Guidelines for Internal Mixing of NORDEL™ MG Hydrocarbon Rubber

The mixing of NORDEL™ MG Hydrocarbon Rubber in granular form is quite easy, though different from that of ethylene propylene diene terpolymers (EPDM) in the bale or pellet forms with which rubber compounders are more familiar.

NORDEL MG Hydrocarbon Rubber has a distinct granular form and has the capability to give very good dispersion after very short mixing cycles. As a consequence of its carbon black-coated small particulate form and high surface area, the product has consistently been found to offer more rapid mixing than other EPDM types. Its mixing behavior is characterized by a very rapid pick-up of torque with short black incorporation time (BIT), which can be used to increase mixer room productivity. Customers should regard NORDEL MG products as new polymers and the mixing cycle should be specially redesigned for the product form and formulation.

Successful mixing of compounds made with NORDEL MG, as for all elastomers, is dependent on good and consistent procedures, and knowledge of the individual characteristics of the polymer. This guide discusses how to improve the mixing time, mixing cycle, mixer fill factor, and mixer speed to get the maximum value from the product. It also describes how to make compounds with very low hardness, and how to prevent black scorch. In addition, some ideas for troubleshooting are included.

Internal Mixing

Elastomer mixing can be modeled as occurring in three stages.

1. Incorporation (Distributive Mixing)

In the incorporation stage, the rubber and other ingredients wet together. The surface area of the elastomer is increased through repeated deformation, trapping the filler inside the rubber. The filler is often still in agglomerate form.

With NORDEL MG, the granular form of the product already has a large surface area and the amount of work and time to increase the surface area is decreased, also decreasing the temperature after this stage. This temperature difference is the key to the observation that NORDEL MG Hydrocarbon Rubber often enables a compound to move from two-pass mixing to a single pass, because the lower temperature rise allows mixing to be complete before the temperature rises above a safe limit with regards to scorch safety. Since no energy is required for bale breakdown, the specific mixing energy needed to achieve a good mix with the granular product is lower than that needed for a good mix from a bale product.

2. Dispersion (Dispersive Mixing)

Dispersion is the process of breaking the filler agglomerates down to their aggregates or primary particles. During this process, the rubber becomes bound to the surface and into the structure of the filler. This is especially critical for reinforcing fillers such as carbon black or silica. A sufficient level of shear stress must be imparted via shear and elongational strain fields in the mixer to break down the particles, and insufficient shear and elongational stress will result in poor dispersion. Likewise, if the mixer fill is too low, the stress level will be insufficient to produce good dispersion.

Summary

The mixing of compounds made with NORDEL™ MG Hydrocarbon Rubber requires a less complex mixing cycle than that needed for elastomers with other physical forms. The key points to remember are:

- Use an “all-in-together” mixing technique to make very good use of the high surface area of the particles.
- Modify the load factor (mixer fill) to maintain sufficient ram pressure.
- Decrease cycle time in a stepwise process to take advantage of the shorter BIT. Mix to time rather than to temperature in most cases.
- Lower dump temperatures can permit change from two- to one-pass mixing.
- Use split oil addition for very soft compounds.
- Heat oil prior to adding to increase oil incorporation rate.
- Always take steps to avoid black scorch.
- Keep the mixer and surrounding area clean to avoid quality and safety issues.
After the incorporation and dispersion stages are complete, the mixer torque passes through a maximum and usually decreases. The torque maximum is called the black incorporation time (BIT). It has been observed that granular form NORDEL™ MG Hydrocarbon Rubber has a black incorporation time 20-40 seconds faster than bale-form EPDM of similar crystallinity and viscosity. Generally, this time can be saved from the mixing cycle, and it will often be found that a good mix is obtained at lower specific energy because the granular form of NORDEL MG products obviates the need for bale breakdown at the start of the mix.

3. Homogenization

During this phase, the batch is homogenized and the curatives and other components are evenly distributed through the material. The time required for this phase is determined by the total shearing deformation that takes place; so it is a function of the rotor design, the rotor speed, and the time, assuming that sufficient free volume is available. The homogenization phase can be accelerated by increasing the mixer speed. If the mixer fill is too high, and the free volume is too low to allow creation of new surface area, then the homogenization phase will be imperfect.

In Figure 1a, the following processes are shown:
1. Fragment Rubber
2. Distribute Powders and Liquids
3. Incorporate Powders and Liquids
4. Fuse Fragments
5. Break Down Carbon Black
6. Disperse Carbon Black
7. Break Down Rubber and Black-Polymer Interaction
8. Shape Compound

Note: Step 1 is not required for the granular product.

Figure 1b shows how the viscosity of the compound changes during the mixing cycle. Improved quality of dispersion decreases viscosity, and the temperature increases, so that the viscosity decreases as the compound is further mixed (excluding any effects of scorch at extreme mixing times). Generally, a lower compound viscosity testifies to a better mix, and the viscosity of the mix is an important quality control measure.

Nakajima estimates that the energy required to reduce the rubber in size is 36 percent of the total energy required to produce an efficient mix. Starting from a granule such as NORDEL MG eliminates a significant proportion of this energy. The granule will still be required to be reduced to micron size, by 2-3 orders of magnitude in length from the primary particle of the granulate, but the primary particles of NORDEL MG are themselves already 2-3 orders of magnitude in length smaller than a bale. In other papers, Nakajima describes the accepted mechanisms for carbon black incorporation and dispersion. Summaries of mixing are also contained in references 7 and 8 (page 6).
NORDEL™ MG Hydrocarbon Rubber Morphology

The particles of NORDEL™ MG Hydrocarbon Rubber have a structure with primary particles that sometimes coalesce during the process to form agglomerates. The photomicrograph in Figure 2 shows that the surface of the particles is a porous coating of carbon black, and that the surface area is very high.

It has been found that oil diffuses readily into the surface of the particles, with the rate of diffusion dependent on the oil temperature – hotter oil induces faster swelling of the particles. This illustrates a key principle for the mixing of NORDEL MG products: the surface area can be used to accelerate oil uptake and mixing. Once the surface area has been decreased by fluxing the polymer, the rate of oil uptake will decrease.

The Dow Chemical Company’s (Dow’s) technical service and development (TS&D) experience has been distilled into a practical mixing guide based on both scientific principles and empirical trials at customers.

Practical Mixing Guide

The internal mixer is the preferred mixing method for NORDEL MG Hydrocarbon Rubber. In fact, it is the only practical method for highly-extended ethylene-propylene rubber compounds. A variation of a single-pass, “upside-down” technique is suggested for compounding NORDEL MG products. Several variations based on the order of ingredient addition exist, but the principle remains that the simplest and most effective procedure is to put all the ingredients into the mixer before lowering the ram.

Figure 2: Photomicrograph through a particle of NORDEL MG Hydrocarbon Rubber showing the core of rubber (white) surrounded by a shell of carbon black.

Mixing Cycle

The granular form of NORDEL™ MG Hydrocarbon Rubber offers wide latitude in mixing procedures, as long as the high surface area of the granular form is used to accelerate absorption of the oil. Mixing can be accomplished via three general methods: single-pass, two-pass, and modified single-pass mixing.

In single-pass mixing, the fillers, oil, process aids, and curatives are added to the polymer in one pass, so that the compound that exits the mixer is ready for use. Single-pass mixes can be used if the mixing cycle can impart sufficient shear energy to the mix to obtain an acceptable dispersion, without the temperature exceeding a safe limit for the curatives and optional blowing agents.

In two-pass mixing, all ingredients except the curatives and optional blowing agents are added in the first pass and often mixed to a high temperature, above what would allow safe addition of the curatives or blowing agents. The mix is dumped, cooled, and rested, before being added to the mixer again cold. The mix is then masticated, heated, and the remaining ingredients added. Two-pass mixes are used when the temperature reached after the first pass is too high for addition of the cure components or blowing agents. It is used for sponge compounds, and also for silica-containing compounds because the silica grafting reactions require temperatures of about 150°C (300°F) for completion. It is debatable whether two-pass compounds offer superior dispersion: the argument in favor is that the second plastification stage from cold imparts high shear stress on the compound and can disperse the filler particles better; whereas the argument against is that a discernable effect is rarely seen.

Two-pass mixing is also sometimes used to transport compounds long distances. The shelf-life of sulphur-vulcanized compounds is from a few days to 2-3 weeks, depending on the cure system, after which the compound viscosity increases significantly. If a compound cannot be used immediately (e.g., due to transport between continents or when refrigerated transport is not practical), the compound is transported without cure package, which is added just prior to use.

Modified single-pass mixes can be used to add ingredients in several steps without the compound being dumped from the mixer. These are used with NORDEL MG Hydrocarbon Rubber to add high levels of oil to soft compounds, or if curatives are added to the mix at the end.
Standard mixing techniques flux (plastify) the polymer before adding the other components. This does not work well with the granular nature of NORDEL™ MG Hydrocarbon Rubber because fluxing the polymer destroys the high surface area of the granules and significantly slows down absorption of the oil. In addition, the high filler levels often used with NORDEL MG favor the use of upside-down-style mixes. The true upside-down technique – where the polymer is added after the fillers, oil, and curatives – was developed for mixing EPDM compounds with low relative volumes of polymer.

Suggested Procedure for NORDEL™ MG Products

A variation of upside-down mixing known as the all-in-together technique should be used with NORDEL MG products. The polymer is added to the mixer with the fillers, followed rapidly by the oil and curatives. This allows the oil to be taken up by the polymer while the high surface area of the granules still exists. A typical scheme is shown in Figure 3.

The suggested loading procedure is carbon black first, followed by the polymer, other ingredients, and then to start injecting the oil just before or as the ram is lowered. A slow rotor speed and a moderate ram pressure are suggested, particularly for first trials. Variations in loading order can be used, and it is usually found that the actual addition order is not critical as long as the general principles are observed.

Figure 3: Schematic of All-in-Together Mixing

Note: It is critically important to add as much of the oil as possible into the mix while the NORDEL MG is still in granular form and has not begun to flux. The porous granular form can absorb oil very fast, and the oil should be added while the elastomer still has a high surface area. Adding the oil to the mixer at an elevated temperature of 100 to 120°C (210 to 250°F) will further increase the oil incorporation rate (consult the oil MSDS for additional information). If the oil is added too late, after the polymer has begun to flux, the surface area will be reduced and oil uptake slowed down.

Where the scorch time is short, the curative ingredients are added at the sweep stage, or at around 90 to 100°C (190 to 210°F). The maximum dump temperature for one-pass mixing should be between 115 and 125°C (240 and 260°F). Typical cycle times for compounds made with NORDEL MG products are between 2.5 and 3.5 minutes.

It has occasionally been found that compounds made with NORDEL™ MG Hydrocarbon Rubber require torque too high for some older mixers with limited motor power. In these cases, raise the ram slightly to decrease the rate of work, or decrease the mixer fill slightly.

Mixing Time

It is suggested that mixing cycles of compounds made with NORDEL MG products are maintained at conventional times at first, and then shortened as experience is gained in mixing technique. This is because the high load factors, particularly of the very high viscosity products, and very short mixing cycles involved can lead to poor dispersive mixing. Experience shows that mixing cycle times can often be shortened by around 30 seconds compared to mixing baled polymers. Obviously, this can represent a significant increase in mixing room productivity.

Load Factor

The use of the appropriate load factor is one of the most important elements in mixing, and the correct load factor can improve compound quality and mixing room productivity. The actual factor will depend on many variables, including the compound, the individual mixer and its state of wear, the rotor speed, and the ram pressure. It is essential, therefore, to adjust the loading on the actual mixer to be used, by watching the ram position during mixing. During the first stage of mixing, the ram under high pressure should be “riding,” lifting and falling in a
very distinct motion, and should not “bottom” on the metal stops. An ideal loading will have the ram bottom after the BIT peak is reached. An underloaded mixer will bottom immediately and, even with some slight lifting of the ram, is likely to give sub-optimum dispersion. In an overloaded mixer, there will not be enough free volume for effective mixing. An overloaded mixer will never have the ram bottom and will leave unmixed material in the throat of the mixer.

Load factors used are generally in excess of 70 percent and may be as high as 90 percent for very soft compounds. While good results can be obtained with both tangential and intermeshing mixers, it is important to keep in mind that:

- Typical load factors are higher for intermeshing machines than tangential machines.
- Tangential mixers are more sensitive to mixer loading, but finding the right load factor is still important for machines with intermeshing rotors.

It is almost always the case that the load factor must be higher for compounds made with NORDEL™ MG Hydrocarbon Rubber than for other polymer types. This has been confirmed in numerous trials at Dow, at customers, and at machine manufacturers. If NORDEL MG is used in a mix adjusted for bales, the chamber will be under-filled and the ram pressure will be too low. Therefore, the mixer load is typically required to be +3 to +5 percent compared to bale-form product.

Medium ram pressures and low to medium rotor speeds are preferred. Very high ram pressures should be avoided on soft compounds, because they can pack too much compound into the chamber, thereby severely limiting the free volume and the rate of distributive mixing.

**Mixing Temperatures**

**Product Temperature**

Conventional baled EPDM needs to be warmed prior to mixing, or the incorporation time is very long and the homogeneity very poor because the material will crystallize at low temperatures. Semi-crystalline products often need to be kept in hot room for an extended period of time before mixing during winter months in cold climates. One of the advantages of the granular form of NORDEL MG products is that, unlike baled EPDM, even the semi-crystalline grades do not need to be warmed because the high surface area allows for rapid heat transfer.

**Mixer Temperature**

It is suggested that the mixer temperatures (body and rotor) be set at 60 to 70°C (140 to 160°F) for semi-crystalline NORDEL MG grades, as these temperatures will help the melting of the crystallites in the polymer and speed up the fusing of the particulates. This shortens the mixing time, especially during the cold winter months.

The speed of pick-up at the beginning of the cycle is affected by the mixer body temperature. Particularly for semi-crystalline grades, if the body temperature is too low, the time to initial torque pick-up can be long. Mixing a warm-up batch at the beginning of a shift is suggested to get the mixer to temperature.

**Mixer Speed**

The ease of mixing NORDEL MG Hydrocarbon Rubber means that good results have been obtained at both high and low mixer speeds.

If the mixer has a variable speed, it is possible to increase the total energy input by mixing at low speed until the BIT is reached (e.g., for the first 45 seconds) and then increasing the speed. This is because polymers are non-Newtonian fluids, with high viscosity at low shear rates and low viscosity at high shear rates. In the first part of the mix, a low speed means a lower shear rate and, therefore, higher viscosity and greater energy input to the batch. Later in the cycle, a higher speed can be used to disperse the ingredients faster through the batch.

**Low Hardness Compounds**

The mixing of low hardness compounds, in the region of 40 to 50 Durometer A, with the addition of high volumes of oil require alternate mixing cycles in order to get good dispersion. A modified single-pass technique typically works well, where the upside-down mixing technique is modified to one in which part of the oil (e.g., 1/2) and carbon black (e.g., 1/3) are withheld from the first stage and added at a second stage. This technique is described by Hess, et al. In effect, the technique is to mix a harder compound to a good dispersion level and then add oil to soften it. In the first part of the cycle, the load factor is lower than in the second part, but because harder compounds generally require lower load factors than softer compounds, the load factor is appropriate for both parts of the cycle. The reason some of the filler is added with
the second shot of oil is to add friction between the batch and the oil, so that the batch will not “go to sleep” too long when the second shot of oil is added. Mixing is carried out as in a normal upside-down mix, with the loading adjusted to produce good mixing in the first stage, containing, for example, only half the oil and two-thirds of the carbon black. The ram should work normally in this mixing stage. At or just before the BIT is reached, the remainder of the oil and carbon black should be added. Mixing will temporarily stop as the oil lubricates the compound. This is seen as a drop in power consumption and temperature (“sleep”). Do not lower the ram until the mixer starts to pick up again. In some cases, a temporary reduction in mixer speed can improve the oil pick-up at this stage.

Around the BIT, the compound has a relatively low density (seen from the high position of the ram), and the compound is therefore still porous and able to absorb oil. Oil will be absorbed into the hot compound very quickly and within a minute the ram can be lowered, the mixer speed increased, and the mixing completed. Soft compounds normally need much higher load factors than harder compounds, so the mixer is not overloaded in the second part of the cycle.

Pick-up time at this second stage can be speeded up by adding filler with the oil. The use of carbon black, calcium carbonate, and/or diatomaceous earth has been found to work well for this purpose.

**Black Scorch**

High viscosity polymers containing medium or high ENB levels used with high levels of high structure black are vulnerable to black scorch. Simple one-pass, upside-down mixes are one of the easiest ways to avoid this problem. However, when adding the curative later in the mix – such as in the masterbatch of a two-pass mixed compound or with peroxide compounds – black scorch is a common problem, particularly when using more than 150 phr of carbon black. It is suggested that 0.2 phr of sulphur be added at the first stage of mixing to counteract the effect.

**Adjustment of the Carbon Black Level**

NORDEL™ MG Hydrocarbon Rubber particles have a core of EPDM polymer with a coated shell of carbon black as a partitioning agent. The carbon black is a general-purpose furnace black (ASTM N-650), currently at a nominal level of 24 phr (or 19.4 wt%). As a result, final formulations need to be adjusted for the level of carbon black already in NORDEL MG products.

**Cleanliness**

Due to the granular form of NORDEL MG products, extra care must be taken with cleanliness around the mixer environment. The high viscosity forms in particular will not disperse if accidentally added to the final mix. It is therefore very important to ensure that the areas below and around the drop door and above the ram are kept free of the particulate material. If particles are picked up in the final mix or if the granular polymer from the next batch falls on the earlier one on the mill, it can appear as a bad surface with undispersed material in the finished product.

**Handling of NORDEL™ MG Hydrocarbon Rubber**

The handling of NORDEL MG products is discussed in the NORDEL Handling Guide available from The Dow Chemical Company.

**References:**

Increase mixer body temperature if the temperature is variable.
Run a warm-up batch at the beginning of a shift to get mixer to temperature.

### Other Issues

**Table 1: Troubleshooting Guide for NORDEL™ MG Hydrocarbon Rubber**

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<thead>
<tr>
<th>Symptom</th>
<th>Cause</th>
<th>Solution</th>
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</thead>
<tbody>
<tr>
<td><strong>Inadequate Dispersion or Distribution</strong></td>
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<td></td>
</tr>
<tr>
<td>Poor surface</td>
<td>Insufficient work input or mixing time.</td>
<td>Typical fixes include: • Adjust mixer fill to increase ram pressure • Change rotor speed – make slow at start, and/or faster later in mix • Change order of ingredient input</td>
</tr>
<tr>
<td>Poor surface</td>
<td>Work input reduced because of excessive wear of rotors or chamber wall.</td>
<td>Adjust (increase) mixer fill and/or refurbish mixer.</td>
</tr>
<tr>
<td>Poor surface or variable part properties</td>
<td>Black-containing defects under microscope.</td>
<td>Material is hanging up on mixer door, on ram, or in crevices in mixer. Keep mixer area free of dust and granulate. Mixer is overfilled, reduce mixer fill.</td>
</tr>
<tr>
<td>Poor surface</td>
<td>Mixer fill not correct, ram bottoms very early in cycle.</td>
<td>Increase mixer fill so that the ram still exerts a little pressure on the mix throughout the cycle. Compounds made with NORDEL™ MG Hydrocarbon Rubber need a mixer fill 3-5% higher than bale- or pellet-form rubber.</td>
</tr>
<tr>
<td>Poor surface or variable part properties</td>
<td>Granular product hangs up in door or at ram because of gaps created by wear of metal surfaces.</td>
<td>Ensure that mixer door is properly closed. Refurbish mixer. An extreme solution is to flux the NORDEL MG product before adding other ingredients. In other words, to revert to a conventional mix. This will work in old mixers but loses the mixing time advantage of the granular form.</td>
</tr>
<tr>
<td>Poor surface</td>
<td>Too much moisture in fillers. Defect in other compound ingredient.</td>
<td>Replace material as needed.</td>
</tr>
<tr>
<td>High and/or variable viscosity, rough extrudate</td>
<td>Possible black scorch.</td>
<td>High molecular weight polymers such as NORDEL MG are susceptible to black scorch. Black scorch can be prevented by adding sulphur (~0.2 phr) to the mix early in the cycle.</td>
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**Other Issues**

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<tr>
<td>Mixing time too long (longer than for bale polymer)</td>
<td>Oil is added after polymer starts to flux and the surface area is destroyed. Oil is not absorbed (mixer &quot;goes to sleep&quot;).</td>
<td>Use modified upside-down mix.</td>
</tr>
<tr>
<td>Power uptake is too rapid</td>
<td>NORDEL MG does permit very rapid power uptake and high maximum torque. This is generally a positive attribute. Some older machines may not produce enough power through the power peak at the black incorporation time.</td>
<td>Decrease mixer fill factor. Raise ram slightly while power requirement is high, or do not lower ram until torque decreases. Alternatively, increase lubricant level. However, do not over lubricate with oil or lubricants as this will cause poor dispersion.</td>
</tr>
<tr>
<td>The compound does not empty from the chamber after lower door opened</td>
<td>The white filler content is very high and the stearic acid is being absorbed, leaving none to act as a lubricant. This makes the mix sticky.</td>
<td>Increase stearic acid level, or use a proprietary fatty acid ester processing aid.</td>
</tr>
<tr>
<td>The compound does not empty from the chamber after lower door opened</td>
<td>Poor distribution or dispersion.</td>
<td>See fixes to improve dispersion/distribution above.</td>
</tr>
<tr>
<td>The compound is very soft (Shore A 40-50)</td>
<td>There are surface defects if all oil is added at start of mix.</td>
<td>Use split oil in a modified one-pass mix. Save 1/3 of the oil for later addition, together with a similar weight of filler. Add to mixer and wait until mixer picks up before lowering the ram. Additionally, warm the oil prior to feeding into the mixer.</td>
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<td>The warehouse temperature is too low in winter</td>
<td>The NORDEL™ MG Hydrocarbon Rubber is cold and there is no heating in the warehouse and/or there is no possibility to pre-heat the polymer.</td>
<td>The granular form of NORDEL MG is less prone than bale-form EPDM to have problems if the polymer is cold, and usually does not need pre-heating.</td>
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<tr>
<td>There is a long dead time at the beginning of the mixing cycle</td>
<td>The mixer is too cold.</td>
<td>Increase mixer body temperature if the temperature is variable. Run a warm-up batch at the beginning of a shift to get mixer to temperature.</td>
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