

Effect of Sequence of Mixing and Internal Mixer Parameters on the Viscosity of Compounds for Tire Components

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Abstract: The objective of the mixing process of rubber compounds in an internal mixer is to blend the ingredients of the formulation into a uniform, well dispersed mix. Hence the mixing sequence and parameters are designed to assure batch to batch uniformity, fast reduction of viscosity and to make the most efficient use of time, power and equipment for optimum productivity. This research aims at showing a better way of achieving reduced viscosity using optimized mixing sequence and parameters by comparing the usual approach of temperature controlled mixing and energy controlled mixing in the internal mixer. The effects of the various mixing conditions, sequence of addition of ingredients and internal mixer parameters were observed in terms of time, dump temperature, energy used and Mooney viscosity of the compounds. The results show that the application of energy controlled mixing criteria as a priority in place of temperature controlled mixing criteria, with properly optimized sequence of addition of ingredients and control of internal mixer parameters (temperature, energy, RPM and ram pressure) is an effective method of controlling compound viscosity.

Keywords: viscosity, energy, temperature, mixing control, rubber compounds.

I. INTRODUCTION

The art of creation of rubber formulations by rubber compounders finds parallels in the art of cooking. By selecting specific ingredients and mixing them carefully the rubber compounders achieve specific effects in rubber products. Science has been able to explain most of the chemistry behind rubber compounding and mathematical modelling is now used to predict the performance of rubber products from material properties. However, in the case of automobile tyres, eventually the experience and skills of the tyre compounder and the raw material supplier is the only sure way to push the frontiers of tyre performance. Novel and exciting materials, machineries and mixing techniques at different intervals offer new opportunities to compounders and help to raise the levels of tyre performance to new heights [1].

Compounders have tried to understand how the different ingredients combine to give certain material properties through various experiments that are carefully designed. Various years of experience and experimentation are employed to evaluate speculative options to improve properties of rubber products such as tyres. Problems usually arise even after the early laboratory stage developments. Magnifying laboratory mixes up to factory scales adds a whole new dimension to the issues. At the end, when the tyres are produced, the performance can be evaluated in different ways. It is almost impossible to track all the many objective and subjective tyre ratings back to those original simple laboratory tests, let alone the raw material formulations themselves. Large databases and 'intelligent' software packages may offer some hope in the future to link raw material recipes to the processability of components and finally to tyre performance, but eventually the interpretation of results has been based on the experience of the compounders [1].

Mixing of rubber compounds is generally done in a batch process using equipment such as internal mixers or two roll mills. The purpose of the mixing process is to blend the ingredients of the formulation into a uniform, well dispersed compound. Mixing control is designed to assure batch to batch uniformity, reduction of viscosity and make most efficient use of time, power and equipment [2].

Three factors contribute to the lack of uniformity of mix: improper control of mixing cycle, variation in raw materials and errors in ingredient weighing. Raw material variation, especially with polymers, can be minimized through tight quality control specifications of the raw materials and by selectively blending different lots of the raw material to give more uniform average properties. Weighing errors are monitored and reduced through automated weighing and addition of materials, more precise weighing techniques and checking of ingredient and final batch weight [3].

The last factor relating to compound uniformity is control of mixing cycle itself which also has a greater effect on viscosity of compounds. Rubber compounds have traditionally been mixed according to a time-temperature and temperature-energy specifications. However, these specifications are extremely dependent on the mixer type, size and condition. Control specifications of time-temperature do not always assure batch to batch uniformity and proper control of viscosity, due to differences in specifications. These specifications are certainly difficult to translate to different pieces of equipment.

Various literatures has however shown that mixing according to energy (work input) specification has shown to accurately predict material properties, improve batch to batch uniformity and effectively control viscosity independently of mixer type and condition. Energy (work input) is the combined effect of power and time during mixing; this is shown as the area under a typical power/time curve (Fig. 1). Initially the power increases rapidly when the mixer is loaded, after the mixer is fully charged the polymer is sheared (masticated), causing a temperature increase and a reduction in viscosity. The power therefore decreases as mixing time increases. The Mooney viscosity of compounds after mixing has been shown to be proportional to the energy (work input). Polymer breakdown is only part of the objective of mixing, uniform incorporation of all ingredients of the formulation must also be achieved. Work input is not only the work put into polymer breakdown, but also into the incorporation of fillers, oils etc [3].

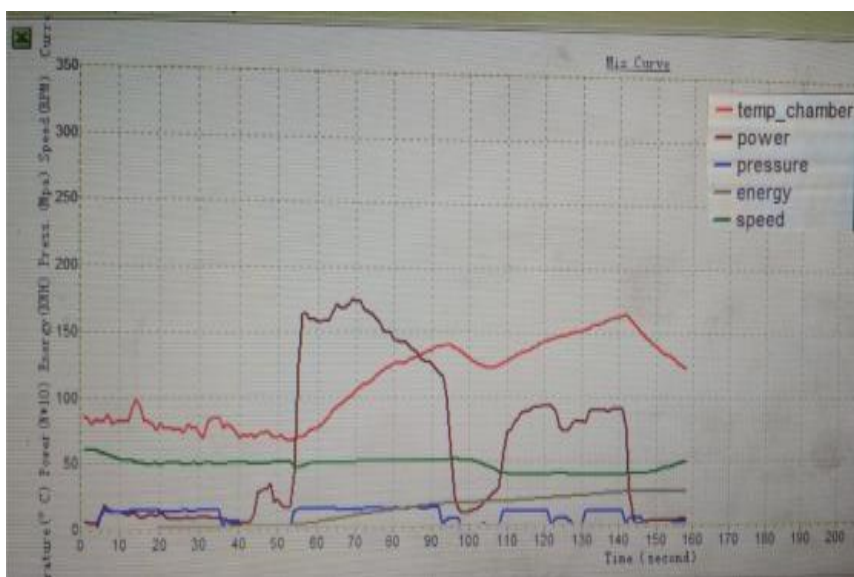


FIGURE 1: POWER/TIME CURVE (MIX CURVE)

II. FACTORS AFFECTING VISCOSITY OF COMPOUNDS IN THE INTERNAL MIXER

A. Shear stress

The magnitude of the force acting against the cohesive force of the aggregates depends on the effective diameter of the agglomerate, the viscosity of the matrix, and the strain rate. High-structure blacks exhibit a higher shear force during the dispersion stage while low-surface area blacks possess low cohesiveness and this affects the viscosity [4]

The approximate magnitude of the values of shear stress (τ), for the various mixers can be calculated using the power law equation

$$\tau = k\dot{\gamma}^n \quad \text{Eq. 1}$$

Where k and n can be obtained from the flow curve and $\dot{\gamma}$, the shear rate, is calculated from the rotor speed

Knowledge of the level of shear stress and its relationship with rotor speed is essential in anticipating the processing problems [5] [6].

B. Shear strain rate

A rubber mix in an internal mixer is subjected to a spectrum of shear rates. In the areas far away from the rotor tip the shear rates are minimum; increasing gradually till a maximum is reached in the nip region. This maximum shear rate, $\dot{\gamma}$, above the tip of the rotor wings can be calculated using equation (2). This relationship holds if pressure flow is ignored and that no slippage prevails.

$$\dot{\gamma} = \frac{v}{h} \quad \text{Eq. 2}$$

Where: $\dot{\gamma}$ – Shear rate, v - Velocity of rotor, h – Clearance

Typical range of shear rates for low intensity mixing is $100 - 250\text{s}^{-1}$ and for high intensity mixing is $200 - 600\text{s}^{-1}$.

From Equation (2) shear strain rate is directly proportional to the rotor speed and inversely proportional to rotor tip clearance. It is important to know at what shear rate a mixer is operating as optimum dispersion is obtained only within a limited range of rotor speeds, with decreasing degrees of dispersion on both sides of it. This is attributed, to the power law type of relationship between shear strain rate and shear stress [4].

C. Shear strain

The shear strain $\dot{\gamma}$, determines the extent of mixing and batch uniformity. Assuming that rubber sticks to both the chamber wall and the rotor tip Bergen found that the shear strain varies between the ratio of width, b , of the rotor blade to the clearance, h , and maximum at the rotor tip. It is best characterised by the shear output, s , which is the shear per unit volume of the material and unit time [7].

$$s = 2 \frac{b}{h} \quad \text{Eq. 3}$$

D. Ram pressure

Due to the low bulk density and fluffy nature of most of the additives ram pressure helps to reduce the incorporation time by decreasing voids and increasing the area of contact thereby reducing viscosity. In addition the resultant increase in hydrostatic pressure reduces slippage on the working surfaces of the mixer. Slippage must be reduced to a minimum as it not only affects the shear rate and the dispersive action at the rotor tips but also reduces the cooling efficiency.

The resultant effect of increasing ram pressure is, therefore, the overall reduction in mixing time and improved efficiency [9].

E. Rotor speed

Up to a certain limit rotor speed determines the mixing time and the output rate. The straight line relationship between rotor speed and mixing time and power consumption is well-known as it helps to control viscosity: a constant number of rotor revolutions are required to obtain the same end result.

The theoretical optimum range of shear rates can be obtained from Equation (2).

With soft stocks the optimum rotor speed for shear rate must be selected to achieve the essential minimum shear stress level which may not be attained at low or normal speed. Equation (1) also reveals the power law relationship between shear stress and shear rate [8]. As such after a certain point further increase in rotor speed may not significantly change the shear stress magnitude. Increasing rotor speed also results in higher stock temperature which counteracts the increase in shear stress. Therefore, since dispersion and viscosity control can be efficient only if the shear stress exceeds a critical value, which depends on the cohesive strength of the aggregates and polymer, mixing efficiency of a mixer is reduced if the rotor speed is too high. The above phenomenon is exploited for variable speed mixing techniques [9].

F. Cooling efficiency

The mechanical shearing action imparted onto the filled rubber mix produces considerable frictional heat due to the dry particulate additives and high viscosity of raw rubber, and the relative motion of the moving material against the rotor surfaces and the chamber wall. In addition, the shear deformation that rubber is subjected to during the mixing process results in hysteretic heat loss [10]

The mixing temperature exerts very appreciable effect on the rheological properties of the mixing stock. The temperature dependence of the viscosity, η , of liquids is usually exponential and can be described by an Arrhenius type of equation

$$\eta = A \exp(E/RT) \quad \text{Eq. 4}$$

Where A is a constant, R is the gas constant, E is the activation energy and T is the absolute temperature. Because of the inverse relationship between the viscosity and temperature and the effect on the shear stress level mixing temperature must be effectively controlled [11].

III. EXPERIMENTAL

Experiments designed to study the effects of mixing sequence and parameters on viscosity of compounds were carried out in the phase 1 and phase 2 fully automated Banbury (6 wings) master mixers of CEAT Limited. A component of tire: AGS (Abrasion Gum Strip) compound (1st stage mixing) was used for the experiment.

All materials were received according to the company’s standard procedure from approved list of suppliers, mixing sequence and parameters were designed only for experimental purposes. Batch weight, fill factor, specific gravity and expected viscosity specification for all the experiments are same.

In this study the temperature controlled mixing and energy controlled mixing was set for each mixer using the mixing sequence and technical parameters as shown in Table 1 and Table 2. In all the test runs performed in this study, the chamber, rotor and drop door temperature were maintained at 55°C, 45°C and 34°C respectively. Total mixing time, discharge time, discharge temperature, discharge energy and discharge power were recorded for each test run by the data logging system.

Samples were cut from every batch but Mooney viscosity test was done for only one out of every ten batch using the Ueshima Viscosimeter according to the company’s standards of testing. The sample is put in the instrument heated chamber, which is then closed. Due to high dependency of viscosity on temperature, sample is given approximately 1 minute to preheat to required temperature (measurement at isothermal conditions). After the first minute is elapsed, rotor starts and the torque measurement is initiated. Rotor torque is read in the fourth minute and subsequently the viscosity is calculated automatically. When the measurement is completed, rotor stops. Mooney viscosity is designated as ML(1+4)100°C.

TABLE 1: TEMPERATURE CONTROLLED MIXING SEQUENCE AND PARAMETERS

Condition	Time(S)	Temp.(°C)	Ener.(KwH)	Sequence	Pressure	Rotor(RPM)
Simultaneous execution	20	135	15	Add Polymer	14	60
				Ram down		
				Keeping		
				Add Carbon		
Temperature	20	135	15	Add dust collector	13	47
				Ram down		
				Ram up		
				Keeping		
Temperature	20	145	15	Ram down	11	41
				Ram up		
				Keeping		
				Ram down		
Temp and Energy	8	165	22	Ram up	42	42
				Keeping		
				Ram down		
				Open discharge door		
Simultaneous execution	8	165	22	Keeping	60	60
				Close discharge door		

TABLE 2: ENERGY CONTROLLED MIXING SEQUENCE AND PARAMETERS

Condition	Time(S)	Temp.(°C)	Ener.(KwH)	Sequence	Pressure	Rotor (RPM)
	20			Add Polymer	14	52
				Close feeding door		
				Ram down		
				Keeping		
				Ram up		
Simultaneous execution				Add Carbon		
				Add dust collector		
				Ram down		
Energy	3	135	15	Ram up	11	41
				Keeping		
				Ram down		
Temp and Energy Simultaneous execution	12	160	19	Ram up		
				Open discharge door		
				Keeping		
				Close discharge door		60

IV. RESULTS AND DISCUSSION

Three different tests were carried out on each mixer using a natural rubber compound (TSR 20) to be used as AGS component of the tire, 20 batches were mixed in each test and the 9th and 18th batches were tested for viscosity in all cases. Recipe (batch weight, polymer, filler and chemicals) are same for all tests with variation in only mixing conditions, sequence and technical parameters. The results (time, temperature, energy and viscosity) are recorded for the temperature controlled and energy controlled mixings.

TABLE 3: TEMPERATURE CONTROLLED MIXING RESULTS

Temperature controlled mixer				
	Time(s)	Temperature(°C)	Energy(KwH)	Viscosity (MU)
Test 1	132	165	22.8	148.2
	135	165	23.3	146.1
Test 2	137	165	23.1	145.8
	144	165	23.3	142.5
Test 3	138	165	25	148.6
	139	165	24.5	150.5

TABLE 4: ENERGY CONTROLLED MIXING RESULTS

Energy controlled mixer				
	Time(s)	Temperature(°C)	Energy(KwH)	Viscosity (MU)
Test 1	149	160	22	139.7
	153	160	22.5	136.9
Test 2	149	160	22.4	138.9
	159	160.2	23	136.1
Test 3	147	160	22.2	132.4
	154	160	22.9	131.1

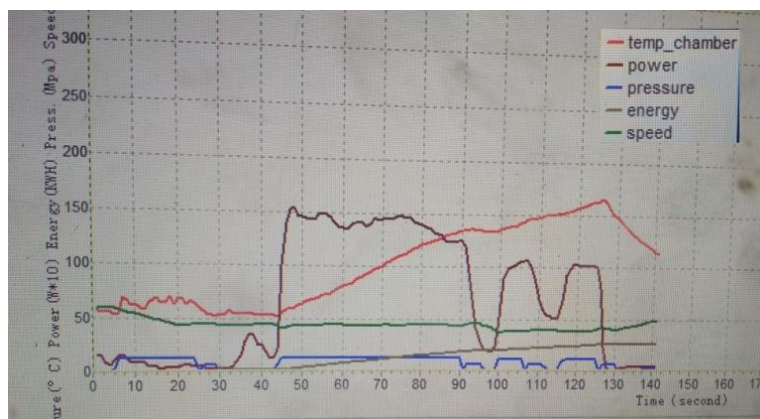


FIG. 2: TEMPERATURE CONTROLLED MIXING GRAPH

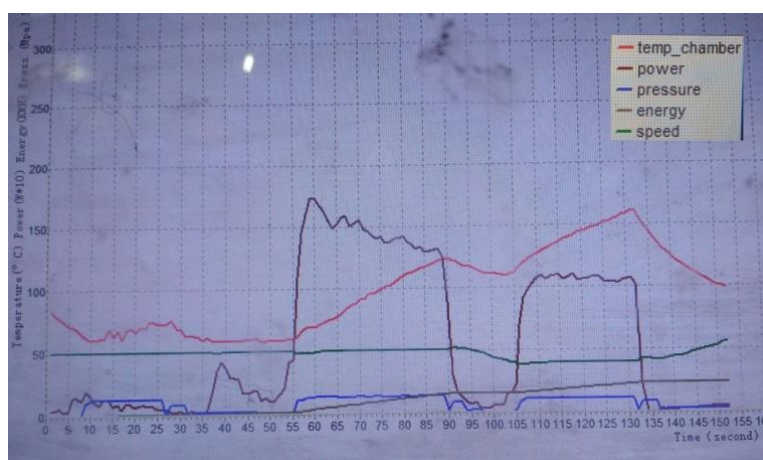


FIG. 3: ENERGY CONTROLLED MIXING GRAPH

From the results, using the temperature controlled mixing, the dump temperature set is at 165°C, an average of 137.5secs and 23.67KwH from the 3 tests was used which resulted in an average of 146.95MU of viscosity while using energy controlled mixing, the temperature set is at 160°C, an average of 151.8secs and 22.5KwH from the 3 tests was used which resulted in an average of 135.85MU of viscosity.

Comparing the results, mixing was done at different optimized RPM and ram pressure deemed fit for the different mixing conditions, the average time used for mixing was less by 14.3secs for the temperature controlled mixing which can be as a result of difference in mixing sequence time and dump temperature (pre-mastication: 20secs each, keeping: 0secs and 3secs, dwell time: 8secs and 12secs, dump temperature: 165°C and 160°C for temperature controlled mixing and energy controlled mixing respectively), while the average energy used for mixing was less by 1.17KwH for the energy controlled mixing which also resulted to an average difference of 11.1MU in viscosity with energy controlled mixing giving a better result. This can be attributed to the fact that polymer breakdown, uniform incorporation and dispersion of fillers and chemicals into the polymer which brings about viscosity reduction is actually more of the work put into mixing rather than temperature because initially the power increases rapidly when the mixer is loaded, after the mixer is fully charged the polymer is sheared (masticated), causing a temperature increase and a reduction in viscosity [2]. The power therefore decreases as mixing time increases which can be seen clearly as shown in the mixing curves (Fig. 2 & 3)

V. CONCLUSION

The results of this study, using energy controlled mixing criteria with optimized parameters helps to regulate energy used in internal mixing of tyre formulations with little variation in temperature for required specified viscosity. This results in reduced temperature in most cases helps to regulate the heat applied to the compound that may affect end product and helps to give the mixing process the required shearing time and work done to get the required specified viscosity. Mixing at high temperature; although good for mixing, affects the shelf life of the end product as dumping at constant high temperature induces more heat into compound without giving specified viscosity.

From the results, it can be clearly deduced that using energy mixing/dumping criteria as a priority in place of time-temperature criteria, temperature-energy criteria or temperature mixing/dumping criteria with properly optimized mixing parameters (RPM, ram pressure, temperature and energy) is an effective method of controlling compound viscosity in the internal mixer.

ACKNOWLEDGMENT

The investigations presented in this report is a part of the research project “**Research for Mixing Process Improvement: Compound Held up Reduction**” carried out by Oluwatobi Elijah Akindele, MTech student, PSRT, CUSAT (2017-2019).

We would like to thank CEAT Limited Halol plant, for supporting this work by providing material and equipment resources. Oluwatobi acknowledges the support of Mr. UnniKrishnan (V.P – R&D), Mr. Biju A.K. Nambiar (GM- Halol Plant Technical), Mr. Biswaranjan Dash, (Sr. Manager R&D – Commercial Compounding).

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