

Processing of Cariflex™ IR Polymers: Cariflex IR0307 and Cariflex IR0310

Mill Mixing Procedure for Rubber-rich Formulations

1. Set mill roll temperature to 50 °C on the front roll, and 55 °C on the back roll.
2. Set {back/front} shear rate ratio to 1.2-1.4 (e.g. 20 & 15 RPM).
3. Set a tight nip on mill.
4. Introduce unwrapped Cariflex™ IR polymer to mill, allowing it to form a thin band on front roll straight away¹.
5. Gradually widen nip setting to draw fresh polymer onto mill rolls¹. Cutting band - to a limited extent - will also allow fresh polymer to be drawn onto mill. If the polymer band splits and a portion goes to the back roll, opening the nip further will encourage the band to return to the front roll². Continue to widen nip setting until only a small 'rolling bank' remains. The band should be almost transparent at this stage with little trapped air. The nip setting is now about 5-7 mm.
6. Add powder ingredients (non-curatives) to portion of polymer band in front of rolling bank. Small cuts to the left and right of the band will assist in getting the rolling bank and any associated loose powders incorporated into the body of the mix and avoid getting powder ingredients on mill rolls. Extensive cutting, e.g. in the middle of the band, should be avoided.
7. Add any liquid non-curative ingredients.
8. Add powder curatives, again use small cuts of about one third of the width of the band to the left and the right to assist in the incorporation of the rolling bank and any associated loose powders. Extensive cutting, e.g. in the middle of the band, should be avoided.
9. Add any liquid curatives.
10. Only when loose powders have been incorporated can band be cut, twice - only - on each side to further disperse ingredients.
11. Cut compound from mill and 'grind' twice - only - pass through a tight nip.
12. Return compound to mill at desired nip setting ready to sheet out.

Mill Mixing of Filled Compounds

1. Processing behavior with fillers is very good.
2. To be active in some filled Cariflex IR compounds (e.g. calcium carbonate), the level of PEG (e.g. PEG400) needs to be increased and compared to equivalent rubber rich compounds. Where a PEG400 level of 0.02 to 0.06 phr is used in rubber rich recipes, levels in the range of 0.10 to 0.40 phr will probably be optimal for moderately filled compounds (15-40 phr).

Notes

1. If it is not possible to widen the nip setting with the mill in operation a band is formed by passing the Cariflex IR polymer through a tight nip then massing the Cariflex IR products together and opening the nip before re-introducing the polymer.
2. If Cariflex IR products stick to the back roll, a little stearic acid added to the back roll will assist in displacing polymer and returning it to the front roll.



The mill mixing operation for unfilled compounds should take of the order of 10-12 minutes to complete on a laboratory mill, or around 15 minutes at production scale. For filled compounds it'll be approximately 5 minutes longer. The Cariflex IR0310 polymer has a wider processing window than Cariflex IR0307.

Internal mixing (Banbury) procedure for rubber rich formulations

A/ Cariflex™ IR0307

Cariflex IR0307 polymer can be mixed using the following procedure. This procedure is based on processing in a 3.5 liter batch size 00C Banbury internal mixer. Adjustments may be required for mixing on a larger scale or if an intermeshing rotor type or mixer is used. The general principles will still apply.

Please note that Cariflex IR0307 polymer cannot be employed in a single pass procedure. A certain amount of mastication of Cariflex IR0307 polymer is required before powder addition can occur. If Cariflex IR0307 polymer is not masticated sufficiently before addition of powders poor mixing will occur and the batch will be dumped as a crumb and be very difficult to process.

Although powders can be added as part of the first stage after the mastication stage, curatives like peroxides cannot be safely added in the first stage. They must be added in a second stage either by returning the batch to the internal mixer or on the open mill. However, either sulphur or accelerators can be added in 1st stage. This may help to shorten the total mixing time and to improve dispersion of these chemicals.

1st Stage

1. In most cases the mixer can be overloaded by 10% as compared with a similar 100% high-cis IR (ZN-IR) or Natural Rubber based stock.
2. Set a low temperature for rotors and mixer body (e.g. 35 °C).
3. Set rotor speed to low (e.g. 25 rpm).
4. Unwrap the bales and load the mixer with Kraton Cariflex IR0307 polymer.
5. Set ram weight press to half of the maximum pressure (e.g. 65 psi) and mix for 30 seconds.
6. Continue the mastication for 60 seconds at maximum pressure.
7. Raise the ram and add the reinforcing fillers (e.g. Carbon Black, Silica etc.) and processing aids (e.g. Stearic acid, Zinc stearate etc.) and mix it at maximum pressure for 90 seconds. Add the powders in 'sandwich' style to avoid loose powders.
8. Add the non-reinforcing fillers (e.g. clay) and mix it at maximum pressure for 60 seconds.
9. Perform 'sweep' operation for 60 seconds (if required) and subsequently dump the compound.

The batch should be well knitted, and not appear crumbly. Dump temperature, measured by probing the mix with a thermocouple, should be approximately 95 °C. When continuing to mix the batch in the 2nd stage at above 100 °C results in a crumbly and difficult to process batch.

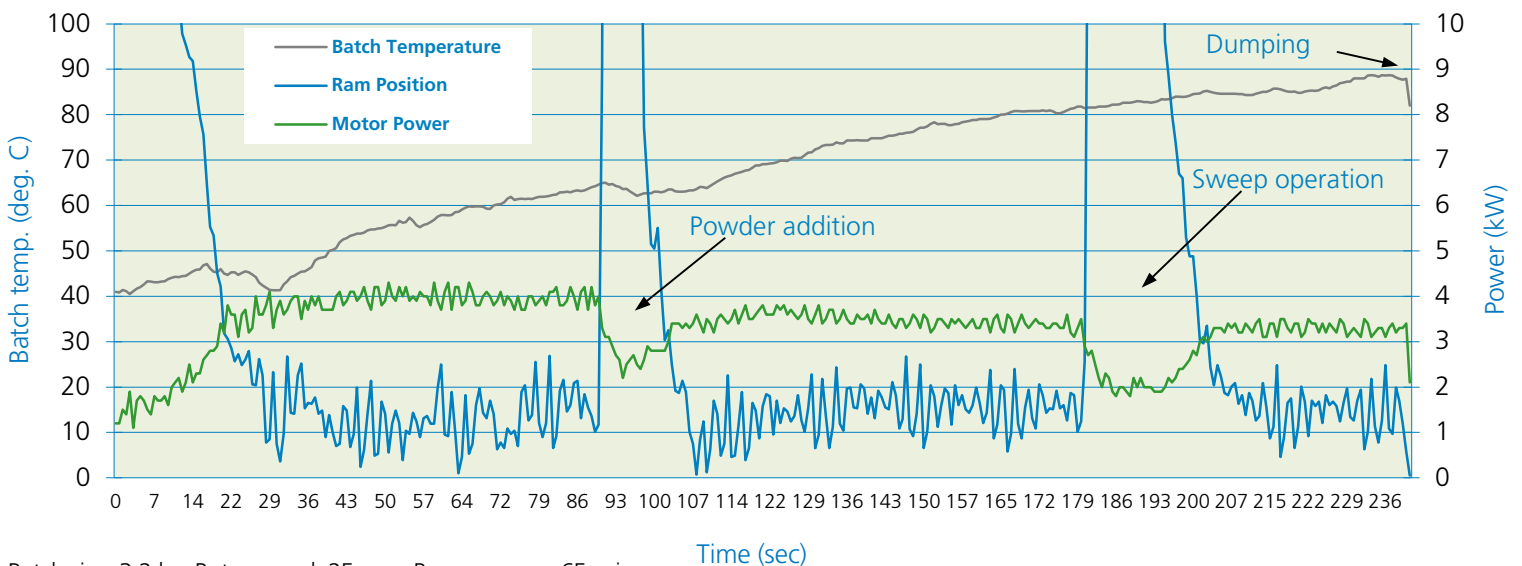
2nd stage (open mill)

10. Transfer the batch to an open mill. The front roll should be set to 50 °C and the back roll to 55 °C. The nip setting is set to about 5 to 7 mm.
11. Add the curatives and mix it properly by cutting and passing the compound twice through the mill. (tight nip; e.g. at 18-inch roll mill, the nip should be set 2mm).
12. Sheet the compound out at the desired nip setting.

2nd stage (internal mixer)

10. If curatives are to be added in the internal mixer, the dump temperature should not exceed 85 °C. Mix the curatives at maximum pressure for 90 seconds.
11. Sheetting of the compound can be done with a roller mill. Before doing so a refining action is needed. Pass the compound twice through the roller mill. (Tight nip; e.g. at 18-inch roll mill, the nip should be set 2mm).
12. Sheet the compound out at the desired nip setting.

Cariflex IR0307 MASTERBATCH



Batch size: 3.2 kg; Rotor speed: 25 rpm; Ram pressure: 65 psi max.

B/ Cariflex™ IR0310

Cariflex IR310 polymer can be mixed using the following procedure. This procedure is based on processing in a 3.5 liter batch size 00C Banbury internal mixer. Adjustments may be required for mixing on a larger scale or if an intermeshing rotor type or mixer is used. The general principles will still apply (profile attached).

Although powders can be added as part of the first stage after the mastication stage, curatives like peroxides cannot be safely added in the first stage. They must be added in a second stage either by returning the batch to the internal mixer or on the open mill. However, either sulphur or accelerators can be added in 1st stage. This may help to shorten the total mixing time and to improve dispersion of these chemicals.

1st Stage

1. In most cases the mixer can be overloaded by 10% as compared with a similar 100% high-cis IR (ZN-IR) or Natural Rubber based stock.
2. Set a low temperature for rotors and mixer body (e.g. 35°C).
3. Set rotor speed to low (e.g. 30 rpm).
4. Unwrap the bales and load the mixer with Kraton Cariflex IR310 polymer.
5. Set ram weight press to half of the maximum pressure (e.g. 50 psi) and mix for 30 seconds.
6. Raise the ram and add the reinforcing fillers (e.g. Carbon Black, Silica etc.) and processing aids (e.g. Stearic acid, Zinc stearate etc.) and mix it initially at low pressure and increase to maximum pressure after 10 seconds. Add the powders in 'sandwich' style to avoid loose powders.
7. Add after 90 seconds the non-reinforcing fillers (e.g. clay) and mix it at maximum pressure for 60 seconds.
8. Perform 'sweep' operation for 10 seconds (if required) and subsequently dump the compound.

The batch should be well knitted, and not appear crumbly. Dump temperature, measured by probing the mix with a thermocouple, should be approximately 85 °C.

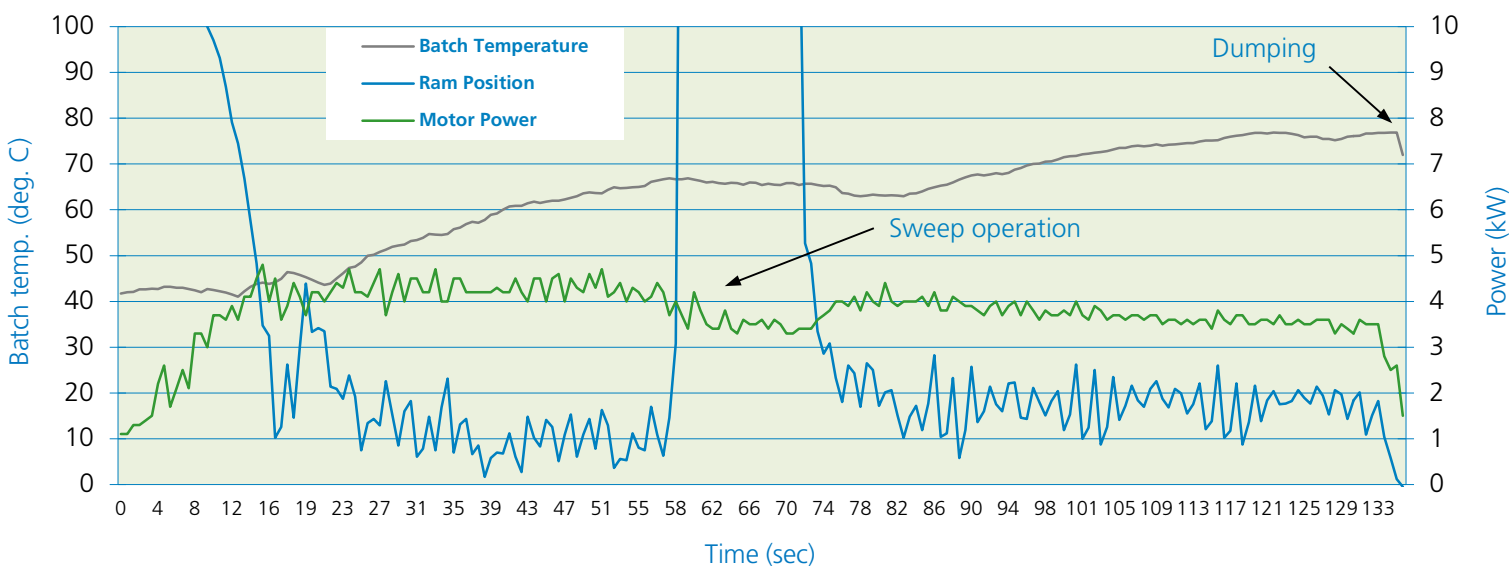
2nd stage (open mill)

9. Transfer the batch to an open mill. The front roll should be set to 55°C and the back roll to 60°C. The nip setting is set to about 5 to 7 mm.
10. Add the curatives and mix it properly by cutting and passing the compound at least twice through the mill. (Tight nip; e.g. at 18-inch roll mill, the nip should be set 2mm).
11. Return the compound to the mill and sheet the compound out at the desired nip setting

2nd stage (internal mixer)

9. If curatives are to be added in the internal mixer, the dump temperature should not exceed 85 °C. Mix the curatives at maximum pressure for 90 seconds.
10. Sheetting of the compound can be done with a roller mill. Before doing so a refining action is needed. Pass the compound twice through the roller mill. (Tight nip; e.g. at 18-inch roll mill, the nip should be set 2mm).
11. Sheet the compound out at the desired nip setting.

Cariflex IR0310 Fully Formulated



Batch size: 3.3 kg; Rotor speed: 30 rpm; Ram pressure: 50 psi max.

Important processing information

1. Finished compounds should mature for at least 12 hours.
2. Compounds based on blends of Cariflex™ IR0307 and IR0310 polymers benefit from an improved processing window, allowing the batch to be mixed at higher temperature and/or for longer cycles.
3. When using a blend of Cariflex IR0307 and IR0310 polymers, Cariflex IR0307 polymer should be masticated before adding the Cariflex IR0310 polymer (see previous steps in the procedure).
4. When employing an internal mixer in the 1st and 2nd stage for processing Cariflex polymers it is recommended to use only IR0310 polymer.
5. While the same processing window identified for filled Cariflex IR compounds exists for unfilled compounds, in terms of the processing temperatures, the width of the processing window for the amount of time that a compound may spend being processed appears to be decreased when processing Cariflex IR without fillers. The mixing time required for unfilled compounds compared with that for a filled compounds is 4 minutes versus 5 minutes for IR0307 and 2 minutes versus 3 minutes for IR0310.
6. For better mixing properties it is also important to increase the fill factor used in the internal mixer with 8%. The temperatures in the internal mixer should be kept below the maximum temperature as mentioned earlier in the procedure.
7. The presence of fillers such as calcium carbonate has a significant effect in reducing the rate of cure of Cariflex IR compounds. The effect of PEG400 in increasing the efficiency of the cure of Cariflex IR compounds is also much reduced in the presence of calcium carbonate. It is likely that PEG400 is absorbed onto the surface of the filler and is not available to participate in the cure. The reduction in the rate of cure of Cariflex IR compounds means that it is possible to mix calcium carbonate filled Cariflex IR compounds in a single stage. Although a relatively high mixing temperature is achieved during mixing, the reduced cure rate means that premature cure is not encountered. It is likely that should a more active cure system be used or increased levels of PEG400 so that a faster cure rate is achieved, then a single stage mix may not be appropriate anymore.
8. If a filler such as calcium carbonate and an internal mixer are used then the PEG400 booster, if applicable, should be added to the mix either late in the mixing cycle or in a separate mixing stage to obtain cure times comparable with the unfilled mixes.



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