Carbon Dispersion Improvement in TBR Sidewall Compound

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Introduction

In this progressive era, transportation is one of the mainstays of a growing and healthy economy. Even after two lakh years of human existence, tons of discoveries and millions of improvements in the field of transportation, transportation via roads is still the most common and most used mode of transport in the world. Various vehicles used in land transportation include cars, trucks, buses etc.

Production of an automobile is one among the most complex marvels of engineering. Various parts of an automobile require perfection during manufacturing and high precision during assembly. While tires do tend to get overlooked, they are arguably the most important component on a vehicle. After all, tires are what allow the vehicles to roll down the road. The main functions of a vehicle's tires include supporting the vehicle load, transmitting traction and braking forces to the road surface, absorbing road shocks, and changing and maintaining the direction of travel.

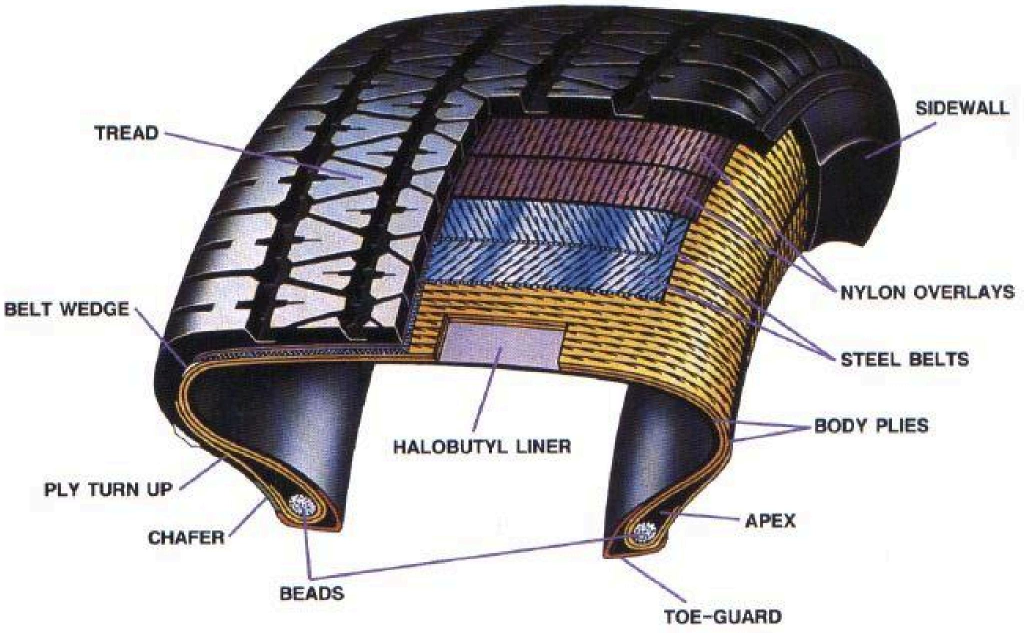
The modern process of Tire Manufacturing comprises of some of the most complicated and high level applications of polymer science and technology, specifically rubber processing.

A tire has many parts, each part having its own importance. A typical tire has the following parts:

1. Tread- It is the part of the tire which stays in contact with the ground. Tread is the most important part of a tire. The main functions of tread is to provide traction and improve mileage while the tire rolls. Tire tread has grooves which are designed to increase the effectiveness of the tread by providing better traction and preventing hydroplaning. Over the period of usage, the tread wears off.

   Tire tread consists of two parts namely tread cap and tread base. The tread cap remains in contact with the ground and has the grooves embarked on it. Tread base sits underneath the tread cap. A tread is made with high abrasion resistant and low heat build-up rubber compound.

2. Breaker- Breaker is a collective group of calendared fabric which holds the entire structural integrity of a tire. Its purpose is to distribute the load on the tire. It is meant to provide reinforcement to the section that's directly underneath the tread.



3. Bead- Tire beads hold the tire to the rim. They're made of copper or bronze plated high tensile steel wires wound into a rubber band. The copper or bronze plating helps in increasing the adhesion of the wires with rubber as adhesion of steel with rubber is poor. Tire beads prevent the tire from sliding out of place when the wheel rolls.
4. Bead filler- Bead filler is a rubber compound inside the beads. It provides stability to the lower sidewall and bead area.
5. Sidewalls- The area of a tire from the bead to the tread—the side of the tire—is called the sidewall. It forms a protective covering for the cord body. Information about the tire is printed on the sidewall. This information includes the tire size, load index, and speed rating.

   The purpose of sidewall is to provide flexibility to the tire. The section of tire in contact with the ground at a particular point of time experiences more stress than other parts. So when a tire rolls it undergoes continuous cycles of stress fluctuations called flexing. The sidewall rubber compounds thus must have high fatigue resistance to withstand flexing. Sidewall rubber compounds are also designed to resist damage from ozone, cuts and snags.

6. Body Plies- The body plies give the overall shape to the tire. It is like a skeleton and maintains the shape of the tire. Depending on the type and specifications, a number of body plies are used to make a tire.
7. Inner Liner- It is the inside part of a tire. It mostly remains in contact with tube or air inside the tire (in case of tubeless tires). It is made using impervious rubber.

In tire industries, formation of rubber with the desired properties is vital. To incorporate desired properties in rubber and to make it useable for further processing, it is mixed with various compounds in large internal mixers. The operation of bringing together all the ingredients required to mix a batch of rubber compound is called Compounding. Each component has a different mix of ingredients according to the properties required for that component.

Rubber and Need for Rubber processing

Rubber is a natural polymer and possesses elastic properties. It is manufactured from rubber latex which is a colloidal dispersion of rubber particles in water. This latex is obtained from the bark of rubber tree and is found in India, Srilanka, Indonesia, Malaysia and South America. It is then coagulated and dried to obtain natural rubber. Most of the industrial rubber is achieved from the tree called Hevea Brasiliensis.

A tire industry mainly uses block rubber (i.e. rubber in the form of blocks, each of about 35 kg) of various grades. Each grade of natural rubber differs in some or the other specifications like ash content, nitrogen content, wallace plasticity etc. The commercial naming of natural rubber blocks is mostly done based on the name of origin country and grade of the rubber. Various types of natural rubber used in tire manufacturing industries are

1. Natural Block Rubber Grade 10

   SIR 10 (Standard Indonesian Rubber Grade 10), SMR 10 (Standard Malaysian Rubber Grade 10), SVR 10 (Standard Vietnam Rubber Grade 10), STR 10 (Standard Thai Rubber Grade 10), SSR 10 (Specified Singapore Rubber Grade 10)

2. Natural Block Rubber Grade 20

   SIR 20 (Standard Indonesian Rubber Grade 20), SMR 20 (Standard Malaysian Rubber Grade 20), SVR 20 (Standard Vietnam Rubber Grade 20), STR 20 (Standard Thai Rubber Grade 20), SSR 20 (Specified Singapore Rubber Grade 20), ISNR 20 (Indian Standard Natural Rubber Grade 20)

Natural rubber may be considered as a linear polymer of isoprene (2-methyl-1, 3-butadiene) and is also called as cis - 1, 4 -polyisoprene. The cis-polyisoprene molecule consists of various chains held together by weak Van Der Waals interactions and has a coiled structure. Thus, it can be stretched like a spring and exhibits elastic properties.

Natural rubber (many times referred as NR) is non-resistant to oxidising agents in the atmosphere like oxygen and ozone. At lower temperatures (below 283K) is becomes brittle and shows high water absorption capacity. Under the action of heat, rubbers change their elastic or hyper-elastic state to a visco-elastic state and they become plastic enough to flow. Hence NR cannot be used directly to produce tires.

Synthetic rubber are artificially created vulcanisable elastomers. These are mainly produced from petroleum byproducts. Synthetic rubber monomers can be processed to impart various chemical, mechanical and physical properties to the artificial rubber polymer. For example Bromo-butyl rubber is used for the inner liner of tubeless tires because of it is impervious in nature. Other examples of synthetic rubber include SBR (Styrene Butadiene Rubber), Halo-Butyl Rubber, Polybutadiene rubber and Blended Halo-Butyl Rubber.

Rubber Processing

Rubber processing mainly involves mixing of rubber mass with other substances to impart various characteristics in the obtained rubber homogeneously. Hence the aim of mixing is to produce a rubber compound that has ingredients dispersed and distributed thoroughly with high level of consistency. High molecular weight means high viscosity, thus it is almost impossible to mix any powder or liquid ingredients into NR at its original viscosity. Before addition of any compounding ingredients, we need to soften the high molecular weight rubber, especially natural rubber, so that a homogeneous dispersion of all ingredients into the rubber matrix becomes possible. This process of softening of rubber by means of mechanical shearing is known as mastication and takes place in banburies or large internal mixers. Raw rubber processing is a batch mixing process i.e a specified amount of rubber (called a batch) is processed at a time.

The natural rubber requires controlled reduction in molecular weight. Masticated rubber is softer and flows more easily but over masticated rubber is tacky is nature. Kneading or softening of elastomer is useful in increasing its receptivity to incorporation of powders. This is the basis of mixing, to make it receptive to other ingredients, yet retaining sufficient stiffness to ensure adequate dispersion. This balance of mastication without undue shear softening can be achieved through means like- close mixing with temperature control, use of specific mixing sequence or in some cases, remixing after cooling.

Rubber Additives

The very first and important class of additives added to rubber is filler. Almost every filler compound added to the rubber firstly makes it stiff and secondly makes it cheap. As fillers make the rubber stiff, they are also called reinforcing agents. The most important filler added to rubber compound is carbon black. Carbon black consists of small spherical particles having diameters of only 10–100 nanometres and made up of concentric graphitic layers of carbon. Another important filler compound of same shape and size is silica. Due to low processability and poor dispersion rating of silica, carbon black is primarily used as reinforcing agent in rubber compounds.

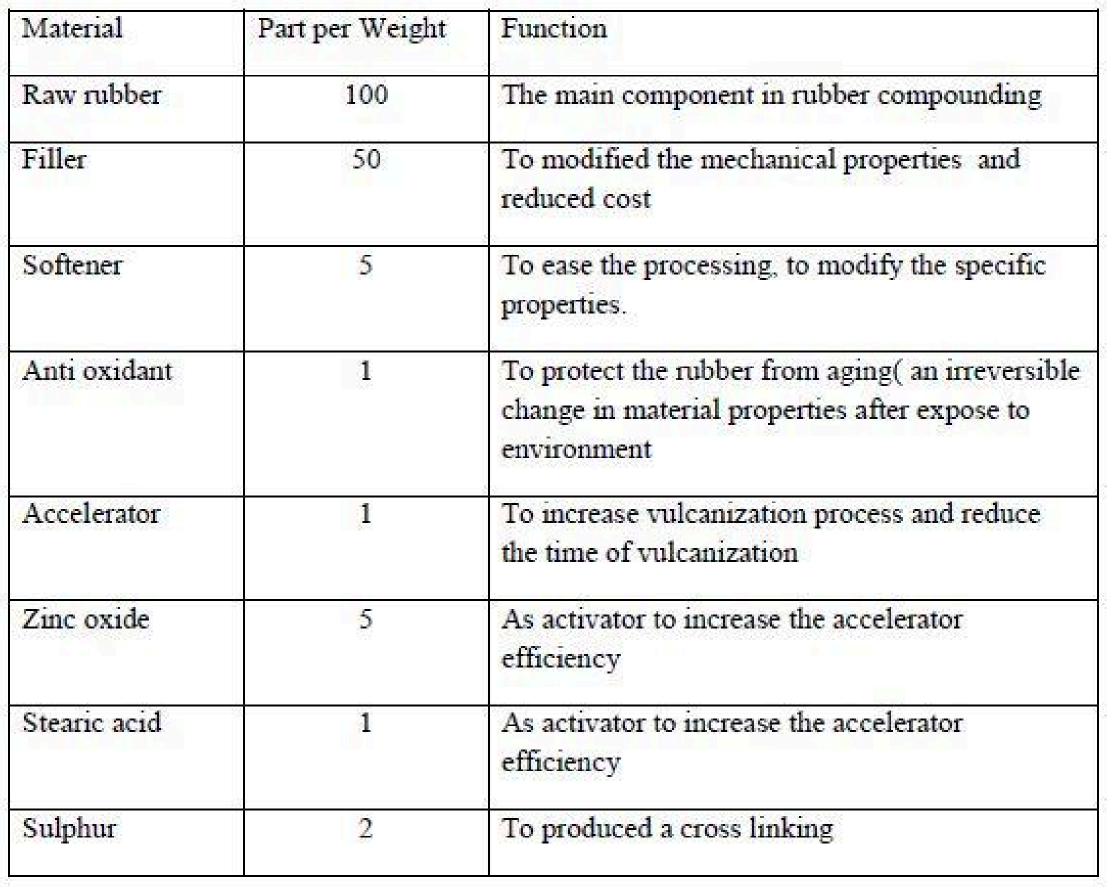
Fillers like carbon black and silica when mixed with rubber compound at a concentration of about 30 percent by volume, raises the elastic modulus of rubber by a factor of two or three. They also impart great toughness and abrasion resistance to weak materials like SBR. Addition of filler compounds increase the Mooney Viscosity, tear resistance and hysteresis. It lowers extrusion shrinkage and resilience.

The next class of additives is Plasticizers and Processing Aids. Processing aids and plasticizers are mixed with rubber compounds to increase plasticity and reduce viscosity. Plasticizers soften polymer compounds in order to improve processing and increase the flexibility. Processing aids can act as stabilizers, strengtheners and fusion promoters. Various process oils used in tire manufacturing include naphthanoic oil, aromatic oil and paraffinic oil. Thus plasticizers and process oils help in ensuring proper distribution of ingredients throughout the bulk of rubber compounds, hence increasing homogeneity. Plasticizers must have low vapour pressure and high boiling point inorder to be retained in the compound for longer periods of service.

Another class of additives imparts protective characteristics to the rubber compound. Most of the polymer reactions with aging or oxidising agents like oxygen, ozone etc are free radical reactions. Amines are powerful antioxidants. Added to rubber compounds in small amounts, they kind of disarray the free-radical oxidation reactions that lead either to molecular rupture and softening or to increased interlinking and hardening as rubber ages. Sequestering agents are added to block the action elemental metals like copper, manganese and iron (these metals are powerful catalysts of oxidation). Atmospheric ozone reacts readily with elastomers containing C=C double bonds, leading to rupture of molecules lying on the surface. As a result, small, deep fissures, termed ozone cracks, are formed if the rubber is stretched slightly. Antiozonants are added to the rubber compound. These antiozonant compounds bloom up to the surface of rubber and protect the rubber from ozone. Microcrystalline paraffin wax is one such compound added to rubber compound. It is incompatible with the elastomer and hence blooms up to the surface preventing it from oxidation.

The last but most important class of additive is the cure package. It causes the interlinking reactions to take place during vulcanisation commonly called curing. The curing package mainly consists of sulfur and one or more accelerators. The rubber-sulfur reaction is a slow and reversible reaction and hence these accelerators are needed to make the interlinking reactions faster. On vulcanisation, sulfur forms cross links at the reactive sites of double bonds and thus the rubber gets stiffened. In the manufacture of tyre rubber, 5% of sulphur is used as a crosslinking agent.

Another set of compounds included in the curing package is accelerator activator. Zinc oxide and stearic acid are commonly used activators. These compounds react together and with accelerators to form a zinc sulfurating compound, which in turn is the key intermediary in adding sulfur to a diene elastomer and creating sulfur interlinks. Inorder to avoid premature curing during compound mixing, retarders or pre-vulcanising inhibitor (PVI) also added in this long list of rubber compounding. These compounds react with the active accelerator fragment which slows down or temporarily prevents zinc salt formation. Thus it increases the time for curing to take place. Some common accelerators used in tire manufacturing includes MBTS (2-Mercaptobenzothiazole) and CBS (N-Cyclohexyl-2-benzothiazole sulfenamide).



Material	Part per Weight	Function
Raw rubber	100	The main component in rubber compounding
Filler	50	To modified the mechanical properties and reduced cost
Softener	5	To ease the processing, to modify the specific properties.
Anti oxidant	1	To protect the rubber from aging(an irreversible change in material properties after expose to environment
Accelerator	1	To increase vulcanization process and reduce the time of vulcanization
Zinc oxide	5	As activator to increase the accelerator efficiency
Stearic acid	1	As activator to increase the accelerator efficiency
Sulphur	2	To produced a cross linking

The Banbury Mixer

Banbury mixer is a batch based internal mixing unit. It is a tangential mixer. Two slightly spiralled rotors revolve side by side. The breakdown of the rubber is accomplished by the shearing action between the rotor blade tips and chamber walls and between the two rotor tips of the two rotors. While one rotor moves clockwise the other one moves counter-clockwise. The chamber has a top opening called the hooper door. The ram applies pressure from the top and ensures that the chamber is completely closed. The mixers have three thermo-couples namely (the name of the thermo-couples are based on their location inside the mixer) door top 1, door top 2 and end of frame. These thermo-couples constantly measure the temperature of the mixing mass. After completion of mixing, the drop door at the bottom of the mixer opens and compound is dropped into the dump mill.

Various functional parts of a banbury mixer

1. Rotors

   The rotors are shaped in the form of wings. Sometimes these wings are also called helical blades. The shape of these wings is important to get a smooth and predictable flow of mix in the mixing chamber. The way the materials get mixed determines the temperature rise in the mixing chamber. Another factor which influences mixing, especially distribution of filler compound inside the rubber matrix is rotor speed. Sometimes for certain stages of mixing even alignment of the rotors can be adjusted.

   The rotors are provided with extensive cooling systems as the temperature during the time of mixing may well reach upto 150 to 170 degree celsius. The cooling at the tip of the rotor wings is very important as here the temperature rise is more than other regions.

2. Sides

   The sides are provided with cooling channels so that whatever area comes in contact with the hot compound dissipates heat to reduce compound degradation or scorching.

3. Hooper Door

   It is pneumatically or hydraulically operated door which serves as the opening for charging in the raw materials into the banbury mixing chamber.

4. Drop door

   Usually operated by a linear hydraulic actuator, the drop door is mounted flexibly against the mixing chamber ensuring minimal material leakage and contamination. When the mixing batch is ready, the drop door opens to dump the batch.

5. Ram group

   The ram is usually hydraulically or pneumatically operated. The ram has sensors to indicate the position of the ram. The ram moves down to completely close the mixing chamber. It can apply pressure up to 100 bars.

Study of Banbury mixing procedure at Alliance Tires Company (A group company of Yokohama), Dahej

The raw materials are weighed on the Weighman Belt. The ATG tires company, has two types of banbury mixers. For preparation of master batch, the mixer with a capacity of 430 litres is used while for final batch preparation a mixer with a capacity of 310 litres is used. The effective available volume of the 430 litre mixer is about 402 litres while for the 310 litre mixer it is about 280 litres.

The batch weight of master batch is different from the batch weight of final batch. The batch weight calculation is based on effective available volume, specific gravity and fill factor.

Batch weight= (fill factor) * (specific gravity) * (effective available volume)

Fill factor is the percentage upto which the effective available volume of a banbury mixer is filled up to. The fill factor is generally kept between 0.7 to 0.85. A very high fill factor (close to 0.9 or above) will allow no space for the ingredients to mix whereas a fill factor too low will leave spaces and might not ensure proper and homogeneous mixing. The whole mixing mass should be moving during the period of mixing to ensure homogeneity. When a banbury mixer is newly set up the fill factor is kept low (about 0.73). As the banbury mixer becomes old, due to wear and tear the effective volume increases and the fill factor is increased accordingly. The fill factor is generally kept low for NR as it has very high viscosity.

The batch weight for master batch is about 350 kg while it is 230-240 kg for final. After the initiation of the start operation, the discharge door closes, the ram moves up and hooper door opens. Once the raw materials of correct weight is loaded on to the weighman belt, a green signal is received and the raw materials are charged into the banbury mixing chamber through the hooper door using the charging belt.

Once the compounds (rubber+master chemicals) are charged into the mixer, the hooper door closes. Desired amount of carbon black is charged into chamber from overhead bins, and the ram moves down. The mechanism of carbon weighing and discharging is completely computerised. The process of mastication begins and the rollers start breaking down the rubber blocks. Within ten seconds, the ram completely moves down. The mixing process continues for some time then the ram moves up and process oil is injected into the chamber. The ram again moves down and the mixing process continues.

A banbury mixer operates on three parameters namely time, temperature and energy. We can even switch among these modes. The mixing process will proceed to the next step if and only if the desired time, temperature and energy is reached for each step. Once the mixing process after injection of oil reaches the desired parameters of temperature, time and energy, the ram goes into float position, the drop door opens and the mixed mass is dumped into the dump mill.

From the dump mill, this mixed batch of rubber compound is milled into sheets of specific width and thickness then dipped in the dip tank. The dip tank contains 2.5 to 3 percent soap solution which reduces the tackiness of the rubber compound so that the sheets when laid down doesn’t stick and increase scrap. After dipping the sheets are passed on to the cooling unit which has cooling fans. For the 310 litres banbury setup there are 48 fans whereas for the 430 litres banbury setup there are 60 fans. After cooling the sheets are laid down in the lay down unit. This is how a master batch is produced.

The final batch is also produced on similar ways the difference being the chemicals to be added only consists of sulfur and accelerator. During final batch production, sheets of master batch are first charged in the banbury and masticated for some time. The hooper door opens again and this time only the chemicals and charged inside the mixer. The mixing starts again and when appropriate time, temperature and energy is reached, the batch is dumped.

Controllable factors at a Banbury

1. Batch Size
2. Sequence of addition
3. Ram cylinder pressure
4. Rotor speed
5. Mixing time
6. Temperature of the finished mix, chamber, rotors, discharge doors
7. Energy for mixing

Carbon Black

Carbon Black is a paracrystalline carbon. It has very high surface-area-to-volume ratio but less than that of activated carbon. Carbon black differs from soot in its much higher surface-area-to-volume ratio and significantly lower polycyclic aromatic hydrocarbon content. Carbon Black is mainly used as a reinforcing agent in tire manufacturing and as color pigment agent in plastics, paints, inks etc. All carbon blacks have chemisorbed oxygen complexes like carboxylic, quinonic, lactonic and phenolic groups on their surfaces to varying degrees based on the conditions of manufacture. The surface oxygen groups are generally referred to as volatile content. These groups increase the surface activity of the carbon black particles.

Carbon black has primary and secondary structures. At the same time that carbon black is formed in a high temperature region that is not completely burned, the particles are chemically combined to form a three-dimensional aggregate, that is, a primary structure of carbon black. Later when two or more such aggregates agglomerate into loose structures called the secondary structure of Carbon black, due to Van der Waals forces. Smaller the particle size of carbon black particles higher is the surface area of the aggregates and thus more is the filler rubber interaction.

Based on the method and conditions of production, there are various particle and aggregate sizes of carbon black. Based on these various sizes there are different grades of it. This series of Carbon Black grades with increasing particle size and decreasing surface area is called carbon black spectrum. In standard ways, the carbon black grades are named in the following syntax-

Carbon Black N XYY

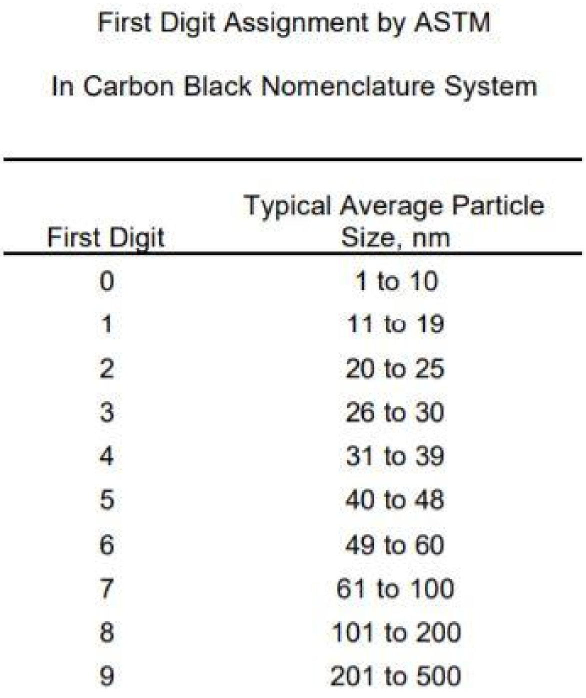
Where,

'N' stands for Normal cure.

'X' represents the First Digit which stands for particle size.

'Y' represents a number which stands for aggregate structure.

Along the carbon black spectrum, the carbon black with the largest particle size (N 900 series) and among the lowest degrees of particle aggregation or structure is called Thermal Black. While other carbon black grades (N 100 to N 700 series) are called Furnace blacks.



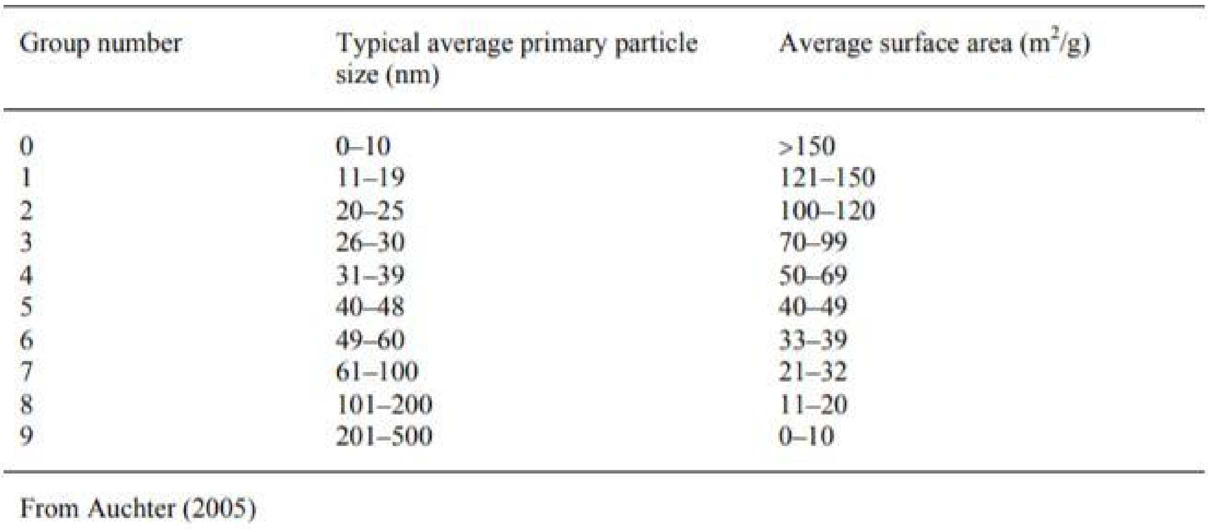
First Digit Assignment by ASTM

In Carbon Black Nomenclature System

First Digit	Typical Average Particle Size, nm
0	1 to 10
1	11 to 19
2	20 to 25
3	26 to 30
4	31 to 39
5	40 to 48
6	49 to 60
7	61 to 100
8	101 to 200
9	201 to 500

There are two methods in use for surface area determination. Iodine adsorption (expressed in mg/g of carbon) measures the amount of iodine which can be adsorbed on the surface of a given mass of carbon black. Nitrogen surface area is a measurement of the amount of nitrogen which can be adsorbed on a given mass of carbon black. Both tests have a good correlation for most rubber grades of furnace blacks, but toluene extractables on the surface of thermal blacks interfere with the aqueous iodine-potassium system of the iodine adsorption method, making nitrogen surface area a more suitable test. Accordingly, the American Society for Testing and Materials (ASTM) does not recommend iodine adsorption for surface area measurements of thermal blacks.

Measuring the structure or morphology of carbon black is a more difficult task. The current industry test index is the dibutyl phthalate absorption number (DBPA). Dibutyl phthalate (DBP) absorption measures the relative structure of carbon black by determining the amount of DBP a given mass of carbon black can absorb before reaching a specified viscous paste. Thermal blacks have the lowest DBPA numbers (32-47 ml/100g) of any carbon black, indicating very little particle aggregation or structure.



Group number	Typical average primary particle size (nm)	Average surface area (m^2/g)
0	0–10	>150
1	11–19	121–150
2	20–25	100–120
3	26–30	70–99
4	31–39	50–69
5	40–48	40–49
6	49–60	33–39
7	61–100	21–32
8	101–200	11–20
9	201–500	0–10

From Auchter (2005)

Importance of Carbon filler Dispersion in Rubber Compound

Better dispersion of filler component ensures higher homogeneity of the mixed rubber compound. Higher homogeneity further ensures uniform physical, mechanical and chemical properties throughout the bulk of the mixed compound. Thus it increases the processability of processed rubber. Eg. If the carbon dispersion of sidewall compound of a tire is not satisfactory (by industrial standards), then fatigue resistance of the sidewall will decrease. Portions where carbon is deficient will be more prone to rupture. Carbon particles within the rubber mass also supports heat distribution. So a sample with low carbon distribution will cause uneven curing during vulcanization.

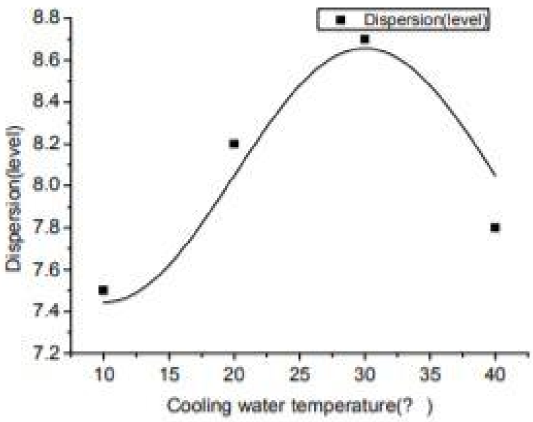
Properties of Carbon Black affecting Filler dispersion

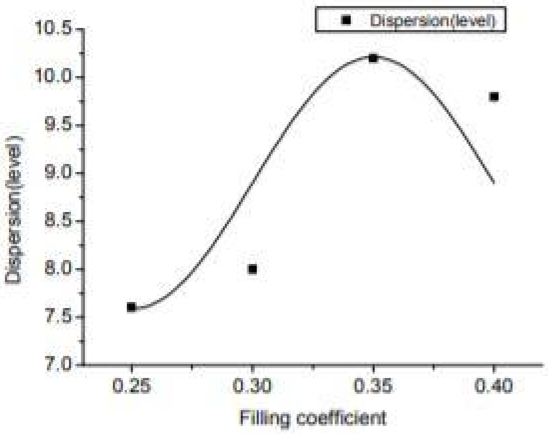
Particle size of carbon black plays an important role in its dispersion throughout the rubber mass. The fillers with smaller diameter have greater surface area of aggregates and thus results in greater rubber-filler interaction, thus improving dispersion. The hardness of the pellet (secondary structure) plays a very vital role in improving carbon dispersion. Pellets of Carbon black are first broken down in the banbury and then incorporated with rubber mass. Higher the pellet hardness, lower is the dispersion.

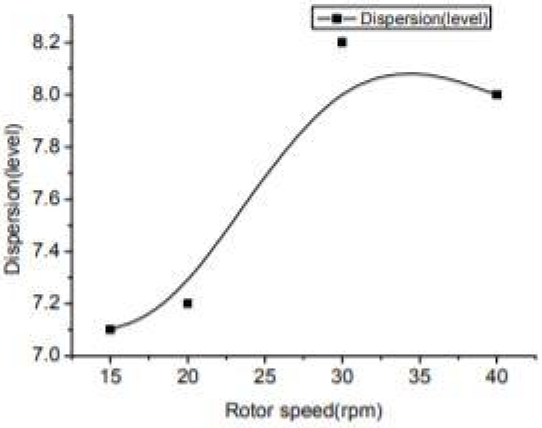
The filler type is also vital. Natural Rubber is non-polar and thus it is easy to disperse non-polar fillers in the rubber compound. Thus it is difficult to disperse silica when compared with carbon black. Chemical compatibility between the fillers and rubber plays an important role in the dispersion of fillers and in the strong adhesion bonding between the fillers and rubber. The adhesion between the rubber and filler may also be induced by a coupling agent.

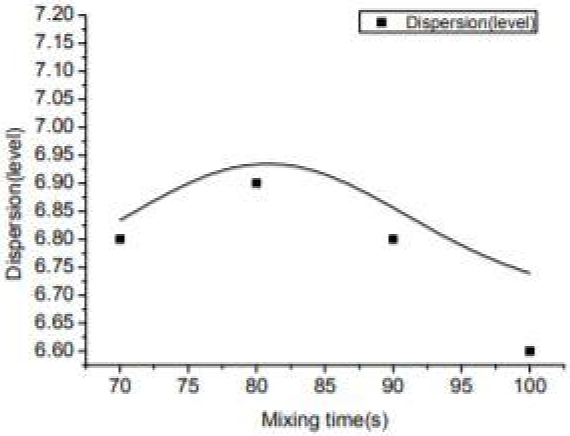
Various Parameters of Banbury Mixer affecting filler dispersion

Parameters like cooling water temperature, rotor speed, mixing time and fill factor have a direct effect on the filler dispersion. A better supply of cooling water will ensure effective cooling and results in better mixing. The optimum cooling water supply temperature is 303K. Slower the rotor speed slower is the heat build up (which allows extended mixing time) but a rotor speed which is too low will increase the batch production time and result in poor mixing. Ideally the rotor speed should be very high during the first few seconds of mixing and then it should gradually decrease. The effect of fill factor on mixing is already mentioned above. Mixing time directly affects the dispersion. Increased mixing time ensures better dispersion.









Important note- These graphs only represent general trends.

General Problem with Carbon Dispersion

In order to achieve the full potential of any rubber compound a high degree of dispersion of the ingredients in the polymer matrix must be realized. This sounds easy but it is not always so, especially when mixing time is important.

With carbon black there are three key properties that significantly influence dispersibility: particle size, structure and pellet hardness. The degree of difficulty in achieving suitable dispersion becomes worse as both particle size and structure are decreased. In fact, exceedingly poor dispersibility has prevented the commercialization of low structure carbon blacks even though the properties they could provide would otherwise assure a market for such a grade.

For any carbon black grade and polymer system a certain minimum batch viscosity must be achieved for a given length of time in the mixer to obtain a given level of dispersion. Trade-offs between time and viscosity are possible. Increasing levels in these two factors can be expected requirements as particle size and structure decrease if adequate dispersion is to be maintained. For example, with the thermal blacks, which have uniquely large particles, very good dispersion is readily attained in nearly any compound. In contrast, reaching the same dispersion level with N110, which has very fine particles, can be very difficult and may require as many as three or more passes through the mixer. But again, increasing the number of levels reduces the productivity.

To achieve dispersion of the very small particle size carbon blacks, a high compound viscosity is required which must be maintained for a relatively long period of time. Since these stiff batches generate heat at prodigious rates, the necessity of dumping such batches before the necessary work input has been achieved is often a fact of life. The only solution is generally multiple passes with adequate cooling and aging between. In any event, it is possible that the only point in the mixing cycle in which the viscosity is sufficient enough to gain further dispersion is at the initial breakdown of the cold batches.

As previously mentioned, pellet hardness can contribute to poor mixing and dispersion. Since the economics of mass production dictates that most carbon black consumers handle the material in bulk, pelletizing is a necessity. In addition, it is common practice to add binder to the pellets of many grades in order to assure that the pellets are hard enough to handle well in existing bulk handling equipment. The more tightly the pellets are knitted together the better they handle and the more likely they are to mix poorly.

Measurement of Carbon Dispersion in a Mixed Rubber Compound

The measurement of carbon dispersion in the cured sample is measured in Process Testing Lab using Carbon Dispersegrader. It is a device which compares the picture of mixed rubber compound sample surface with 100 magnification with a standard picture. The standard picture basically represents one hundred percent dispersion. More similar the sample surface picture is with the standard picture, higher is the dispersion of carbon in the sample. Based on this percentage similarity, the Carbon Dispersegrader gives a carbon dispersion rating on a scale of 1 to 10.

Overview

"TBR Tyre" stands for "Truck Bus Radial Tyre". While "Truck Bus" refers to the vehicles for which these tyres are made for, "Radial" is the tyre manufacturing technology used for making the tyre. TBR tyres are very high speed tyres and thus the sidewall should have very high fatigue resistance and low heat build up. This makes carbon dispersion vital in the TBR sidewall compound.

Currently the compound used in Alliance Tires Company (a group company of Yokohama), Dahej plant for TBR sidewall is a blend of Natural Rubber Grade 20 and Polybutadiene (Neodymium) rubber in equal proportions. NR Garde 20 and PBR (Neodymium) along with other master compound chemicals like activators (zinc oxide and stearic acid), protective compounds (TMQ, Koresin, 6PPD, MC wax), peptizer and tactifier (to increase tackiness), are charged inside the banbury with 50 phr Carbon Black N-330. The ram pressure was 14 megapascals. After a mastication of about one minute when the temperature reached 410K, Aromatic Low PCA oil was injected in prescribed amounts. The mixing continues for another one to two minutes and when the temperature reaches 423K, the batch was dumped. The batch weight is 350 kgs. This master batch was cooled and then mixed again with curing package to produce final batch of batch weight 240 kg.

Now we will try to improve the Carbon dispersion rating of this sidewall compound by altering mixing parameters and sequence.

Goals

To improve Carbon dispersion rating of Truck-Bus Radial Sidewall Compound by modifying mixing sequence and banbury parameters.

Steps Taken

1. **Selective rubber carbon mixing in place of blend mixing:** The carbon loadability of synthetic rubber PBR is better than NR. Hence when NR and PBR are simultaneously mixed with carbon black in the mixer, carbon incorporation first takes place in PBR and then in NR. This further reduces the mixing time for NR polymer and carbon reducing the dispersion. So here in first step we will be mixing only NR with 20 phr carbon black. It will ensure better incorporation, distribution and dispersion of carbon black in the NR polymer compound. In the next stage this premixed natural rubber compound will be processed with PBR (Neodymium) and other chemicals. With the introduction of this new stage, the mixing time will also increase.
2. **Premastication of NR before charging in Carbon:** NR when premasticated becomes sticky. When carbon will be introduced in this sticky rubber matrix, it is expected to result in better dispersion.

3. **Increasing rotor speed:** Increasing rotor speed will help in increasing the shearing forces and also help in better breakdown of pellets. But a high rotor speed increases heat buildup.
4. **Decreasing batch weight:** It will result in lesser heat buildup and increased mixing space.
5. **Decreasing ram pressure:** It will also result in lower heat buildup and thus provides scope for extended mixing time with higher rotor speed.

New specifications

Master Batch 1

Raw materials - NR Grade 20, Carbon N330, Aromatic Low PCA Oil, Plasticizer

Mixing sequence

Only NR Grade 20 rubber will be charged into the banbury mixing chamber and masticated for 25 seconds with rotor speed of 55 rpm. It will lower the molecular weight of NR polymer and make it sticky for better incorporation of Carbon Black. Then 20 phr Carbon Black N330 will be charged in the chamber and mixed with 45 rpm rotor speed along with a temperature and time control of 408K and 35 seconds. Oil injection takes place (it means ram will moved up after one minute of mixing) at 408K and then mixing continues for further 50 seconds. For the entire process the ram pressure will be 12 megapascals. The batch weight is reduced to 330 kg.

Master Batch 2

Raw materials - Masterbatch 1, PBR (Neodymium), Carbon Black N330, Zinc Oxide, Stearic Acid, Tackifier Resin, 6PPD, Koresin, MC wax, 77PD and TMQ

Mixing Sequence

Masterbatch 1, PBR (Neodymium) and other master compound chemicals will be charged into the Banbury mixing chamber and masticated for 15 seconds at 45 rpm rotor speed. The remaining 30 phr Carbon N330 will then be charged and this combined mass will be mixed with 45 rpm rotor speed in temperature and time control of 403K and 30 seconds respectively. This will be followed by injection of oil and 77PD. The mixing continues till temperature hits 423K and the batch is dumped. The batch weight and ram pressure are reduced to 330 kg and 13 megapascals.

Final Batch

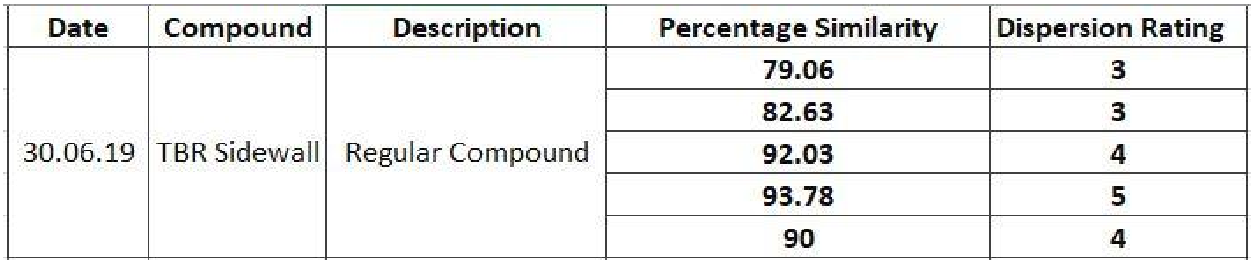
Raw materials - Masterbatch 2, sulfur, CBS, PVI, Insoluble Sulfur 33, 6PPD, Zinc Oxide

Mixing Sequence

Masterbatch2, 6PPD and zinc oxide will be charged into the banbury mixing chamber and masticated for 15 seconds with 30 rpm rotor speed. It will be followed by the charging of the rest of the chemicals. It takes place for about 75 to 90 seconds till temperature hits 378K and batch will be dumped. The ram pressure remains 14 megapascals but batch weight is reduced to 225 kg.

Results

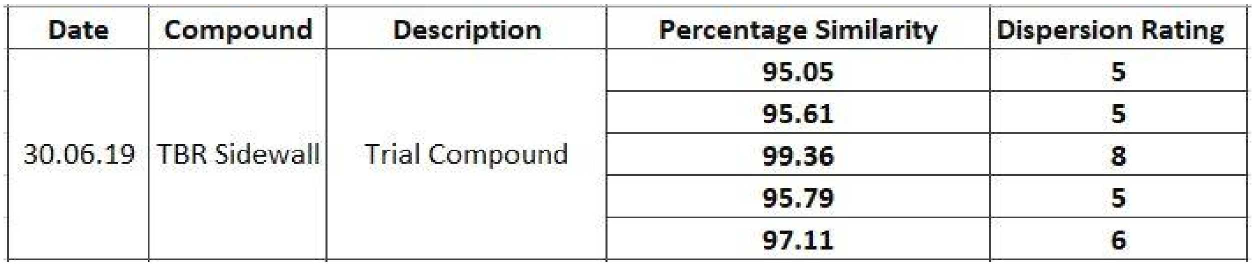
The regular TBR sidewall compound produced in this plant has an average dispersion rating varying from 3 to 4.



Date	Compound	Description	Percentage Similarity	Dispersion Rating
30.06.19	TBR Sidewall	Regular Compound	79.06	3
			82.63	3
			92.03	4
			93.78	5
			90	4

Average - 3.8

With the new specifications the sample produced had an average dispersion rating of 5.8.



Date	Compound	Description	Percentage Similarity	Dispersion Rating
30.06.19	TBR Sidewall	Trial Compound	95.05	5
			95.61	5
			99.36	8
			95.79	5
			97.11	6

Average - 5.8

This indicates improvement in carbon dispersion.

Date	Compound	Description	Chracteristic	Average	Standard Deviation	Target value
30.6.19	TBR Sidewal Compound	Regular	Thickness	0.21 cm	0.01 cm	-
			Hardness	54	0.01	56
			Tensile Strength	205.25 kg/cm sq.	19.91 kg/cm sq.	195 kg/cm sq.
30.6.19	TBR Sidewal Compound	Trial	Thickness	0.19 cm	0.01 cm	-
			Hardness	54	0.01	56
			Tensile Strength	202.83 kg/cm sq.	1.43 kg/cm sq.	195 kg/cm sq.

We also tested the tensile properties of the newly made compound and compared with the regular compound. It showed that, with better carbon dispersion the standard deviation decreased.

References

1. Rubber Chemistry- Matador Rubber S.R.O
2. NCERT Class 12 Chemistry Part 2
3. A Review On Rubber Compound Mixing In Banbury Mixer At Tire Industry by Sunny Shokeen (December 2015 | IJIRT | Volume 2 Issue 7 | ISSN: 2349-6002)
4. Rubber Lecture by Dr. Qahtan. Adnan
5. Rubber Grade Carbon Blacks by D. T. Norman
6. Carbon black dispersion in rubber assessment methods and process studies (Loughborough University Institutional Repository)
7. The Effect Of Filler Loading On The Tensile Strength Of Natural Rubber Compound by JUAN ANAK TUGAU
8. Method Of Improving Carbon Black Dispersion In Rubber Composition (Patent No.: US 7.964,656 B2 | Date of Patent: Jun. 21, 2011)
9. Effects of Different Process Parameters on Rubber Mixing in Tandem Internal Mixer by Chuansheng Wang, Meng Zhang
10. Chemistry of Rubber Processing and Disposal by Robert L. Bebb