

Admixtures And Special Concretes

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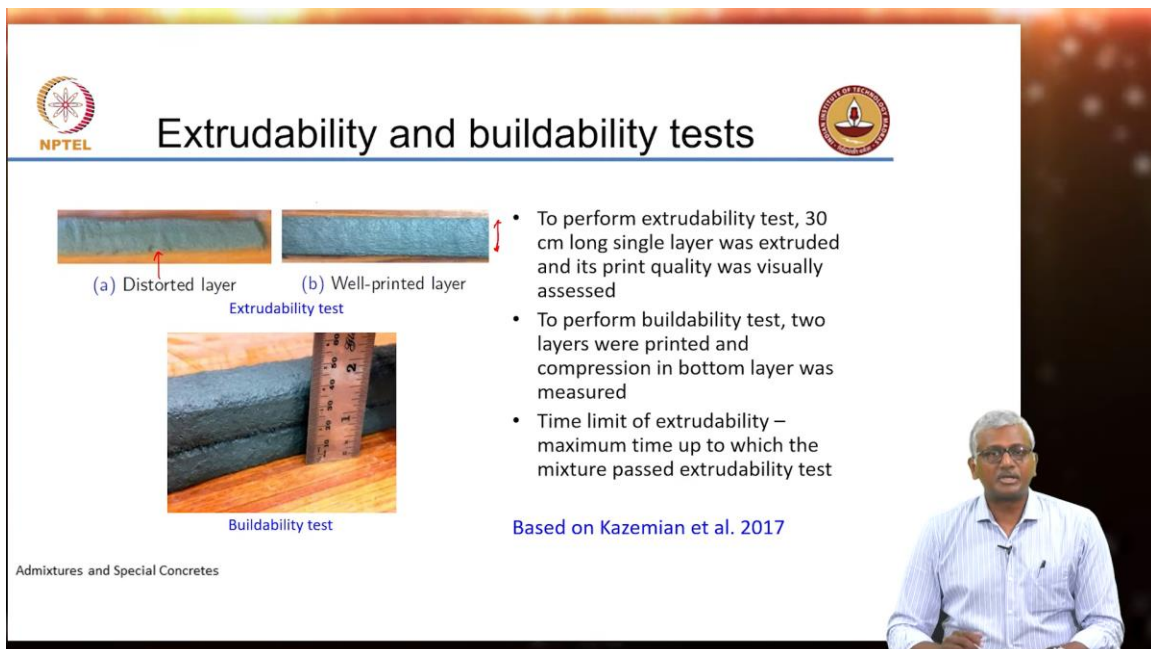
Indian Institute of Technology Madras

Department of Civil Engineering

Lecture -75

Special concretes - Concrete for 3D printing - Mix design approach, admixtures,

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Extrudability and buildability tests

(a) Distorted layer (b) Well-printed layer

Extrudability test

Buildability test


- To perform extrudability test, 30 cm long single layer was extruded and its print quality was visually assessed
- To perform buildability test, two layers were printed and compression in bottom layer was measured
- Time limit of extrudability – maximum time up to which the mixture passed extrudability test

Based on Kazemian et al. 2017


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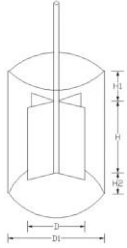
Okay, so let us resume our discussion on 3D-printed concrete. We are talking about different types of simple tests that we could do to assess the extrudability and buildability. So, this was later seen whether we could produce a robust mixture with 3D printed concrete and what were the characteristics that influence that robustness.

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Robustness based on yield stress






Soil vane shear apparatus

- Stress growth experiments by using soil vane shear apparatus to determine yield stress
- To quantify robustness, the SP dosage was changed by $\pm 0.01\%$ and $\pm 0.02\%$
- The variability factor, V is given by:-


$$V = \sqrt{\frac{\sum (YS - YS_{ref})^2}{N}}$$




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So, as I was defining in the last class, robustness is basically mentioned as the ability of the mix to still meet its demands in terms of workability and strength even if there is a small variation in the design characteristics. So, for instance here in 3D-printed concrete, we can assume that the robustness can be affected by the dosage of the superplasticizer. So, in this case, we fixed a central dosage and varied the SP dosage above and below that level. And the variability factor is essentially another way to represent the standard deviation and that is how it was determined when we changed the dosage from the central dosage to above and below that central dosage. And the yield stress was measured from the vane shear test in this case.

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Mix design approach




Mixture proportion

Material	Quantity (kg/m ³)
Cement	663.0
Fly ash	165.7
Quartz powder	497.2
Quartz sand 1	372.9
Quartz sand 2	372.9
Water	265.2
Fibers	1.8
SP	0.83 (0.10 %)

Binder : Aggregate = 40 : 60

Rahul et al. 2019

- Granular skeleton optimized by using a particle packing model
- SP dosage based on extrudability and buildability test
- Polypropylene fibers were used at a nominal dosage to prevent plastic shrinkage cracks
- To increase packing density, two grades of quartz sand and quartz powder used as FA
 - Sand 1 (max: size = 2 mm)
 - Sand 2 (max: size = 1 mm)
 - Quartz powder (max: size = 60 μ m)




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So, the actual mix design that was used for the base mix involved 660 kilograms of cement, about 160 fly ash, quartz powder was used as filler, and quartz powder was provided in sizes between 5 and 25 microns, essentially used as filler, very fine filler and in terms of the coarse aggregate we did not have anything beyond 2 millimeters. So even the fine aggregate was controlled to be of size less than 2 millimeters. So, two sands, both are quartz sands, plain pure quartz sand and from 2 to 1 millimeter and 1 millimeter and below. The total binder-to-aggregate ratio, binder included cement and fly ash, the binder to aggregate ratio is 40 to 60 or 1 is to 1.5.

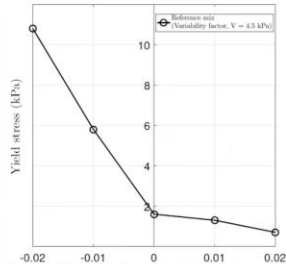
Now here of course the quartz powder which is a very fine aggregate is also considered to be a part of the aggregate, not part of the paste. Fibers were used, polypropylene fibers were used because you have to lay this concrete in the open so there is obviously a great danger of plastic shrinkage because early drying can happen from the surface and polypropylene fibers will tend to reduce the extent of drying shrinkage. Then superplasticizer was a polycarboxyl ether-based, PCE-based superplasticizer and this was determined based on what mix gave a good extrudability and weldability. The mix was able to extrude well so we fixed the superplasticizer dosage at 0.1% by weight of the cementitious materials. And the aggregate packing was optimized by using the granular packing model which we have discussed previously.

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Lack of robustness in reference mixture





Reference mix (Variability factor, V = 4.5 kPa)

Yield stress (kPa)

Change in SP dosage from that of the reference mix (%)

Robustness test

Table: Extrudability test


SP dosage (%)	Extrudability test
0	Fail
0.02	Fail
0.04	Fail
0.06	Fail
0.08	Fail
0.10	Pass

Table: Buildability test

SP dosage (%)	Bottom layer comp. (mm)
0.10	0
0.11	Collapse

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- The yield stress for reference mixture was 1.6 kPa
- The yield stress changed considerably with even a slight change in SP dosage
- High variability factor, V = 4.5 kPa indicating lack of robustness
- Mixture was found to fail either extrudability or buildability test with even a small change in SP dosage




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So, in this reference mixture, there was an absolute lack of robustness. So, if the dosage was exactly 0.1% it was passing the extrudability test and it was passing the buildability test also. There was no deformation in the bottom layer. But going less than 0.1 made the mix fail the extrudability test which means the filament that came out did not have a very

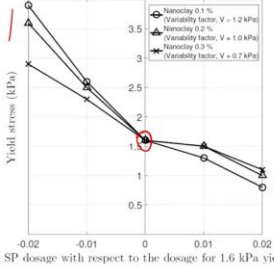
clear geometry it was distorted. So, it did not pass the extrudability test and when the SP dosage increased beyond 0.1 the mix was not able to retain its shape it collapsed. So, that means it is failing in the buildability test. So, in other words, when you make a design like this without any additional additives with just a superplasticizer it is not robust enough.

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Addition of nanoclay






Improved robustness with nanoclay addition

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- Nanoclay @ 0.1, 0.2 and 0.3 % of binder content
- For each nanoclay content, SP dosage was adjusted to obtain the same yield stress of reference mix (1.6 kPa)
- Yield stress was found to vary less with nanoclay addition
- **Variability factor decreased from 4.5 to 0.7 kPa with 0.3 % nanoclay addition**

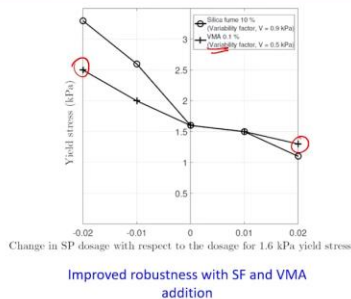


So, to increase the robustness we added some nanoparticles like nano clay for instance. Here nano clay is added at 0.1, 0.2, and 0.3% you can see that there is still variability as compared to the central dosage. But if you compare here (refer to reference mix results) the variability is significant it goes all the way from 1 to 10 whereas here (refer to mix with nanoparticles) we are talking only about 1 to about 3, 3.5. So, it is not really changing much. The yield stress is not changing much. All these mixes passed with respect to extrudability and buildability. So, the variability factor was decreased from 4.5 which was in the case of the reference mix to about 0.7 kilopascals.

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Additions of VMA and SF



- SF @ 10 % and VMA @ 0.1 % of binder content
- SP dosage was adjusted to obtain the same yield stress of reference mix (1.6 kPa)
- **Variability factor decreased with both SF (0.9 kPa) and VMA (0.5 kPa) addition**

Improved robustness with SF and VMA addition


Rahul et al. 2019

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And with VMA and silica fume similar results were obtained with the VMA the best results were obtained where the range was only from 2.5 to 1.4. So, the variability factor was down to about 0.5 kilopascal when VMA was added to the system. So, as we have discussed this before also VMA controls the viscosity of your concrete well and in this case, it was able to provide much better robustness. It could help the mix accommodate a slight difference in the superplasticizer dosage. So, what I wanted to say is you get the flowability or extrudability with the help of the superplasticizer but in order to ensure that you are able to make the mix stable and buildable you need an additional ingredient perhaps very fine material like silica fume or you may need something like a VMA that can adjust the viscosity to such a level that you are able to build up significantly.

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Flow table test results




Flow table test (ASTM C1437-15)

Mix name	Yield stress	Flow value (%)
VM	1.6	80
SF	1.6	80
NC	1.6	95

- Mix with nanoclay showed maximum flow value
- Flow value can depend on both the plastic viscosity and yield stress (Ferraris, 1999; Bauer et al., 2007)
- Since all the three mixes have the same yield stress, the lower flow value indicates that NC has a lower plastic viscosity




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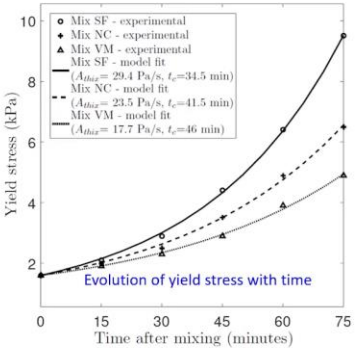
Now you can also do simpler tests with this material and correlate that with the rheological tests. So, for instance, a flow table test will help you understand the flow diameter and the flow diameter can be then related to the yield stress. You can see that the flow value between 80 to 100 percent was equivalent to the yield stress that we had in the reference mixture.

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Structural build-up with time





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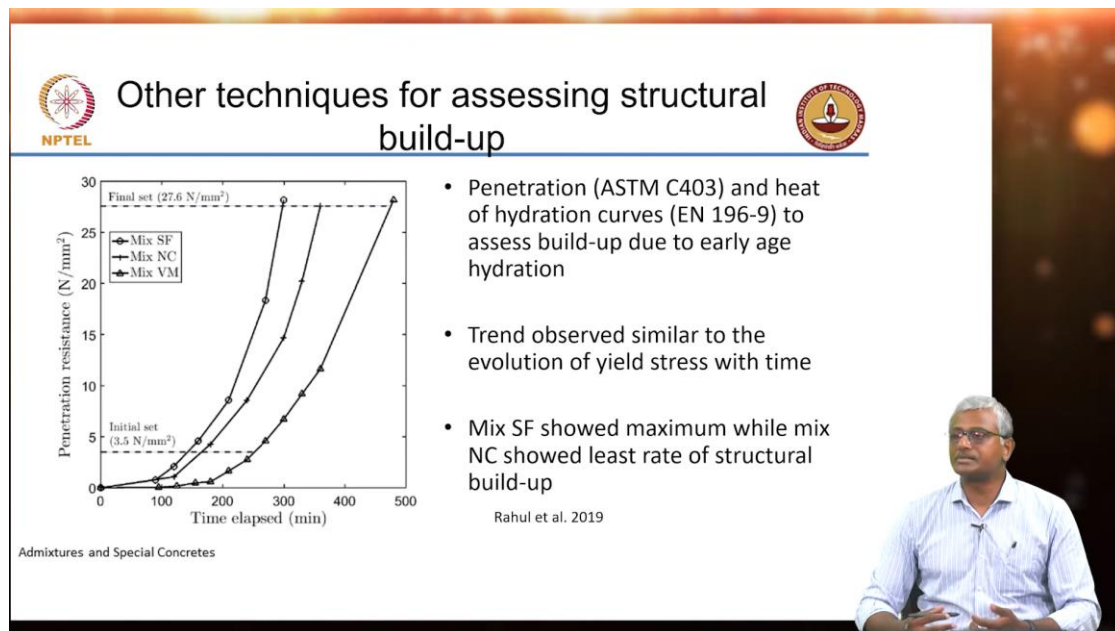
- Yield stress determined after different rest duration
- Experimental data fitted well with the exponential model proposed by Perrot (2015)
 - ▶ $\tau_y(t) = A_{thix} t_c \left(e^{\frac{t}{t_c}} - 1 \right) + \tau_{y,initial}$ ←
- More studies are needed to assess the range of model parameters needed for 3D printable mixtures



And for all these mixtures the structural build-up, and internal build-up we discussed earlier how this internal build-up is important because the internal yield stress has to build up to a level that is able to overcome the stress due to the weight of the additional layers that come on top. So, we have to understand this behavior also, and as I was telling you earlier there are non-linear rheological models which can help you fit this equation also.

So, you can see that the data that have been experimentally obtained by measuring the yield stress at different times seems to match quite well with the model predictions that can be used from the literature. So when you want to design 3D printed concrete it is not just enough to measure the flow value or the yield stress you also need to determine what its behavior is likely to be with time so that you can plan your operation better in terms of when should you bring the next layer on top, should you wait for a certain period of time, when should the third layer come right all of those things need to be understood clearly.

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And you can also instead of using a vane shear to determine yield stress and measure the build-up with respect to yield stress, you can also substitute that with a simpler technique like penetration, which we use for setting time determination, penetration resistance test could also be used for determination of how 3D printed materials are building up an internal stress.

(Refer to slide time: 08:02)

The slide is titled "Mechanical characterisation" and features the NPTEL logo on the left and the Indian Institute of Technology Bombay logo on the right. It contains three main images: 1) A layered specimen being tested in a flexure machine, with a red line indicating the crack path. 2) A cylindrical core taken through two layers, with a red circle highlighting the interface. 3) A mini wallet specimen being tested in a compression machine. Text on the slide includes: "Layered specimen - flexure", "Layered specimens show 10 – 15% lower strength compared to cast specimens", "Interface strength – core taken through 2 layers", and "Compressive strength 70 – 80 MPa". A man in a light blue shirt is visible in the bottom right corner of the slide.

Layered specimens show 10 – 15% lower strength compared to cast specimens

Compressive strength 70 – 80 MPa

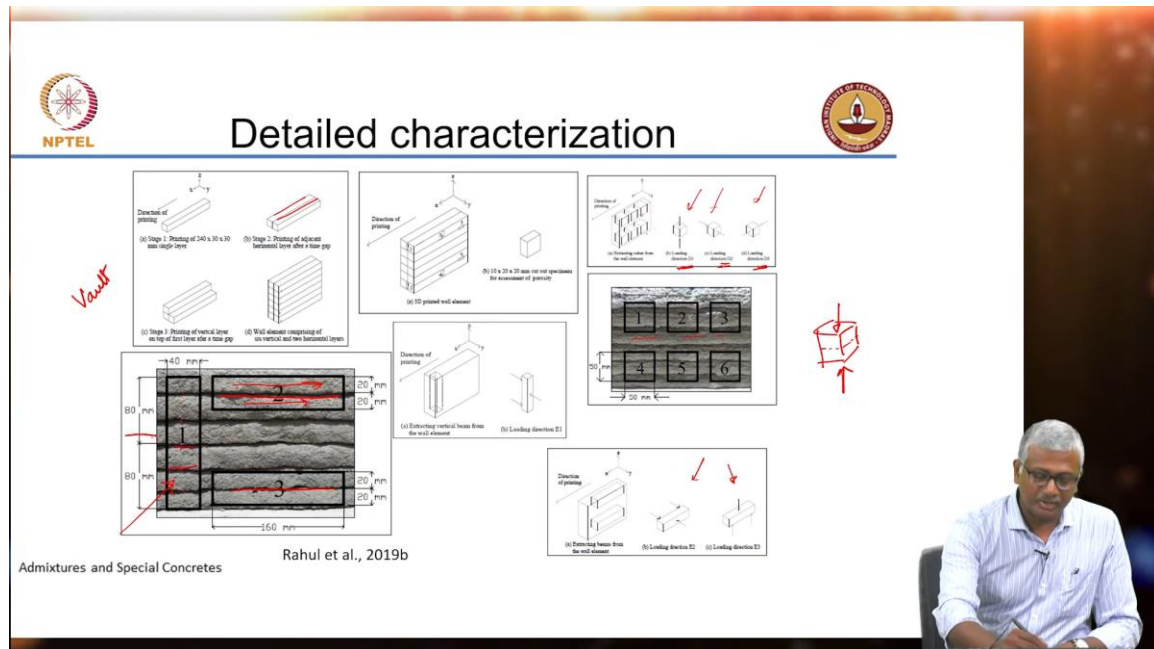
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Now the difficulty comes in the mechanical characterization of 3D printed materials because it is no longer going to be possible for you to characterize the strength just based on a cube or a cylinder.

You need to ensure that you are able to bring in the effect of this layering in your test. So, for that we attempted various different configurations for instance this mini wallet which had 4 layers, so 3 interfaces between them, was tested in compression this was one of the ways. The other one was a shear test where a core was taken through the 3D printed material and then the core was fitted in this direction in this modified shear apparatus and then it was simply pulled apart so that it would break exactly at the interface. And that would give a determination of the bond strength between the 2 layers. And of course, the flexural test also indicates that while the failure is like normal concrete, there seems to be some deviation at the layer so there is some passing of the crack at the layer.

So, overall it was seen that the layered specimens were seen to show about 10-15% lower strength than specimens that were mould cast that were put in the moulds.

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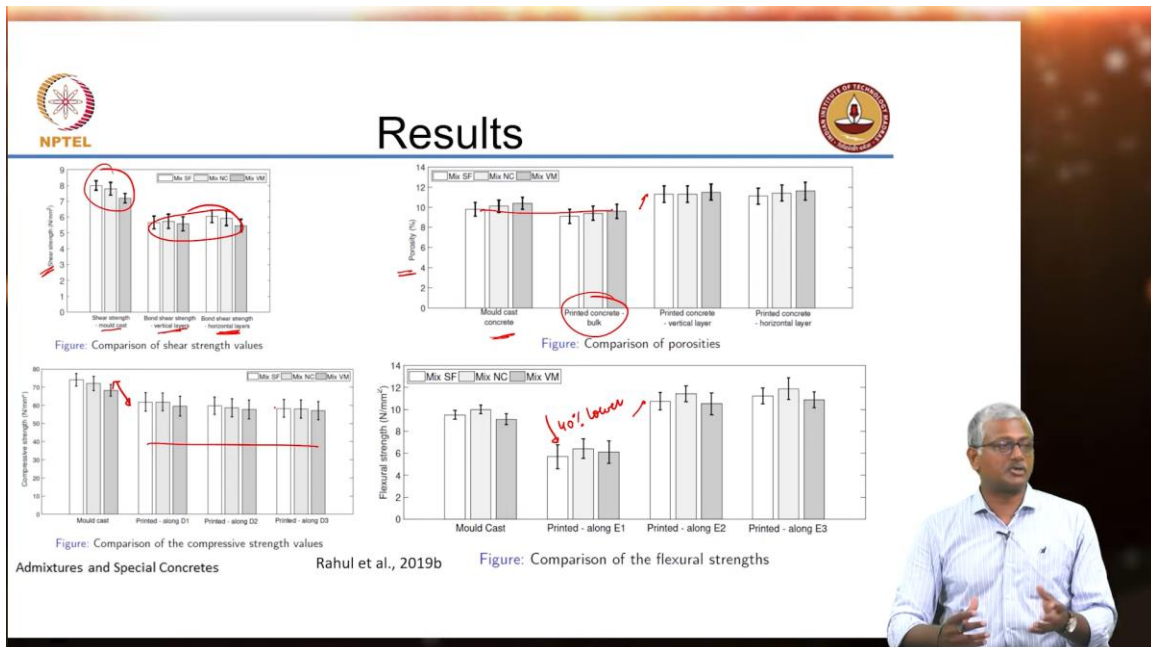
However, this was not the end of the mechanical testing. We also went in for a detailed characterization. So, in this case, the prints were made in this way, we had like 2 horizontal layers and then stacking on top we had 6 total vertical layers that were cast (refer to the top left figure in the above slide), so this was a vault type specimen that was made and from this specimen, we extracted specimens for testing. So, for instance, we extracted cubes like this (Refer to the top right figure in the above slide) we cut the larger printed specimen into cubes of 40 mm size and these cubes were then subjected to loading in different directions because in one direction the interface may not be very crucial. For instance, if your cube is like this and the interface lies exactly in the center and you are loading it in this direction that interface may not really have a major effect on the overall strength. So, we want to understand whether our assumption of concrete being isotropic with respect to strength is true or not.

Is that true? Concrete is isotropic with respect to strength? Normal concrete. No, why? Yes, concrete is isotropic. Does not matter which direction you test it should give you the same strength. Of course, because of the preparation methods your strength could be different because your cube you are casting in one direction you do the testing in the other direction just to ensure that you do not have to prepare the specimen properly. Otherwise, in a cylinder, you can only test it in one direction. So, you have to prepare the top by grinding it or capping it, and so on. So, a cube gives you the flexibility of not having to prepare the specimen.

Similarly for flexure also prismatic specimens are obtained from the vault. These prismatic specimens (refer to the bottom left figure in the above slide) were different for

instance these type 2 and 3 specimens exactly had the interface in the center. Whereas the type 1 specimen had interfaces at several locations. So now which specimen do you think will give you a poorer result in flexure? So, when you are going to do a flexural test you are going to be obviously loading it using a midpoint or third-point loading. So which specimen will give you? One obviously because there are so many interfaces so when you are actually loading it in this direction the crack will easily go right through the interface. It does not have to really go into the bulk material at all.

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So, the results are shown here. So, we tested the shear strength with the methodology that I showed you previously. So, shear strength of mould cast, shear strength through vertical layers, and the horizontal layers. You can see that is not much different. What are the vertical layer and the horizontal layer? The bond which is there in the vertical direction between the 2 filaments is the vertical layer and the bond which is there in the horizontal direction is the horizontal layer.

Whereas the mould cast specimen gave you a higher bond strength. But then you can see the decrease is only of the order of about 20 to 25%. It is not significantly large. In terms of porosity, we collected small chunks of concrete from near the interface and from the bulk and compared them to the mould cast concrete. When we took printed concrete from the bulk the porosity was nearly similar.

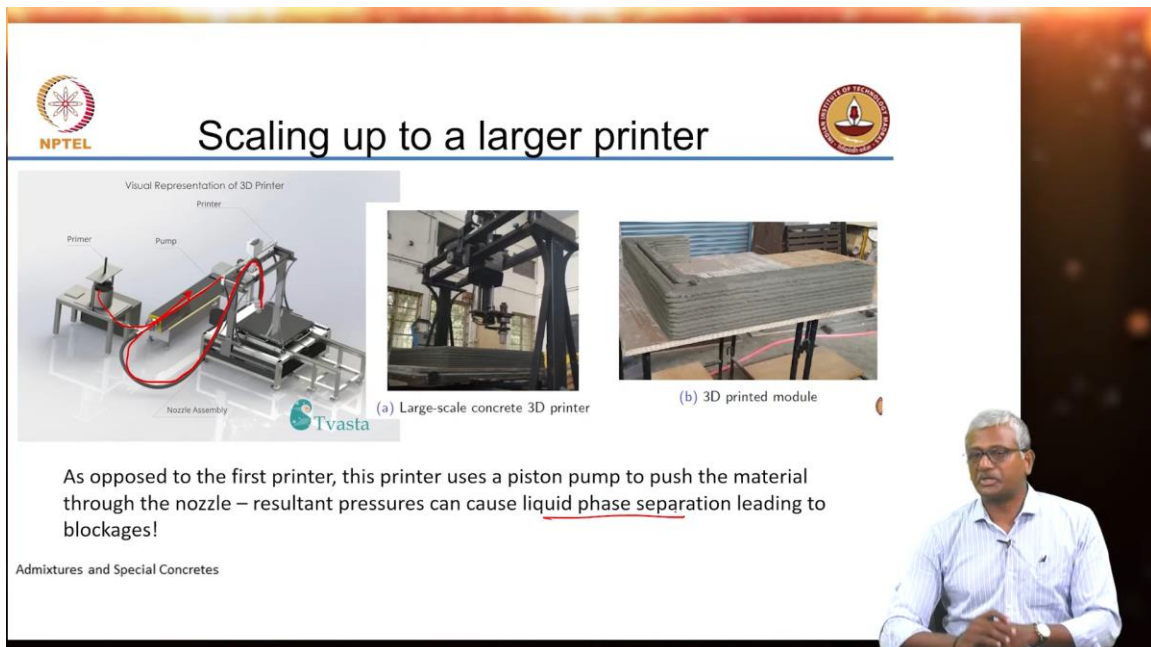
But when we took it from the interface there was an increase in the porosity. So, interface is a region where there are more voids present. Even though we have printed it almost without any much time gap so that there is no setting happening still because of the

interfacial effect there is some porosity that is getting included here. Now in terms of compressive strength, you can see that whichever direction you test it is giving you the same value but as compared to the mould cast specimen it is lowered by about 10%, 10 to 15%, not that much more. The interesting thing about flexure is that when you print along that E1, so when you test it in this direction, you are getting a strength which is about 40% lower.

Whereas when you test it in the other two directions you are actually getting a marginally higher strength as compared to the mould cast material. Why? As you can imagine when you are testing this material the failure crack will start coming at the bottom and the failure crack is coming in the bulk of the 3D printed material. The material is passing through a nozzle so there is extra compaction that is happening in the center of the material. So that is why there is a marginal enhancement in the strength of the concrete as compared to the mould cast system. The second reason is we have put fibers in this mix so when you are doing mould casting, the fibers do not necessarily align themselves they are randomly distributed but when you are extruding the fibers will tend to align in the direction of extrusion.

So that may also be the reason why you have a higher strength with E2 and E3. So, what up short of it is that it is not easy to determine a mechanical property for 3D-printed concrete you need to somehow bring in the effect of the interface. What kind of specimen you choose will determine what type of strength result you actually get. So, all this is part of a big process today which is being undertaken in several labs across the world to deal with how we can actually create standards for testing 3D-printed concrete.

(Refer to slide time: 15:21)




The slide features the NPTEL logo on the top left and the Indian Institute of Technology Bombay logo on the top right. The title "Scaling up to a larger printer" is centered at the top. Below the title, there is a diagram of a 3D printer with labels for "Printer", "Pump", and "Nozzle Assembly". To the right of the diagram are two photographs: (a) a large-scale concrete 3D printer in a factory setting, and (b) a 3D printed module consisting of several stacked layers of concrete. Below the photographs, there is a text box that reads: "As opposed to the first printer, this printer uses a piston pump to push the material through the nozzle – resultant pressures can cause liquid phase separation leading to blockages!". At the bottom left, it says "Admixtures and Special Concretes". In the bottom right corner, there is a small inset video of a man in a light blue shirt speaking.

So, from that small printer that I showed you, we scaled up to a larger printer which was capable of printing up to about half meter height specimens.

We could print up to about half a meter in height. So, this printer had a slightly different way of working. So here the concrete was actually pushed from a primer into a pump and then the pump pushed the concrete out through a hose to the nozzle and the nozzle ended up doing the print. Now in the previous system if you remember it was a simple delivery system. We put the material inside and this screw basically pushed the material to the nozzle and then it printed.

So, the mix that you printed and the mix that came out that you got out of the nozzle were similar in the case of the simpler printer that I showed you previously. But here is what is happening, your pump has to pressurize and push the concrete. Your concrete is travelling through a length of the hose so it will lose some workability. The pressurization further creates an opportunity for segregation to happen because when you pressurize the water may separate. So, in addition to the test that I described previously, you may need to add on one more test to understand the liquid phase separation because of pressure and that is what we undertook to do in this case.

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Printing in progress

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So, you can see the printing is in progress here and this structure is there in BSB. The structure that was created by printing these modules and assembling them, stacking up like a masonry structure, this is there in BSB. I do not know how many of you have seen it. It is between the front and the back wings out in the open.

(Refer to slide time: 17:00)

The slide, titled "Desorptivity approach", features the NPTEL logo on the left and the Indian Institute of Technology Bombay logo on the right. It includes a photograph of the experimental apparatus on the left, a schematic diagram of the filtration cell in the center, and a graph on the right. The schematic shows an "Argon gas" inlet at the top, a "Filtration cell (79 mm dia. and 85 mm height)" containing "cement mortar", and a "Bleed water" outlet at the bottom. The graph plots "Bleed water (mm)" on the y-axis (0 to 10) against "Square root of time (s^{1/2})" on the x-axis (0 to 20). Three data series are shown: FA_1 (0% HPMC) with a steep slope, FA_2 (0.125% HPMC) with a moderate slope, and FA_3 (0.25% HPMC) with a shallow slope. A red handwritten note "Hydroxy Propyl Methyl Cellulose" is written across the graph. The text "Terzaghi's approach from soil mechanics used" and the equation $i = R \sqrt{t}$ are also present. A presenter is visible in the bottom right corner of the slide.

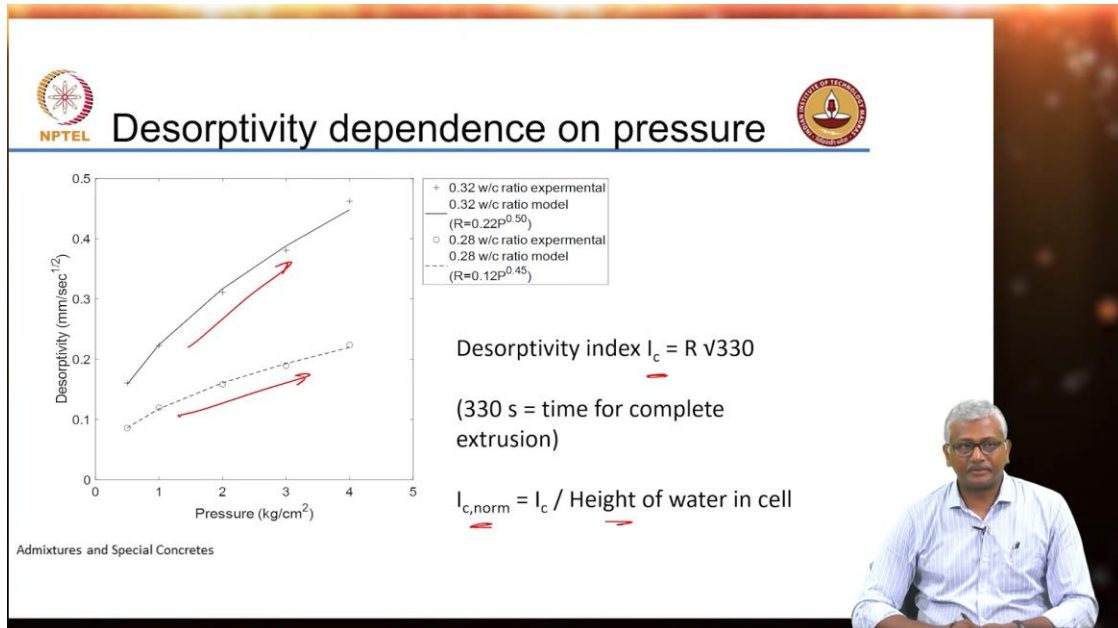
So, we adopted this test methodology which is typically used for water retentivity of mortars.

So, what we simply do is in the water retentivity test for mortars what is done is you take a fresh mortar, you put it in this sort of a cell where you can apply pressure using gas. So when you apply pressure using gas if the water inside is not held tightly it will start bleeding out. So, you have bleed water you can collect at the bottom. In the case of mortar what is done is after the test the same sample that is inside the cell is taken and you study the flow properties. It should not be much different as compared to the original flow.

That is how you define a good quality mortar for plastering or binding mortar applications. Here what we did was we took the same test and we then assessed the amount of bleed water that was getting collected and plotted it with respect to the square root of time to get a coefficient for each of these mixes. For instance, here you have the effect of a VMA which is being tested. VMA here is HPMC, hydroxypropyl methylcellulose. That was the VMA that was used at different dosages as you can see and you see that the extent of bleed water with time basically comes down significantly and if you plot, take the slope you can call that as the desorptivity coefficient.

The slope of this curve is the desorptivity coefficient. Obviously, this coefficient will depend on how much pressure you are applying. You can take a realistic value based on the pumping scheme that you have and apply that pressure or you can study it with respect to different types of different amounts of pressure that you apply.

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So, here the desorptivity coefficient is obviously dependent on the pressure. As the pressure increases the coefficient also increases. So, based on this value we actually came out with what is known as a desorptivity index which again was taken as R times square root of 330 where 330 seconds was the time for complete extrusion of the mix. And then we normalized the entire value that is obtained here by dividing by the height of water in the cell.

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Results for different binders

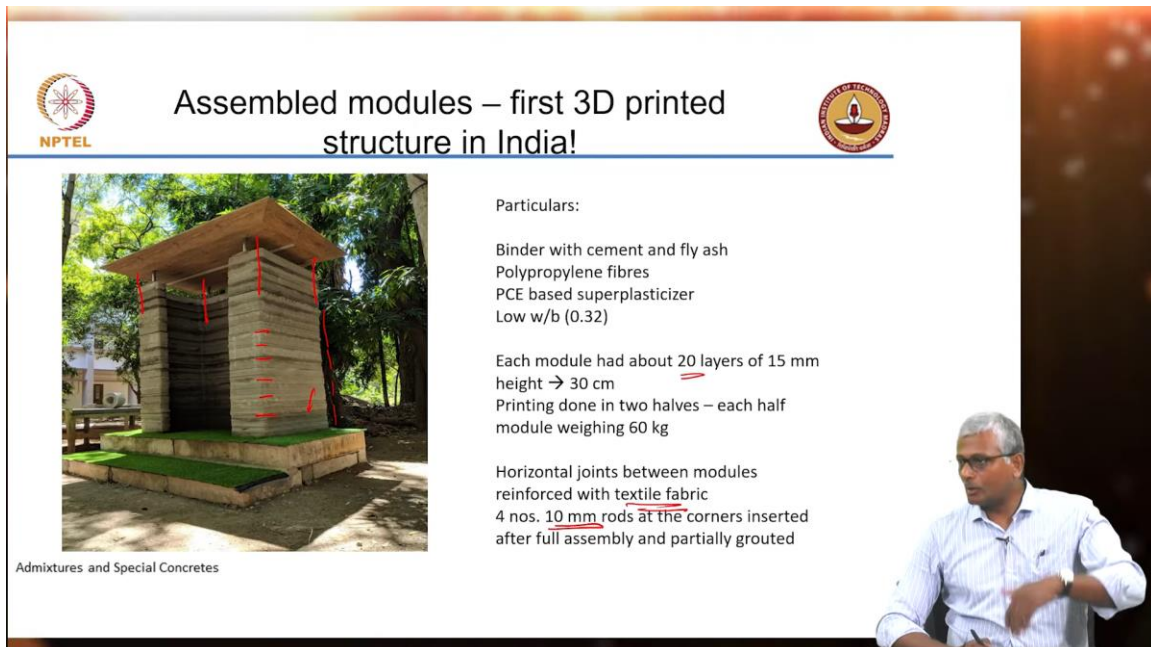
w/c ratio	Mix name	Extrudability (pass/fail)	Desorptivity - pressure relationship	Index (I_c)	Normalized index ($I_{c, norm}$)
0.32	FA_1	Fail	$R = 0.22P^{0.50}$	7.99	0.40
	SL_1	Fail	$R = 0.16P^{0.41}$	5.13	0.26
	LC_1	Fail	$R = 0.05P^{0.39}$	1.56	0.08
	FA_2	Fail	$R = 0.02P^{0.69}$	0.95	0.05
	SL_2	Pass	$R = 0.03P^{0.15}$	0.67	0.03
	LC_2	Pass	$R = 0.02P^{0.29}$	0.54	0.03
	FA_3	Pass	$R = 0.01P^{0.95}$	0.68	0.03
	SL_3	Pass	$R = 0.01P^{0.50}$	0.36	0.02
	LC_3	Pass	$R = 0.01P^{0.74}$	0.51	0.03
0.28	FA1_1	Fail	$R = 0.12P^{0.45}$	4.07	0.23
	SL1_1	Fail	$R = 0.07P^{0.25}$	1.80	0.10

Critical $I_{c, norm} = 0.05$; Lightweight expanded clay aggregates – up to 30% replacement for sand – also used successfully

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So, just to cut a long story short we applied this test to various mixes that were extrudable and buildable and we saw that several of those mixes actually ended up failing the extrudability test after this desorptivity was done. So, the idea is that one has to be able to assess the possibility of liquid phase separation when the system for delivering concrete to the nozzle changes. So, it is not required to be done in all the cases but when your system, printing system is changing you need to build an additional safeguard to ensure that the mix that you have chosen will be capable of handling that operation.

(Refer to slide time: 20:26)



The slide features the NPTEL logo on the left and the Indian Institute of Technology (IIT) logo on the right. The main title is "Assembled modules – first 3D printed structure in India!". Below the title is a photograph of a 3D printed structure, which is a small, square, multi-layered tower with a flat roof. Red arrows point to the horizontal joints between the modules. To the right of the photograph is a list of "Particulars:" including the binder composition (cement and fly ash), polypropylene fibres, PCE based superplasticizer, and a low water-to-binder ratio (0.32). It also states that each module has about 20 layers of 15 mm height, totaling 30 cm, and is printed in two halves, each weighing 60 kg. Further details mention horizontal joints reinforced with textile fabric and 4 nos. 10 mm rods at the corners inserted after full assembly and partially grouted. A small caption "Admixtures and Special Concretes" is located below the photograph. In the bottom right corner of the slide, a man in a light blue shirt is shown speaking.

Assembled modules – first 3D printed structure in India!

Particulars:

- Binder with cement and fly ash
- Polypropylene fibres
- PCE based superplasticizer
- Low w/b (0.32)

Each module had about 20 layers of 15 mm height → 30 cm
Printing done in two halves – each half module weighing 60 kg

Horizontal joints between modules reinforced with textile fabric
4 nos. 10 mm rods at the corners inserted after full assembly and partially grouted

Admixtures and Special Concretes

So, this is the structure I was telling you about earlier this is there in BSB still. So, it was printed in modules you can see approximately the heights of modules here. We tried to get each module to have about 20 layers about 15 mm in height so total of about 30 cm per module and then these were stacked one on top of the other using a mortar joint in between to get sufficient bonding between the modules. Some of these mortar joints were also reinforced with textile fabric like we have an active work going on in textile-reinforced concrete and we use the textile fabric to reinforce the horizontal joints. At the end of the cavities that were there, there were 4 numbers of rods that were 10 mm rods that were actually inserted and partially grouted to ensure that they are connecting all the members together and at the same time they are also providing some reinforcement although the structure does not need any reinforcement because it is hardly about 2.0 I think just over 2 meters tall.