Rheology and Processing of Paints, Plastic and Elastomer based Composites
Prof. Santanu Chattopadhyay
Rubber Technology Centre
Indian Institute of Technology Kharagpur
Lecture 36

Practical demonstration of Rubber mixing on a two roll and using an internal mixer

Welcome back to NPTEL online certification courses on rheology and processing of paints, plastic and elastomer-based composites. Today we are in week 6, lecture 6. So today we will be demonstrating you all about the two-roll mill which is also referred to as the open mill as well as the internal mixture. So we all know that mixing or compounding of rubber includes many ingredients. So as you can see over there, there can be polymers, there can be other ingredients like the resins, zinc oxides, stearic acid, there is fillers and there is a gamute of accelerator and curing agents. So while mixing we use combination of polymers, we can use combination of fillers, we can use combination of resins, combination of various accelerator, curing agent as well as protective agents like anti-ozonant, anti-oxidants.

To start with, we will first go through the description of the machine. So in case of two-roll, as you can understand from the name, it is having two rolls. Two cylinders are there, one we call as a front roll. Why front roll? Because the operator will be in front of this.

So the roll which comes nearer to the operator is the front roll and we also have a back roll. Along with that, the distance between the rolls is called nip gap. Now the nip gap can be adjusted using these two wheels. So you can see there are graduations like 0,.5, 1, 2.

Here also we have the same. So these are in mm, millimeters. So both the graduations are to be at the same graduation of the rolls. So that the nip gap for both the sides of the roll remains same. Now along with this, inside the machine we have bull gears which actually drives the machine.

It is inside the gear assembly which is inside this machine and we also have a panel which controls the overall functioning of the machine. Coming to the panel box, we have back roll RPM, we have front roll RPM and the ratio is called the friction ratio. So RPM you can understand how many rotations per minute the front roll is or the back roll is moving. And their relative ratio will determine the friction between the rolls. And this can be controlled using these two discs.

Now if you start the machine, you will initially find that the RPM is increasing. You can also see over the rolls, it is slowly and gradually increasing the speed. And after a certain time you can see that the RPM of the rolls are fixed with a friction ratio of 1.12 or 1.

15. This is determined according to the user of the two roll. So we have fixed it as 1.13. Next is about the safety feature of the machine. As you can understand the machine is running very fast.

So there is a high probability that an error of a human being can cause catastrophic failures. My hand can go inside this, any loose clothing or hair can go inside this. And for that we have a safety system. So as soon as my hand goes inside, we have our knees free which we can just press it. Immediately the rolls stopped and it is rotating backwards.

See the roll is rotating backwards. So this is only because of the safety of the user. Another thing is while the mixing is going on, the roll will be getting heated up because of the shearing forces and the shearing action of the rolls. So in order to cool down the rolls, the rolls are drilled peripherally and there are holes inside the rolls through which water can be circulated. Even in the colder weather, we can pass hot water so that the rolls can be heated up for easy mixing of the ingredients.

Permission of band, whether it will be on the front roll or on the back roll is mainly determined by the two forces. One is the elastic forces, another one is the adhesive forces. So when the elastic force dominates, the rubber sticks to the front roll. But when the adhesive forces dominates, the rubber sticks to the back roll. So as soon as the adhesive forces is overcome by the elastic forces, rubber will be shifting to the front roll.

And as soon as the elastic forces gets dominated by adhesive forces, the rubber will be shifting to the back roll. So we'll try with another rubber, which is the NBR. We're starting with a higher nip gap. Usually at a given temperature and nip gap, if the rubber shifts from the front roll to back roll, that is called a critical nip gap at a given temperature. We have added a small amount of carbon black.

We'll decrease the nip gap. The rubber has started to go towards the back roll. It has just started. So after a couple of passes, it will be going completely towards the back roll. So now you can see that the bank has shifted from the front roll to the back roll.

So this transition at this constant nip gap is called the critical nip gap. So this completes the whole gamut of things which are related with the machines and its safety. Next we will move on to the various ingredients we will be mixing today in the two roll. So in case of

rubber mixing, we always follow a standard formulation. So here you can see on the screen, we have given you a formulation which has the following ingredients.

Now in case of rubber mixing, the ingredients are broadly classified into four categories. First is the polymer, which is in our case is the natural rubber and it is RSS3. Then we have a gamut of antioxidant, anti-degradant and anti-ozonant. Next we have a group of accelerator activators. So we have used zinc oxide and stearic acid.

Then we have filler, which is in our case we have taken carbon black N330 and we are also having oil. And lastly, we will be adding accelerator, in our case it is CBS and sulphur. Now we will start mixing our formulation. So the first step of mixing is mastication. So we have put a nip gap of 1 mm.

As you can see from the dial, it is 1 mm. So initially we will keep a bigger nip so that the rubber can pass. As you can see from the texture of the rubber that initially it is difficult to pass the rubber. So after a few passes, the rubber becomes soft. So we will wait until the rubber becomes soft.

So now we can see that it is almost soft. Now we will change the nip gap. We have chosen a smaller nip gap. Now we are trying to form a band, which is very essential for mixing. So initially the rubber is not adhering with the rolls.

As you can see, it is almost like slipping. So we will give some time for mastication. So in the stage of mastication, the rubber molecular chains are broken down, which will be helpful for easier incorporation of the fillers into the rubber matrix. Now you can see that it is already, the band formation has happened as well as you can see some bank on top of the nip. Now with this phenomenon, we are ready for adding our first additive.

So first, because it is a natural rubber, it is a unsaturated rubber, we will start adding the antioxidants and anti-ozonants so that we can restrict the further degradation of the rubber chains. You can see that we are cutting the band. So this cutting is done to ensure that the incorporation of the additives are good. And we are also following the crisscross mixing in order to have a uniform mixing. In industries, this is automatically done by a knife.

Here also we are using knife, but we have to do it manually. After the band is again reformed, we can add our next ingredient, which is the accelerator activator package. Now we can slowly add zinc oxide and stearic acid. As you will already know, which Professor Chattopadhyay has told in his lectures, that rubber mixing is more of an art than science. So this mixing is actually very important because it determines the quality of the product which will be formed.

So uniform dispersion and distribution of each and every ingredient plays an important role. Now we will take out the whole material from the roll and we will put it again. We will again form the band. Now rubber is almost ready for taking the fillers. So now we can add carbon black, which is a reinforcing filler.

We will slowly add carbon black. Addition of high loading of carbon black is usually accompanied with an addition of oil. So we will put half of the carbon black and we will put the oil. Again we will put some carbon black, but before that we will again ensure the proper mixing by crisscross cutting and mixing. During the course of mixing, the rolls get heated up. As I have mentioned earlier, the rolls are peripherally drilled and we will ensure that the water is running throughout the roll surface for cooling the surface.

Now we are adding the rest of the carbon black. So it is two-stage carbon black mixing and we will add the rest of the oil. Two-roll mixing is an age-old practice of rubber industries and rubber for making rubber products. Oxidative degradation in 2 rolls are faster because it is an open mill. Following this we will demonstrate about the internal mixture, which you can see that it is a lot easier because it is not open and also the loss of material is less.

It takes a lesser time to incorporate the fillers into it. So from the bank you can easily understand why this crisscross mixing is very necessary. Again we will follow the earlier step. We will take out the material. We will try to form roll, which is usually termed as pig roll.

So we are almost ready with our carbon black mixing. Now we will add sulphur and accelerator. So by this we are achieving the four stages of mixing. The mastication, Incorporation, Distribution and Dispersion. In order to mix it more homogeneously, we will make pig rolls.

So this enables more better dispersion, distribution and these are called pig rolls. You can see the rolls are made and we perform this a couple of times. The final batch is sheeted out at a higher nip gap. So this is our final batch and we will do various rheological testing as well as other characterization using ODR, MDR and other instruments which we have already shown you in earlier week. So in this particular sheeted out texture you can find two types of things.

One is a blister. There are many blisters. Another one is a crow's feet. This crease like marks. So these are the two types of problems which appears when we are mixing on a two roll. One is due to the overheating of the roll.

Another one is due to the cooling of the rolls. So this is not a problem while mixing but this can be a problem while we are calendaring a product. While mixing on a two roll we always keep a direction, we keep a directional mark because rubbers, specially the strain induced rubbers or the fillers which can give directional property in the direction of the mill. So we call it as a milling direction. So after we have demonstrated you all about the mixing on a two roll, now we will be focusing on internal mixer. So in case of internal mixer there are few parts you have to understand first.

So if I open the machine you can find, so this is the mixing cabinet and you can also find the rotors over here. So there are many types of rotors like the cam type rotor, the roller type rotors, the intermeshing type rotors, the sigma type rotors and the banbury type rotors. So here we have the cam type rotors. We also have a ram which slides on top of the cabinet.

This is just for showing you all. So this completes the cavity of the internal mixer. So I will show you after the mixing is done how the rubber is usually occupies the volume inside the internal mixer. Now to have a better understanding of how the rotors work I will operate the machine with the help of the software which is connected with the machine and I will give a small rotation of the rotors. So now you can see I have given a rotation of 10 rpm and the rotors are moving. So the shearing action is generated by the rotors at different points like the wall.

See this particular area will be experiencing the shear. Another portion will be if I open this cabinet, so now if you see the material which will be coming inside these two portions will be experiencing shearing actions. So usually we mix at a rpm of around 50 to 60 depending upon the requirement and we give the shearing action to the rubber material or the polymer material we are mixing. So now we will be discussing about what is the importance of the internal mixer. Now as you have seen that in case of two roll we are mixing it openly.

So spillage of the material is a concern which is actually can be avoided in case of an internal mixture. Second the control over the temperature like on a two roll mill if you want to mix a temperature of 80 degree centigrade or you want to mix at a temperature of 150 degree centigrade you can't get a control proper control over the mixing temperature but in case of an internal mixture you have the heating coils at different stages of the mixer and you can control the temperature effectively. Also the shearing action is much more in case of an internal mixture which actually helps in better dispersive mixing. Also we need to take care of the fill factor that means suppose this cavity is of 85 cc now we also have a fill factor suppose 0.

7, 0.6. Why fill factor is needed? While the rotors are rotating it takes some of the volumes so you need to have a empty space in order to have an efficient mixing of the materials. So this fill factor is calculated with the help of various rotor speeds. So we have a master curve which has been described by Professor Chattopadhyay in the theory classes and in this mixing we will be following a fill factor of 0.7 and a rotation or the rotor speed of 60 to 70 rpm. So now we will be demonstrating two mixings one with a reinforcing filler another one with a non-reinforcing filler and we will show you the mixing curve that is a torque time curve.

So why the torque time curve is important because while mixing in a industry they usually go for the energy mixing so we call it as energy mixing or the torque time mixing. So what happens is while we are putting down the RAM the shearing action of the rotors takes place and also the torque which is being generated by the rotors is calculated or directly proportional to the energy required for the mixing. So in usually the industrial practices what we follow is: we try to minimize the energy because that will be adding to more cost and also while mixing we have to take care that the number of RAM up and RAM down should be minimum. One of the disadvantages of this type of internal mixer is because of the temperature issues we usually don't mix the curatives inside an internal mixer but after the mixing is done we dump the material and we mix the curatives on a open well. So for the first batch these are the ingredients we are taking the natural rubber, the antioxidants, zinc oxide stearic acid and the non-reinforcing filler which is a calcium carbonate.

At first we will be putting the rubber and you can see that the torque is gradually increasing. Another advantage of internal mixer is we don't have to be afraid of over mastication or over oxidative de-gradation which occurs in case of the two roll mill. So you can see that the torque has increased. You can also see in case of this ram this wheel it's moving a bit so this ensures that the ram is in dancing position and this is very much important for efficiency of the mixing. So we'll wait for a two to three minutes or maybe two minutes for the mastication stage and then we will add other additives.

So as you can see the torque has almost become constant then we will again open the ram and we will put our next ingredients which is the antioxidant. See as we are putting up the ram there is a decrease in the torque. Now we will add zinc oxide and stearic acid. You can see inside the rubber is mixing and it's coming upwards so we have to put the RAM down. We will wait for few minutes then we will start adding non-reinforcing filler.

So this is usually decided by the play to nature of the torque. So now we will be adding our filler which is non-reinforcing in nature. You can see that the rubber is getting mixed. So we will slowly add the filler. In industries these materials are directly fed from various hoppers and various silos. It's automatically fed the time is calculated from the computer

and the programming system.

Now see in the graph we can see that there is a bit rise in the torque. This is because of the effect of the non-reinforcing filler. But the rise is not much. In case of a reinforcing filler there will be a significant improvement in the torque for the particular mixing cycle. So we will wait until the mixing is complete because we have set a mixing time of 10 minutes.

So we will wait for the mixing to happen till 10 minutes. So you can see that the mixing is over. We are at 10 minutes. Now we will stop the machine. Now as soon as we are stopping the rotor we can see that the torque is instantaneously coming to zero.

Now we will open the chamber and we will show you. So now we will open the chamber and we will show you the condition of the rubber. So you can see that the rubber has been mixed. So next we are opening this chamber and we will dump the material. So whatever we got from the internal mixer this will be passed on a two-roll mill and we will be mixing the curatives according to the batch weight and we will again follow the same pathway like doing the rheology and doing the various curing studies and we will be going for the end use or the product making.

So now we will be doing our second batch. We will be mixing a reinforcing filler that is silica. So the same steps like we will first add rubber. You can see that the rubber is being mixed inside. Again at two minutes we will be giving the antioxidant, ram down. So actually from the torque time curve we can pretty much determine the number of ram up, ram down, addition of low molecular weight additives and also addition of various fillers like whether it is a reinforcing or a non-reinforcing filler.

Next we will add zinc oxide stearic acid. So now we will be adding silica. You all know that silica is a reinforcing filler but the interaction between silica and natural rubber is poor. That is why we need to have a silane for the interaction, good interaction between the rubber and the filler. So silane and silica reaction takes place around 145 to 150 degree centigrade. So that is why we need to have a mixing temperature around 150 degree centigrade and we have given the silane over here and to start the reaction we also have given the DPG inside this particular filler.

Now we will add this filler slowly and gradually. So in a single go it is sometimes very difficult to add. The whole filler so usually we will be giving it in a two-step mixing so we will be waiting for some time. Again we will be doing a ram up and adding the rest of the silica. We will try to add the rest of the silica. So you can see that the mixing is complete and we will slowly reduce the rotation, the rotor speed and we will dump the material.

So from the torque time curve we can easily understand about the energy required for the mixing of the material. So now in a similar way we will be opening the chamber. It is again a repetition that in case of an internal mixer dispersive mixing is good. Again we will use this to pass on a two roll mill and we will be adding the required curing agents and we will be further processing the same with the help of a ODR or MDR to calculate the OCT. It is optimum cure time and accordingly we will be following standard procedures or standard molding techniques for making the products.