

**Food Packaging Technology**  
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**Week – 04**  
**Lecture – 18**  
**Testing of packaging material P3**

Hello everyone. Welcome to another session in the Food Packaging Technology course. We are now in week 4, module 3. We have gone through some of the testing of packaging materials in part 1 and 2. A quick recap of what we did. We talked about testing of paper and paper boats and plastic films.

Now, these tests were concerned with the strength of the packaging material. For example, for paper we looked into the grammage, thickness, COBB's value, tear strength, puncture resistance. Similarly, in plastics we went through the methods of measuring their thickness, yield, tensile strength, seal strength, their trouser tear and the puncture test. Today we will be going into the third part of this testing materials and we will be looking mostly at the barrier properties.

One is the water vapour transmission rate, WVTR. The other one we will be dealing with is GTR, gas transmission rate and the overall migration of the components from the package to the food and how do we measure these. Let's start with the water vapour transmission rate. The water vapour transmission rate the testing, the first part is conditioning just like you do for your plastic. Once you condition your plastic material you can test the water vapour transmission either by the conventional method or by using a water vapour transmission rate testing machine.

Water vapour transmission rate is a measure of the passage of water vapour through your test packaging material. Now, it is a rate of water vapour transmission per unit area, per unit vapour pressure differential under test conditions and the unit is grams of water vapour per meter square in 24 hours that is in the SI unit. So you will see how much of the water vapour is actually passing through your test material. So if you want a test material which has got good barrier properties the water vapour transmission rate should be less. Now coming to the conventional method.

Now this is an important property of the packaging material under 3 mm thickness and it is important to do this test in order to select barrier packaging material for hygroscopic foods or foods that are very sensitive to water. So these foods have to be packed in a material which is actually a good barrier or has got less water vapour transmission rate.

So in this test you will measure the quantity of water vapour in grams that will be transmitted from one side to the other side of the film. Consider 1 meter square in 24 hours when the relative humidity difference between the two sides is maintained around 90 plus or minus 2 percent at 37 degree centigrade. So, this is the condition.

So, what you will do is you will take these shallow aluminium dishes and the diameter is usually similar to that of a can. You will wax seal it and keep your packaging material between that just above the dish where you will have a desiccant. Now this picture here which is taken from the mentioned source shows a clear picture of how it is done. So these shallow cups here have got the desiccant like calcium chloride that is placed. Your test material that you want to test that is placed over this.

It is sealed and it is kept in an atmosphere which has got high humidity. A humidity of around 90 percent plus or minus 2 degree percent and 37 degree centigrade. So what happens over time is that from the atmosphere water slowly sweeps in or it will penetrate into the desiccant. So you will find out what is the increase in weight of the desiccant. You will find out the initial weight of the desiccant and once it is sealed after 24 hours you will find out what is the increase in weight of the desiccant.

So, this will show you how much water has penetrated or moved into, through the test packaging material. Now the water vapor is, WBTR is determined by sealing the open end of the dish containing the desiccant, usually calcium chloride, by the test specimen. Test plastic is kept over this dish. Inside you will have your weighed desiccant and you will expose the dish to the desired relative humidity and temperature conditions. For the standard test like I said it is 37 degrees centigrade, 92 percent relative humidity.

When the desiccant used exerts just 2 percent of relative humidity. Increase in the weight of the desiccant after a known period of time. You check it after a time. It gives the amount of water vapor that has been transmitted by the specimen. And it is calculated Q which is the quantity of water vapor that has been transmitted in the area, whatever area you have taken for your packaging material, for the time T hours, 24 hours if possible.

And the relative humidity on either side is maintained as H1 and H2. And the sealing wax that you use to seal the ends so that no water vapor will get in through the sides, that is usually a combination of microcrystalline wax and paraffin wax in 60 to 40 ratio. The area test specimens mostly is 50 centimeter square. So depending on the area you can substitute here and find out what is your water vapor transmission rate. Now we will be doing the water vapor transmission rate of the packaging material by the conventional method based on gravimetry.

WVTR or water vapor transmission rate measures the permeability of the packages to water vapor. We first cut the packaging material to the desired size. We take shallow aluminum dishes and add anhydrous calcium chloride which is a desiccant. Cover the dish with a test film. Apply petroleum jelly on the sides of the plate so that it can be easily removed later.

Make the film airtight using paraffin wax and microcrystalline wax in the ratio 60 to 40. This wax is first melted and poured over the plate. This ensures that water vapor will not enter through the sides. Once it sets, lift the top plate. You can see the test film is now firmly fixed over the aluminum dish with the desiccant.

This is the area of sample exposed to the atmosphere. Now take the initial weight. Next place it in a conditioning chamber which is maintained at 37 degree centigrade and 90% relative humidity. Note the increase in weight every day till you get constant weight. The increase in weight per 24 hours gives the WETR of the material in grams per meter square per 24 hours.

There is also a water vapor permeability tester. So all water vapor transmission tests are based on a humidity sensor detection method. Here weight measurement is done or gravimetric method and this measures the gain or the loss in the mass of the desiccant. Basic thing is that a humidity difference is created between the inner and the outer chambers of the cup that holds the sample and this what happens is once it tests around the cup, this will lead to water vapor permeation through the test material. The weight change that occurs is monitored with the software until you get an equilibrated value and this is calculated as the transmission rate.

So, the earlier one which we just mentioned was the conventional method and this is usually has a, you have a tester which will, using a software, it will help you to find out what is the water vapor transmission. The only difference here is mentioned as the conventional method, the sample cup is filled with the desiccant before sealing it with the sample and the increase in weight observed due to the absorption of water by the desiccant under the test condition that is calculated. The rate of absorption is usually a measure of the permeability of the sample. So the more the weight gain, more will be the permeability. Less the weight gain, less will be the permeability or it will be a better barrier property.

Now in the case of the software assisted method which we just mentioned, the sample cup is filled with water and it is sealed with the plastic sample which you are going to test. Then it is placed in a chamber where it gets heated up and so there what happens is

this leads to water vapor formation inside the chamber to around 100% relative humidity while the outer chamber is 90% relative humidity. So there is a 10% difference. So inside it is 100% relative humidity, outside it is 90% and because of this 10% relative humidity in the inner and outside chamber, there is still, there might be a migration dependent on your test sample. The water vapor migration occurs through the sample to the sample cup to the outer layer leading in a weight loss.

So, there is in the software assisted method, there is actually a weight loss which is a measure of the permeability of the sample. So there is an opposite direction in both these. So though the migration is opposite in both the cases in the conventional and in the software method, in both these cases you are going to find out what is the permeability or the barrier property of your test packaging material. We will now look into the video which will show you the demonstration of the water vapor transmission rate. The water vapor transmission rate can also be measured using a water vapor permeability tester.

This is based on humidity sensor detection method and on the weight measurement or the gravimetric method which measures the gain or the loss in moisture. Here the condition sample is first cut. In the software assisted method, the cup is filled with water and then sealed well with the test packaging material. Thank you. It is then placed in a chamber which gets heated up leading to the formation of water vapor inside the chamber, almost 100% relative humidity.

The outer chamber is maintained at 90% relative humidity therefore creating a 10% difference in the relative humidities between the inner and the outer chambers. The water vapor migration occurs through the sample from the sample cup to the outer chamber leading to weight loss which is a measurement of the permeability of the testing packaging material. The WETR value can be directly read in terms of grams per meter square per 24 hours. Moving on to the gas transmission rate. So, we know that many of the products are susceptible to oxygen, carbon dioxide.

Some of the products they might undergo oxidation and might spoil especially your fatty foods. So you want a packaging material which is a good barrier against oxygen. In certain other cases you would like a package to be a little more permeable especially in fruits and vegetables. So gas transmission rate actually measures the rate of gas that passes through the packaging material. The volumetric rates of transmission of the gas under known pressure differential through a known area of film, it is usually expressed as transmission rate in ml per square meter per 24 hours per atmospheric pressure.

Now the permeability of the plastic material to different gases is of considerable

significance. Usually instead of considering for all the gases and making the package completely impermeable it is important to consider what is the permeability of your packaging material to certain gases. Maybe it is for oxygen or how is it impermeable or permeable to carbon dioxide. You go in for specific gases that gives you a better idea based on your product and the package that you want to design for it. Now the phenomena of gas permeability are dependent on one the physical nature of the film.

It depends on its density, degree of crystallinity and the thickness and also on the size and mobility of the gas. So that is why we said if you can do the test on a particular gas it makes more sense because depending on the gas also the permeability might change. The degree of polarity of both the plastic material and the gas as well as the tendency to be either hydrophobic or hydrophilic will influence the permeability to the gas. Two methods in which this can be done, you can use the differential pressure method or the equal pressure method. In this particular diagram which is taken from this particular paper they have used ethylene mineral alcohol but any test sample that is represented by this blue line that you see that will be a test plastic material.

So, there will be two chambers, your inner and your outer chamber. In the differential pressure what they do is the lower chamber it is completely evacuated or vacuum is formed here. Now the upper chamber, the test gas, you can either have oxygen or carbon dioxide or nitrogen, you can have your test gas go in and you will check after particular times what is the increase in upper that gas in the lower chamber. If your plastic material is more permeable you will have more gas permeating through and the level or the increase in volume of that gas in the lower chamber will be higher. But on the other hand, if your plastic material is a good barrier the increase in the gas concentration will be less.

That is one method, another method is called equal pressure method. Here you will allow a particular gas in both the lower and the upper chambers. You might have nitrogen on both the upper and lower chambers and in the upper chamber you will pass in the test gas. Once the test gas is added, again depending on the permeability the concentration of the test gas might increase in the lower level. If it does increase that means your test material is a good permeable or it is not a good barrier.

So that will help to find out what is the permeability of your packaging material to different types of gases. In the software assisted apparatus that we use, it is usually based on the differential pressure method that we just saw. The preconditioned specimen that is your plastic test sample that is mounted in the gas diffusion cell form a sealed barrier between the two chambers. You can imagine the two chambers so this forms a sealed barrier between them. The lower chamber is first evacuated and then this is followed by

evacuation of the entire cell.

A flow of gas is then introduced into the higher-pressure chamber, the upper chamber and a constant pressure difference is generated. Now the gas permeates through the specimen, your test plastic material from the higher pressure side to the lower pressure side. Gas permeation can be obtained as monitoring the pressure change. As the pressure increases that means more of the test sample is permeating through the plastic material to the lower chamber. That gives you an idea of the gas permeability rate of the plastic test material.

Let's look into the video which will demonstrate the same for you. First, we will be doing the Oxygen transmission rate. In this software-assisted apparatus, Oxygen transmission rate is measured on the basis of differential pressure method. The test sample is first conditioned and cut. Filter paper is also kept along with the sample so that any moisture that may be present in the test gas may be absorbed by the filter paper, thus preventing entry of moisture into the chamber.

This is the OTR tester attached to the oxygen cylinder. Grease is applied along the sides to make it airtight. The preconditioned test specimen is mounted in the gas diffusion cell so as to form a sealed barrier between the two chambers. The lower pressure chamber is first evacuated followed by evacuation of the entire cell. A flow of gas or oxygen is introduced into the evacuated higher-pressure chamber and a constant pressure difference is generated between the two chambers.

The gas permeates through the test sample from the higher-pressure side into the lower pressure side. The gas permeability of the specimen can be obtained by monitoring the pressure change. The tester directly gives the values in  $\text{cm}^3$  per  $\text{m}^2$  per 24 hours. Now that we have gone through the water vapour transmission rate and gas transmission rate, they are both indications of the barrier properties of the packaging material. Let's go into another factor which is overall migration.

Now what is migration? It is the mass transfer material from the plastic to the food under specified conditions. Migrants are materials thus transferred from the plastics to the food. If you want to assess the toxic effects of these packaging materials, you have to go in for short term extraction test which is known as global migration test. For quantifying the migration, you want to see how much of the migrants have come from the packaging material and the test is known as a global migration test. It is difficult to estimate and quantify the amount of migrants in actual foodstuff because of the complex nature of the food.

You might have all kinds of different components in the food. So generally in the food industry, they usually use food simulants. What are food simulants? These are chemicals which will mimic the actual liquid in which it is placed. For example, if you have food which has got major as water, so your food simulant is going to be water or very light or dilute alcohol.

Suppose your food is an acidic food like pickles. So vinegar is the most important major component in the pickles. So here the food simulant will be, you can use acetic acid to simulate the vinegar atmosphere. So basically simulants are used instead of the whole complex food system. Accordingly, the global migration test, it will test gravimetric that will measure the ink weight or the amount of migrants from the plastic to the different food simulating systems. And this is based on the categorization of food which is mentioned as per IS98451998.

The two types of migration, one is called global migration and one is specific migration. In global migration, it actually measures the total transfer of the quantity of substances migrating from the package into the packaged food. It's the total, all the components that are migrating from the package to the food is measured in global migration. On the other hand, in specific migration, one or more identifiable substances has to be measured. You're not going to measure all the substances that are going to migrate.

One or two specific components, what are these quantities that is migrating into the food, that is done in specific migration. This is again a pictographic representation of this. You have your plastic food packaging and this is added into the food simulant. It can be alcohol, it can be vinegar, acetic acid, it can be water. And once you put them in, what happens if you keep them in 24 hours? You would expect some of the components to migrate from the packaging material into the simulant.

So, you'll see what is the amount that has been migrating. If you want to specifically see what is the quantity or what is the component, you will go for GC or LC testing to specifically identify these components. Let's look at the overall migration simulants that are used. It's based on the European Commission 2002. Here, look at the food simulant. 10% ethanol is being used for aqueous food and that abbreviation is food simulant A.

A food simulant B will be 3% acetic acid, dilute acetic acid, which is usually used to mimic aqueous and acetic foods like your pickles and your aqueous foods you can use. Slightly acetic foods around pH 4.5, you can use 3% acetic acid. On the other hand, if your food is alcoholic but less than 20% alcohol or even fatty foods, you can use food simulant C, which is 20% ethanol. For fatty acids and alcoholic foods which have got more than 20% alcohol or even for emulsions like oil and water emulsions, you can use

food simulant D1, which is 50% ethanol.

High concentration, it's more concentrated. Now for fatty foods, which is food simulant D2, you can use vegetable oil with less than 1% unsappable matter. And for dry foods, generally we do not need a global migration test but for specific migration testing you can use food simulant E, that is poly 2,6-diphenylparafenylenoxide. What's the method? How do you do this global migration? Generally what they do is fill the container of the pouch that you want to test. They will fill it to the filled capacity with preheated simulant, whichever liquid like we mentioned that is filled into it at the test temperature.

And then they will close it and expose it to specified temperatures. For example, retort, if your package actually has to go through retorting, they will pass this also to a high temperature. If it has to go through another type of temperature changes, it has to go through this test also will go through that temperature. Now after it is exposed to that specified duration, you will remove the container and you will quickly transfer the extract in the simulants that you have added into that into a clean beaker with three washing of the simulant. Once you have completely taken it out, you evaporate the extract it to about 50 to 60 ml and transfer that into a cleaned weighed jar. And then you evaporate it completely to dryness at an oven at 100 degrees centigrade.

Now what happens, you have the empty weight dish and you have an increased weight if the migraines are there. You cool it, you weigh it. Because you do see an increase in weight, it means migraines have leached from packaging material into your simulant. Calculation overall migration rate, mass of the residue or the increase in weights that you see into 100 divided by the area that's exposed. That gives you the overall migration rate milligrams per decimeter square.

On the other hand, mass of the residue into 1000 divided by the volume of simulant that you have taken, that will give you milligrams per liter. The maximum value of migraines, the overall migration rate is 60 milligrams per liter or 10 milligrams per decimeter square. As per the source, this is the above the conditions that you use at different temperatures. For example, suppose you're a real food, you use high temperature and heat sterilization.

If its water based, you use 121 degrees for two hours. If it's acidic, use 3% acidic acid, 121 for two hours. Again, it's a high fat food, you can use n-heptane, 66 degrees for two hours. Similarly for each type of foods, you can suppose there are products which are room temperature filled and stored in refrigeration. What you use if it's a water based, you do the test at 40 degrees centigrade for two hours.

If it's an acidic based product, use acetic acid at 40 degrees for two hours. So similarly, this table can be used to find out the conditions in which the pouch can be kept during the test. Now in 2019, India has announced a new packaging rule. The Food Safety and Standards Authority of India announced that the overall maximum migration limit of 60 milligrams per kg or 10 milligrams per decimeter square and specific migration for certain contaminants in food plastic packaging. So these are to be adhered to in all packaging materials in India. There are also some key provisions that are given under the new packaging regulation.

It prohibits packaging of materials in recycled plastics, including carry bags. That is not allowed. It prohibits the use of newspaper and other such materials which carry ink and dyes for use in food packages. It also gives the overall and specific migration as I've just mentioned. And the packaging food products, they shall confirm to all the Indian standards and all food businesses supposed to comply to this by July 2019. So I hope you've got a fairly good idea of the different tests we mentioned today, which gives an idea of the barrier properties of the packaging material, including your water vapor transmission rate, gas transmission rate, and your overall migration test. Hope to see you in the next class. Thank you so much. Thank you.