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Pavement Materials Professor Nikhil Saboo Department of Civil Engineering Indian Institute of Technology, Roorkee Lecture 15 Mineralogy of Aggregates and its Importance

Hello everyone. In the last lecture, we have discussed about the classification and gradation of aggregate. And if you remember that we ended discussing about the use of the software simple tool for aggregate blending step and we solved one problem. Overall, in the last lecture, the main idea was to understand what we actually mean by the gradation of aggregates, how different stockpiles which are typically formed in the plant through fractionation after the crushing process, they are mixed together, so that finally, we can have a combined distribution of aggregate which falls within some standard range or standard band and this standard band is typically decided empirically or based on experience we can say, considering the maximum density line as the reference gradation.

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Today, we are going to start discussing a new topic, which is on aggregate mineralogy and the importance of aggregate mineralogy. Usually, this topic is not conventionally discussed in pavement materials classes, because, typically, mineralogical properties of aggregates on the one side they are complicated in nature to understand because there are various forms of minerals which exist in different types of aggregates. And also, there is no correlation in measurable form, when we try to see the performance of the final mix and try to relate it to the mineralogy of the aggregates.

In fact, many studies have proven that the mineralogy of aggregates has a considerable effect on some of the very critical parameters, which are related to the performance of the mixture. So, our discussion today will be aligned to understand those parameters, which are typically affected by mineralogy, and we will try to understand that how the choice of aggregates can impact the control over those properties, which are very critical.

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So, as I was mentioning, the relationship of the mineralogy of aggregates with performance is not directly proportional. The reason being the mineralogical properties are complicated to study with conventional experimental procedures. A single aggregate can have various combination of minerals.

So, from source to source from aggregates to aggregates, the mineralogical composition will drastically change and as I said, it is not conventionally easy to quantify the mineralogy of the aggregates through simple laboratory testing. And many times, this correlation and in fact, the mineralogical attributes of the aggregates are they are not known or they are mostly unknown in nature.

If we try to see the main parameters, which get affected directly by the mineralogy of aggregates are the adhesion characteristics and the skid resistance properties. Adhesion, here, we are referring to the bond between the bitumen and the aggregate particles in the hot mix asphalt or the asphalt mixture, because in asphalt mixture, moisture damage is one of the critical modes of failure we can say or moisture damage leads to several problems in the bituminous mixture or the surface of the flexible pavement. Therefore, adhesion becomes one of the parameters of interest of study, because if we can improve the adhesion, if we can control the factors affecting adhesion, we can also control the performance of the hot mix asphalt.

On the other hand, the vehicles they move over the surface and in the surface, we can have an asphalt mixture we can have a concrete mixture and a large portion as we were discussing of these mixtures comprises of aggregates. Now, when the vehicles move over the surface, there is a frictional force which is acting between the tires and the pavement surface and because of these frictional forces, there are chances that the surface will wear off depending on the type of surface and depending on the type of aggregates we have. So, the aggregate mineralogical composition is also related to its resistance to this wearing action between the tires and the pavement surface. So, that is something again which we are going to discuss from the perspective of Mineralogy of aggregates.

So, now, let us start discussing about these performance parameters one by one and let us start talking about adhesion. Adhesion here it refers to the bonding between the bitumen film and the aggregate particle in a conventional hot mix asphalt. So, here we first we have to understand that what facilitates this adhesion and the vetting of the aggregate surface with the bitumen particles and then what are the factors that affect adhesion or that will reduce the adhesion characteristic. Because, it is this reduction in the addition characteristic that the bitumen can strip off from the aggregate surface and finally, this leads to moisture damage and it aggravates many other failures in the pavement.

Now, the reduction in addition of the bitumen from the aggregate surface itself is a very complicated phenomena and there are various mechanisms which have been highlighted in the literature that leads to the reduction in addition. So, if you try to list out some of the factors that affect or some of the mechanisms that leads to the removal of the bitumen film from the surface of the aggregates, these include detachment.

Now, detachment is a phenomenon which will depend on the mineralogical attributes of the aggregates, it will depend on the free surface energy of the different components including aggregates including bitumen and including the presence of water. So, we are mostly going to discuss about the factors controlling detachment from the perspective of the mineralogy however, the removal of the bitumen from the aggregate surface is not only limited to the concept of detachment.

There are other mechanisms including displacement we have hydraulic scouring. Now, this mechanism is related also to the movement of the vehicle that when there is a film of bitumen and there is a suction from the tire of the vehicle, so, then there is the water suction when this force is applied, the water can also peel off some of the bitumen from the aggregate surface. So, that is hydraulic scouring.

We have also spontaneous emulsification which is a function of the temperature of the surrounding. Then we have the effect of pore pressure, if we have pore pressure built up within the mixture and there is movement of the vehicle report pressure can be high enough to induce impulse forces on the aggregate particles separating it and thus breaking the bond between the bitumen and the aggregate. We also can have Ph instability. And, of course, there can be other environmental factors which can affect the adhesion characteristic between the bitumen and the aggregate particles. So, as I said the mechanism is not very simple, it can be a combination of these mechanisms that actually happens in the field. But our idea here is to understand from the mineralogical perspective of the aggregates, that how the adhesion is related.

Talking about the wettability of the aggregates by the bitumen in presence of water, this process is a physical chemical phenomenon and it is not very well understood. In fact, the aggregate wettability is actually a function of the amount of surface tension or surface free energy of adhesion. It all depends on how easily the material the liquid material which is bitumen here is able to coat the surface of the aggregate and when I say the surface energy it means that how much resistance is offered by the aggregate surface to coat it.

From the concept of thermodynamics, if you try to understand the aggregates are in stable form, so, it will ideally not allow any other material to interact with it so easily, because it has its inherent resistance when other materials come closer to the aggregate particles. So, first the bitumen should be able to overcome this energy requirement, so, that it can wet the aggregate surface. So, not only bitumen, any other liquid when it tends to wet the surface of any solid particle, it has to overcome this surface energy requirement.

So, lower will be the surface energy requirement or the surface tension of adhesion, easily a liquid material will be able to wet the solid surface. So, the same thing applies in case of aggregate and bitumen and also aggregate and water. So, water reduces the surface energy requirement higher degree in comparison to the bitumen as a material.

Therefore, the water has better affinity to wet the surface of the aggregates in comparison to bitumen and this is the reason when bitumen has been coated with aggregate and water comes in there are more stronger forces which acts between water and the aggregate surface which can cause disruption to the bonding which has taken place.

Now, this bonding is weak in nature which has taken place between the bitumen surface and the aggregate surface. So, adhesion can also be understood from the perspective of surface free energy concepts. But of course, we are not going to discuss in detail about the mathematical details of the surface free energy concept here. Now, aggregates have different mineralogy. In fact, there are some predominant minerals which commonly exists in different types of aggregates with which construction is done.

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Aggregates can have various active minerals such as silica, we have feldspar, aluminosilicates, we have carbonates, etcetera, and the various elements which forms these minerals, they include, if I write elements here, they will include elements like aluminum, they will have iron, we will have magnesium, we can have sodium, we can have calcium, etc. So, these are some elements which are present in the active minerals of the aggregates which are used in construction.

Similarly, bitumen ideally is inert in nature but because of the presence of functional groups containing Sulphur, oxygen and nitrogen, which has a tendency to interact with other surfaces. Now, these polar functional groups contains molecules which can be either acidic in nature or can be basic in nature and that has a tendency to interact with surfaces such as aggregates.

So, I will write down some of the very common acidic and basic types of molecules which exist for example, we have carboxylic acid, we have anhydrides etc. and if we talk about the basic form, we have sulfoxides we have pyridines, etc. this is the presence of these functional groups that bitumen has some forces attached to the aggregate particles or the surface of the aggregates.

And as I said in the surface of the aggregates also, we have active minerals, which keeps tendency to interact with other materials such as bitumen and water. Of course, the interaction of the aggregate with water is more stronger in nature, because they both tend to be more polar in comparison to bitumen which is less polar in nature.

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Siliceous aggregates are acidic in nature and in presence of water they have negative surface charges. Now, important question here is How do we understand that Siliceous aggregates will have negative surface charges mostly, but not necessarily this we will discuss in further slide. Then we also have calcareous aggregates they are basic in nature and in presence of water they have positive surface charges. So, I will make it clear why I say that Siliceous aggregates are acidic in nature whereas, calcareous aggregates are basic in nature.

Bitumen because of the presence of this functional polar groups for example, let us say carboxylic acid. So, we will see that the presence of carboxylic acid can impart overall negative charges to the surface of the aggregates and therefore, the bitumen have strong bond with calcareous aggregates like limestone in comparison to Siliceous aggregates like granite that is why it is very aggregate quarries with high silica content we mostly need the use of lime which are used which is used as an anti-stripping agent or these days.

There are various proprietary products also that falls under the anti-stripping agent and the function of this anti-stripping agent will which we will discuss is to improve the bond between the aggregate particles or surface and the functional group of bitumen, we will try to discuss about this bond in a better way. What happens that when bitumen comes in contact with the aggregate? It gets absorbed at the surface of the aggregates.

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Now, polar group in the bitumen as I said for example, carboxylic acid, Sulfoxide, pyridines, phenols, and others they get converted into salt. Now, this is the chemical mechanism which has been explained in literature that these functional groups they get converted into salts of a sodium, magnesium, potassium or calcium. this depends on the presence of active minerals on the surface of the aggregates. So, depending on the type of minerals, the corresponding salt will be formed.

So, this acid it reacts with these active minerals and they get converted into salts of sodium, magnesium, potassium or calcium and these salts can have monovalent bond, they can have divalent bonds. For example, salt salts of sodium, magnesium and potassium, they get easily dissolved in water. So, when water comes in as a third material between the aggregate particles and the bitumen, this salt which is formed, it gets dissolved in water and therefore, the bond between the bitumen and the aggregate surface they get broken easily.

On the other hand, the calcium-based salts they are made of divalent bonds, and they are more stronger in nature, so they are not soluble in water, therefore, they cannot be broken and this is one of the reason. Calcium based material for example, lime, they are used as anti-stripping agents. So, these anti stripping agents, they promote the formation of these divalent in bonds, so that when water comes in this bond does not get broken up and the adhesion between the bitumen and the aggregate particle is not disturbed.

What we have discussed, that sodium, magnesium and potassium salts they are water soluble, while calcium salts are not and does limestones have a tendency to perform better in adhesion in comparison to granite or any other form of Siliceous aggregates. The pH of water also affects the affinity however, this effect is not very clearly defined. So, but in general it is believed that higher is the pH of water, which means more basic the water is so, poorer is the adhesive bond between the aggregate and the bitumen.

Lime and anti-stripping agents, which can be amine based or silane based they are used typically to resist moisture damage. And our experiments in the laboratory have also indicated that lime is a very-very strong and also easily available, a cheaper product you can say which gives very good resistance to moisture damage.

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So, this slide is an attempt to explain the mechanism of understanding the acidity and the basicity of Siliceous aggregates and calcareous aggregates and also to understand how the surface charges might get formed on the surface of the aggregate particles. So, adhesion of course, is a function of interaction between polar groups and all these materials have some form of polarity, some materials are more polar in nature for example, aggregate and water and some materials like bitumen they are weekly polar in nature.

Let us try to understand about the formation of these charges. Let us say we have Siliceous aggregate and we have a freshly cut Siliceous aggregate when this freshly cut Siliceous aggregate having a silicon oxide  $SiO_2$  it comes in contact with the atmospheric moisture. So, the hydration process it converts the moisture into H<sup>+</sup> and OH<sup>-</sup> and this H<sup>+</sup> and H<sup>-</sup> interact with SiO<sub>2</sub> to form the silanol group which is Si-O-H.

Now, water has a tendency to form strong hydrogen bond with Siliceous segregate, which can replace bitumen polar groups when bitumen is coated with the aggregate. So, what happens that when this silanol group comes in contact with basic water, so, if it is a basic water, it will have a tendency to remove H<sup>+</sup>. So, it will pull out H<sup>+</sup> from the silanol group and therefore, the overall charge is negative in nature. So, the net charge is negative.

On the other hand, it can also happen that there is a presence of acidic water which is less likely. So, in that case OH<sup>-</sup> ions will be pulled out and the net charge will become positive and as I said this is usually unlikely. But when water comes in contact, they form strong hydrogen bonds and hydrogen bonds are stronger in nature. And therefore, in Siliceous aggregate water has higher tendency to disrupt the bond between aggregates and the binder especially in Siliceous aggregate.

Talking about calcareous aggregate, let us say we have limestone. So, we have calcium carbonate here, because of the hydration process, the atmospheric moisture can get broken down into  $H^+$  and  $OH^-$  ions and when this interacts with the calcium carbonate ions, we have the formation of  $Ca^+$  which means the overall charge becomes positive and the other parts get converted to water molecules and carbon dioxide. So, therefore, there is an electro positive surface which is available on the calcareous aggregate and now, both bitumen and water they will compete with each other to form bonds with  $Ca^+$  ions.

But in general, as I said that bitumen has overall negative charge. So, of course, it can form stronger bond with Ca<sup>+</sup> in comparison to the negative charges which is present in Siliceous aggregate and therefore, limestone has better adhesion with bitumen in comparison to granite or any other form of Siliceous aggregate. This is just an overview to understand the mechanism which takes place and how the mineralogy is involved in controlling the adhesion characteristics.



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This slide again is another form of attempt you can say to understand that how the bonding between the aggregate and bitumen can get disrupted. So, as I was mentioning that there are so, many factors controlling adhesion and we are just trying to look at from one perspective here, so, talking about bitumen to understand the charges in the bitumen. So, bitumen is basically a complex hydrocarbon and the details of which we will be discussing in another module, which is our next module.

So, then we will talk about the chemistry of bitumen, but for now, let us just believe that bitumen is a complex hydrocarbon and has various functional groups containing a hetero atom such as oxygen, Sulphur and nitrogen and because of the presence of these hetero atoms, these functional groups get formed in the bitumen structure and carboxylic acid is one of that molecule which gets formed.

So, carboxylic acid can be defined as R-COOH, where C-OOH describes about the presence of carboxylic acid where R is the hydrocarbon chain here. Now, when this comes in contact with the basic water, again, the basic water will have a tendency to pull out  $H^+$ , so the overall charge becomes R-COO<sup>-</sup>.

So, in case of Siliceous segregate, we have bitumen, which has an overall negative charge, we have the surface of aggregate which again has an overall negative charge. So, there is an inherent resistance between the two materials to come close to each other and form stronger bonds.

We can try to understand this with this animation also here, which talks about the polarity between different materials. So, we have water, we have aggregate, which are both polar in nature. So, of course, they have strong forces between each other or they have a higher tendency to come in contact with each other. On the other hand, bitumen an aggregate, aggregate is polar in nature however, bitumen is weakly polar in nature depending on the types of functional group it has and the forces which acts are weak dispersive forces.

So, they in their natural form, they do not like each other very much and therefore, it is easy for water to come in between them and replace the bit went from the aggregates surface. I think these points we have already covered when we started our discussion that the polar groups in bitumen in contact with the metallic ions from the aggregates, they form bond and this bond is in the form of salts of sodium potassium and calcium or others and these bonds get broken by water or because this salt get dissolved in water.

Now, since sodium and potassium forms weak mono-valent bond, they get dissolved in water easily whereas, calcium form strong divalent bond which is not affected by presence of water and therefore, we say that limestone is more moisture resistant in comparison to other forms of aggregate especially Siliceous aggregate. However, we have discussed about this mineralogical concept in in a simpler form, the actual phenomena is much more complicated here.

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With this now, let us start discussing about the skid resistance of the aggregates again, which is related to the mineralogical aspects of the aggregates. If you talk about the skid resistance and try to connect it with the mineralogy, so, in general the minerals like silica and feldspar they are stronger in nature and they can resist wear more than minerals such as calcite, which are weak in nature.

So, how do I find out whether this aggregate has more resistance to wear or not? So, there are certain tests which can be done in the laboratory to understand. So, one of the tests test is aggregate polished stone value which can be used to assess the skid resistance, we will talk about the process.

We have another method, acid insoluble residue test, which is mostly done on carbonate aggregate to see how much of minerals we have that is insoluble in the acid, because if we have weak minerals, they will get dissolved in this acid easily. We also have some full scale test that tells us about the skid resistance of the pavement surface. Again, we will discuss about this full-scale test.

We will start discussing about the polished stone value. So, in the polished stone value test, what we do we use single sized aggregate particles. So, usually we take 36 to 46 particles, which typically has a size of 10 to 6.5 mm. So, we take 36 to 46 particles and this is a pad which is there in the machine and in this pad using an epoxy we will attach all the individual particles one by one. After doing that, this pad is kept in this tire frame and you can see that this is just one pad so, we can have different pads here, at least there are like 14 specimen holders. So, all these 14 specimens will be placed on the surface of this tire in the similar way.

And then we will take this tire and we will put it here in this way. And then you see that this is the surface with exposed aggregate particles and from here we will insert another tire we will insert another tire here which will be in touch with the periphery or the surface of this tire. So, it will be something like this.

We have one tire here and then we have another tire here and this is in contact, and this contact will create the wearing action when this machine starts. So, this is the polishing machine and in this polishing machine, we do the polishing for 6 to 9 hours, typically 9 hours of polishing. Using a British pendulum tester, we will measure the frictional resistance of the aggregates surface.

So, first what we will do we will take unpolished aggregates and we will carry out the British pendulum test. So, this will tell me the actual value or the frictional number of the aggregate particles. And after doing the polishing using this machine. So, after doing the polishing, what will happen the aggregate particles will get polished off will wear off and the extent of wearing will depend on the type of aggregates the mineralogical aspects of the aggregates. For example, if weak minerals are there, they will be more wear in the aggregate surface in comparison to strong minerals. And therefore, we see that skid resistance is related to the mineralogy of the aggregates.

Then after doing this polishing, we will take out the mold, again, we will place it here and then here we have a pendulum, it acts something like this. So, this is the pendulum and here is the free end and from here it is free to move and we place the sample beneath. So, when this pendulum comes it touches the surface and it goes up. So, more is the resistance by the surface lower will be the height of this movement, less is the resistance of the surface more will be the height of the rotation.

So, depending on this height of rotation, you can see we have markers here, we will see that how far the pendulum travels once it has touched the aggregate surface which is placed here. So, this will give us an indication about the polishing of the aggregate particles of the or the resistance to a wear of the aggregate particles and this is called quantified using polished stone value and as per specification, the minimum polished stone value should be at least 55. I think this test is clear to you that how this test is done.

So, this is a full-scale test which can be done using a trailer vehicle we will discuss this in the next slide. So, here let me try to discuss the other two test which is the insoluble residue in the carbonate aggregates and the full-scale test.

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So, let us first discuss about the insoluble residue in carbonate aggregates, this test it gives us the amount of non-carbonate or acid insoluble material, it tells us about the amount of acid in soluble material. So, here we take 500 gram of aggregates that are retained on 4.75 mm sieve, we put it in a glass beaker with 1000 ml hydrochloric acid solution. So, this is what we do, then after putting this mixture and the acid solution we will agitate it and we will agitate till the effervescent stop, effervescence is these bubbles which will get created here.

So, until this formation of the bubbles stop, we will keep on agitating this beaker, then we will add additional 300 ml of solution and again agitate it, so this is what we will do in the next step. And then what we will do will heat the beaker will heat this beaker 210 degrees Celsius and then we will add some new acid solution incrementally until the effervescence stops. So, this is what we will do and then after the

entire process we will take out the material, we will sieve it, we will dry it, and we will again sieve it through 75-micron sieve.

So, and then we will see that what is the weight retained on the sieve. What will be the effect of the acid that it will break down the aggregate particles if the aggregate has strong minerals the amount of breakage will be less so, we will expect that higher amount of material will be retained on the 75 microns sieve. So, larger is the value of the weight retained more is the presence of insoluble material. And here we desire that minimum 10 percent should be at least retained, at least 10 percent of the material is desired here.

Now, talking about the other test which is the full-scale test, which is also called as a lock wheel skid tester, and here we have a skid trailer towed using a vehicle which I just showed you in the previous slide here you can see that this vehicle is actually towing the skid tester. What is done here that the skid tester is moved at a speed of 64 kmph approximately, and once this speed is reached what we will do will spray water in front of the tire spray water in front once the speed is reached.

And once the speed is reached and the water is sprayed, then we will lock the wheels, lock the wheel of the skid tester, and we will see that based on the amount of resistance offered we will see that what is the resistance to movement. The torque which is required after the locking it gets converted into friction numbers. Torque gets converted to friction number alright and higher is the actual value more will be the resistance to the movement and better will be the skid resistance properties. So, higher is the value more is the resistance.

I hope procedure of these tests are clear to you. And with this we will stop here today and just to recall that we have discussed about the mineralogical aspects and we have discussed the mineralogical aspect from two perspectives one is the adhesion characteristics of the aggregates and the other is the skid resistance properties of the aggregates.

So, in the next lecture, we will start discussing about the shape and surface structure properties of the aggregates which again is a very interesting topic, because the shape and surface structures are directly related to the performance of the mixtures with this thank you everyone we will meet in the next presentation.