskets by accident.

b) The manual sorting of the samples into baskets may have included preforms taken by accident into the wrong baskets.

c) Despite high concentration and checks, typing errors could have occurred when measuring the sample weight.

The first two trials showed similar results. The machine was first set to 21 rpm pump speed representing a 484 kg/h output and the second to 31 rpm equivalent to a 738 kg/h output. The measured preform weight standard deviation was 50% less than the manufacturer’s internal specified accuracy, showing the samples were very uniform in weight and the machine was operating as intended. The brass did not influence this cutting process so the results on preform accuracy could be directly compared. The difference in speed was not significant, but there was a tendency for slightly better accuracy at the higher speed. This could be explained by improved homogeneity in the compound at this level caused by a faster rotating screw and higher compound temperature as a result of increased specific mixing energy.
The first mixing set was carried out with 50% of the final dosing, at 8.705 % dosing rate. Due to the higher density of the mixture and a constant output volume per pump revolution, the same pump set speeds led to higher outputs of 512 kg/h and 780 kg/h respectively. At each speed two p1 settings were set and samples were taken. Only the higher setting of p1 at the highest speed was unsuccessful. The screw reached its maximum speed and was not able provide enough throughput to fill the pump cavities reliably. In this case, the cutter calculation was possibly faulty and produced unreliable preform weights, so test #1.6 was eventually discontinued.
With increasing \( p_1 \) pressure, the uniformity of the measurements increased. The standard deviation dropped from 3.5g at 30 bar \( p_1 \) to 1.5g at 70 bar \( p_1 \). It fell almost to the level of the reference measurements without brass at 1.2g as shown in Figure 64.

Similar behaviour was observed for the third set of measurements. Here the dosing rate was at the final product rate of 17.41%. The available amount of brass limited this trial series to one speed level. The level was also set to actual planned production rates at 21 rpm equalling 540 kg/h. Here also the standard deviation fell from 2g at 30 bar \( p_1 \) to 1.2g (the base line) at 70 bar \( p_1 \). This indicated a high quality of mixing with a constant and even distribution of brass flakes. This situation is shown in Figure 64.

![Figure 64: Comparison of inlet pressure versus standard deviation](image-url)
Figure 65 and Figure 66 show the differences in detail. In both figures the base line from trial #0.1 is marked with green triangles (▲). The red squares (■) mark the samples from test #1 while the blue diamond markers (♦) show samples from trial #2. Figure 65 shows all measurements for a 50 bar inlet pressure, #1.3 and #2.3 sets in relation to #0.1.

![Figure 65: Brass mixing trials with input pressure at 50 bar and base line](image)

The second diagram, Figure 66, shows the comparison at an inlet pressure p1 of 70 bar for trials #1.5 and #2.1.

![Figure 66: Brass mixing trials with input pressure at 70 bar and base line](image)

The bandwidth by comparison with
Figure 65 is much narrower indicating a far superior mixing quality. The lines are almost as constant as the base line. A visual inspection of the cut surfaces showed a uniform pattern of brass in the surrounding rubber compound matrix. A comparison, as for the first brass flake mixing experiment, by counting the flakes per surface, was not performed as the weight deviation provided more accurate results than the counting method.

The temperatures stayed within specified limits in all trails. As a consequence of these outcomes, the aim of obtaining a high quality mix was met.

5.2 Overall energy consumption

By comparison with the work described in section 5.1.3, the specific mechanical energy (SME) input of the compound was calculated based on the test data and according to Equation (8) [6].

No direct power reading was available for the AC motors driven by frequency converters in this machine. Therefore the machine was considered as a closed system, measuring only the output per time and the temperature change of the compound before and after processing. The heat quantity equalled the mechanical mixing energy put into the system. Bearing in mind the poor heat transfer properties of rubber compounds and the relative short residence time, only small losses via the cooling system of the machine were not taken into consideration in this calculation. As these factors stay constant during the trials, the different measurements can be compared with each other on the basis of a revision of Equation (8) given by Equation 14.
The overall energy of the output was split up into the thermal energy of the compound ($Q_{\text{comp}}$) and the energy of the brass flakes ($Q_{\text{brass}}$). This was necessary because of their different heat capacity. Values for $c_p$ were taken for neoprene rubber as 2176 J/(kg K) [75] and for brass as 377 J/(kg K) [20]. $Q$ was determined using Equation (15).

$$Q = c \cdot m \cdot \Delta T \ [J]$$  \hspace{1cm} (15) [76]

The brass flakes were heated by the compound in which they were embedded but not as a result of shear, so this additional energy had to be added to the mixing energy of the compound. The sum was then divided by the mass of the compound $m_{\text{comp}}$ as per Equation (16)

$$SME = \frac{Q_{\text{comp}} + Q_{\text{brass}}}{m_{\text{comp}}} \ [kWh/\text{kg}]$$  \hspace{1cm} (16)

A significant difference in energy consumption between the different p1 pressure settings could not be observed. With increased dosing rates the SME also rose as shown in Figure 67.

The transported volume per screw revolution was considered as constant while running a set-point. Adding brass flakes reduced the ratio of compound per volume element. The remaining compound per volume element experienced more shear as the whole volume was sheared during transport.
Figure 67: SME brass mixing trials

The SME average value in the absence of brass was 0.050 kWh/kg, while with brass mixed into the compound, the value rose to 0.056 kWh/kg.

During the first acceptance trials for the specified machine on 17th and 18th December, two mixing set-ups were tested. The limiting amount of brass supplied by the customer did not allow more expansive testing. On the other hand, these tests provided a comprehensive database of relevant information for comparison between the trials without brass flakes and those performed later with flakes added. It also facilitated the planning for the on-site trials.

Using the same calculation described earlier in this section, the SME with no brass flakes added averaged of 0.051 kWh/kg, while the value for that with brass flakes was larger at 0.052 kWh/kg.

The TCU setting was different in the two trials. This makes a direct comparison impossible. The basis for the trials in December was obviously another batch from the same compound. This also influenced results [36]. However, the trends for both measurements were similar, indicating that the same conclusion could be drawn that
distributive mixing of the brass flake into the compound requires a relatively low energy input.

Compared with the trials in Chapter 4, only a distributive mixing procedure and analysis was performed in the work described here. Also there were no small-scale agglomerations in the brass which would require extra energy to break up. The brass was free flowing and individual flakes did not tend to stick together.

5.3 Conclusion

The trials provided good results for distributive mixing. Mixing of granules fed into a feed hopper or via side feeding to the extruder can be considered as distribution because the granules consist of a compound which includes the chemicals which are already dispersed. This can be considered opposite to the mixing of chemicals as powder. The compound only needs to be evenly distributed into the master batch. By doing this, the chemicals are also well distributed in the master rubber matrix. Usually the granules are no longer visible if mixing is successful in the extruder.

The two colour tests are considered to demonstrate distributive mixing on a very fine scale. The trials were in accordance with the previously discussed models. Different settings allowed control of the process. Distribution of small-scale fillers was proven to be successful. Elongation mixing proved to be the most efficient way for distributive mixing for small scale and dispersive mixing.

The brass flake mixing was not strictly compliant with the previously mentioned model based on liquids. Considering the free flowing metal flakes as liquid, the behaviour complies with the model with only the limitation that the single particles cannot be
broken down to a smaller size. The aim of the trials was to achieve a comprehensive
distribution and not to dissolve the brass flakes.

Mixing of brass flakes was primarily characterised by low agglomeration forces
between the particles and solely distributive mixing in the machine with no dispersive
mixing. Therefore it was possible to keep the energy input for mixing very low. Shear
stress from the screw was combined with backmixing.

Results showing an excellent distribution were reached with medium inlet pressures.
Observing the flow behaviour discussed in Section 2.3, at medium p1 pressure,
considerable backflow occurred at the screw tip in this set-up.

Calculation of energy consumption proved that considerable savings could be made
compared with currently used mixing technologies. However, temperature limits for the
mixed compounds will limit the maximum mixing energy dissipation. Therefore mixing
zones have to minimise shear flow while improving elongation flow to deliver
efficiency.
Chapter 6

Conclusions and recommendations for future work

The primary aim of this research project was to uniquely redesign, develop and validate the continuous final mixing of rubber compounds using single screw extruder gear pump combinations. This was achieved by introducing a novel method of assessing the quality of mixing and in consequence reconfiguring known processes.

Introducing metal flakes into a rubber compound mix was shown to be successfully implemented for a single screw extruder gear pump combination using the advantages offered by gear pumps for metering output and control of the input of the additional material together with the mixing abilities of a single screw. This allowed the control of mixing to be independent from the output.

The process is not limited to distributive mixing of metal flakes. The capability of dispersive mixing of chemicals or other fillers has also been demonstrated successfully using a similar approach.

The trials also showed that a standard extruder and its screw and barrel set-up are incapable of reaching a sufficient mixing performance without the inclusion of special mixing elements. It is possible to integrate such elements, but the increased energy input leads to higher temperatures or conversely might limit the output for certain compounds in particular mixing cases.

Optimisation of heat transfer capabilities in the mixing zone or the regions beyond this zone will be an important topic for future research.
However, the process delineated in this work has several advantages allowing savings by using less energy when compared with an internal mixer. Also further savings can be realised by the ability to miss out one mixing step in the internal mixer.

The trial and validation of the proposed novel changes for industrial scale production at a customer site was successfully carried out. The machine used was equipped with a transfermix mixing section in the barrel and screw. The additional vacuum section added more extrusion length and allowed devolatilisation for other compounds which will widen the possible product spectrum.

The samples produced fulfilled the quality requirements for successful extrusion by having very low weight variation. Some minor issues will have to be resolved in the industrial process. Particularly, the cleanout and changeover to different material processes need to be improved to reduce time and effort. Long-term abrasion issues caused by the brass are unlikely due to the high hardness difference between brass and the steel of the gear pump wheels. However, the brass flake mixing process was proven to work well and be viable when working on the industrial scale shown. The equipment produced a good mixing quality with the correct set-up. Furthermore material waste will be reduced in future processes as a result of the developments described here. The conditions during the trials were indicative of a typical production situation. Operation of the machines used in industry was carried out partially by company operatives under supervision without problems.

Future applications which will exploit the advantages of this process will be in the mixing of macroscopic particles such as flakes, short fibres or other free flowing materials with the requirement for constant distribution in highly viscous compounds. It will also be possible to manufacture magnetorheological elastomers (MREs) on a large
scale to allow them to be used in future industrial applications. These materials consist of a rubber matrix incorporating microscopic ferromagnetic particles enabling the application of an external magnetic field to influence the elastomer behaviour.

The transition of the research results into an industrial system allows the improvement of the production processes. However, it is necessary to have a control system that will be easily assimilated and appropriate when used by operatives in the workplace. Hence, the system needs to be self-controlling as far as is possible. The system also needs to be sufficiently robust to be able to react to unplanned situations such as master batch variations, which are not part of the process.

The wider aim is ultimately to establish a system that is as easy to use as today’s simpler machine systems. To reach this goal, integration of measuring systems, but also the addition of further analysis systems, will be necessary. In particular, the control of mixing parameters, including temperature and screw speed in relation to sensor systems or scientific calculations, is a necessary future development. The models discussed here need to be developed to allow this aim to be achieved.

Further, it will be essential to validate such a system in a real world industrial environment.

Further research needs to be carried out on the necessity of optimising mixing zones in screws and barrels to allow more efficient elongation mixing and reduce the shear as much as possible. The advantages of elongation mixing have been clearly demonstrated. Together with the potential for improved heat transfer, allowing cooling of the active compound during mixing, the optimisation described here would allow higher mixing energy input and widen the field of application for continuous final mixing.
Finally a variable set-up for such mixing elements will allow users to influence the process without stopping the machine or exchanging machine parts, which will considerably increase the flexibility of the machine and processes.
References


