

# 3D-Printed Environmentally Friendly Class C Fly Ash-Based Alkali-Activated Mortar

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# Introduction

## ❖ 3D printed concrete (3DPC)

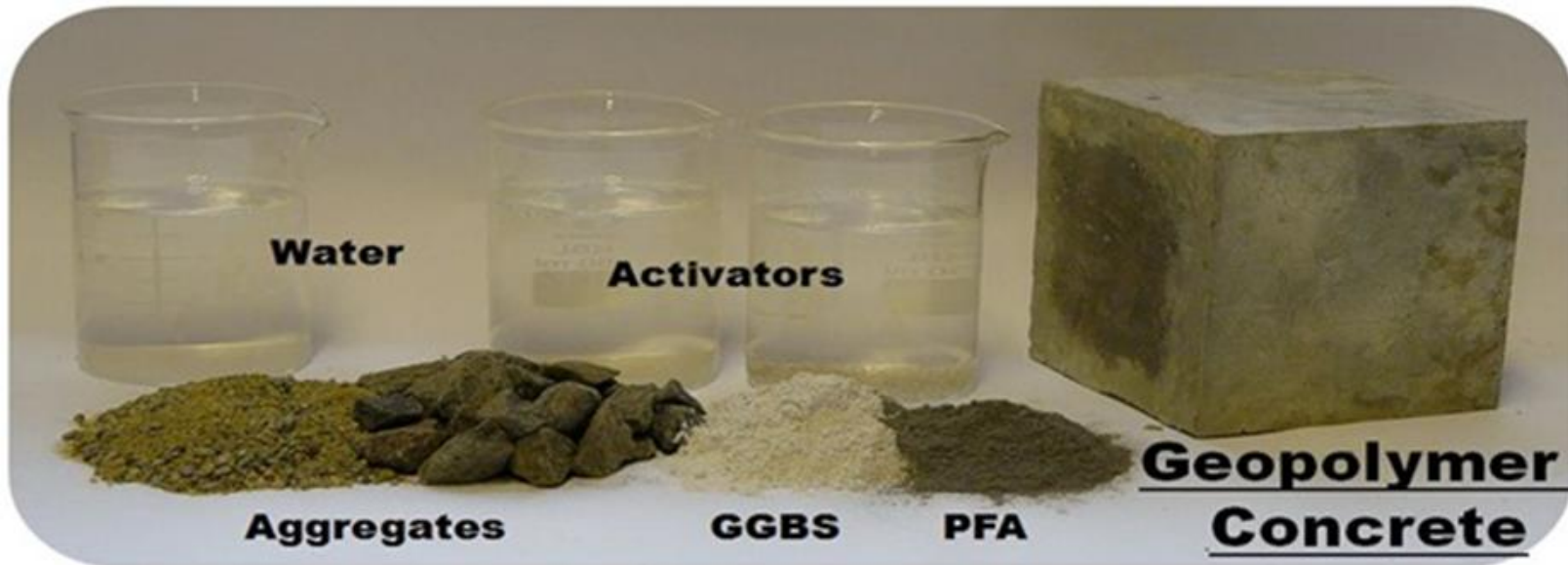
- 3DPC is a new technology used to fabricate buildings or construction components by depositing material layer by layer



# Introduction

## ❖ Alkali-Activated concrete

- Portland cement is responsible for **5-8%** of CO<sub>2</sub> emission.
- Replacing OPC with by-product is an environmental potential solution.
- Alkali-Activated concrete is made from utilization of waste materials such as fly ash and ground granulated blast furnace slag .



# Introduction

## ❖ Fly ash (FA)

- Fly ash (FA) is a by-product of coal combustion in power plants and composed of a combination of coal impurities and flue gases
- The average annual FA production is approximately 55 million tons in the U.S.
- Missouri State is one of the largest generators of FA in the U.S.

### ASTM C618-19

Class F

CaO < 18%

SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>  
>50%

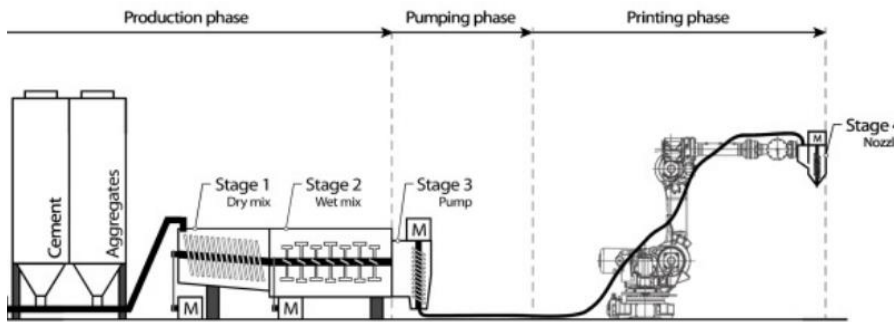
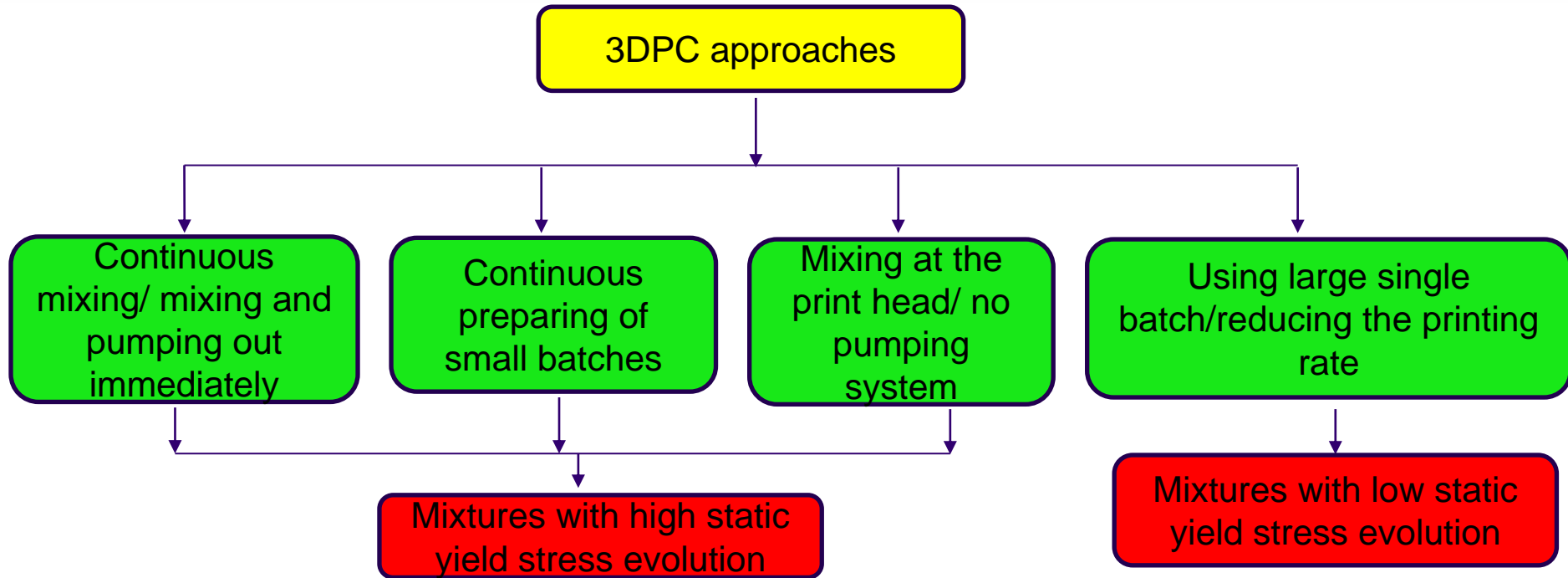


Class C

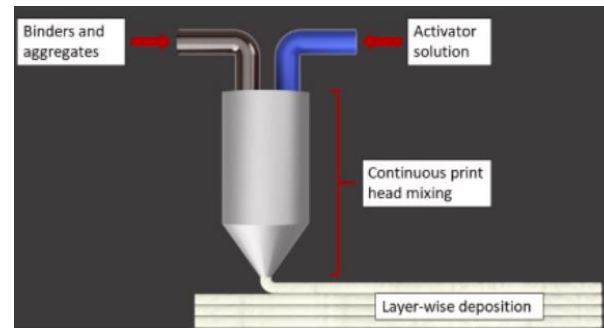
CaO > 18%

SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>  
<50%

# 3DPC procedures



Continuous concrete preparation and pumping  
(Reiter, Wangler et al. 2018)



Mixing at the print head (Muthukrishnan, Ramakrishnan et al. 2022)

# Research objectives

- ❖ Develop a wide range of mixing proportions of class C FA-based alkali-activated mortar (AAM) for different 3DPC approaches including:

# Investigating the fresh properties

## ❖ Material properties

### ➤ Class C FA

Chemical composition using XRF

Composition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	MnO	LOI*
wt.%	36.9	14.0	3.52	37.0	4.80	1.62	0.62	0.87	0.70	0.03	0.50

Physical properties

Specific gravity	Surface area (kg/m <sup>2</sup> )
2.82	2800

### ➤ Sand

Physical properties

Fineness modules	Maximum particle size (mm)
2.25	1.18



# Investigating the fresh properties

## ❖ Material properties

### ➤ Alkali activators



Sodium Silicate (SS)  
55.9% Water  
44.1% Solids



Sodium Hydroxide (SH)  
10M



# Investigating the fresh properties

## ❖ Mix design and mixing procedure

- More than 20 alkali activators mortar (AAM) mixtures were prepared to investigate the fresh properties of 3DPC

**W/FA = 0.36-0.40**

**Alk/FA= 0.25-0.30**

**SS/SH= 0.5-2.0**

### Mix design of AAM

Mix no.	W/FA	Alk/FA	SS/SH	Sand	FA	SS	SH	Extra Water
1	0.36	0.300	1.0	1513	550	82.5	82.5	102.8
2	0.38	0.300	1.0	1499	545	81.8	81.8	112.8
3	0.40	0.300	1.0	1471	535	80.3	80.3	121.4
4	0.36	0.275	1.0	1513	550	75.6	75.6	111.3
5	0.36	0.250	1.0	1513	550	68.8	68.8	119.7
6	0.36	0.300	0.5	1513	550	55.0	110.0	99.8
7	0.36	0.300	2.0	1513	550	110.0	55.0	105.9

# Investigating the fresh properties

## ❖ Open time

- Open time test (OT) → Extrudability



After lifting the cone



One minute



Two minutes



Three minutes

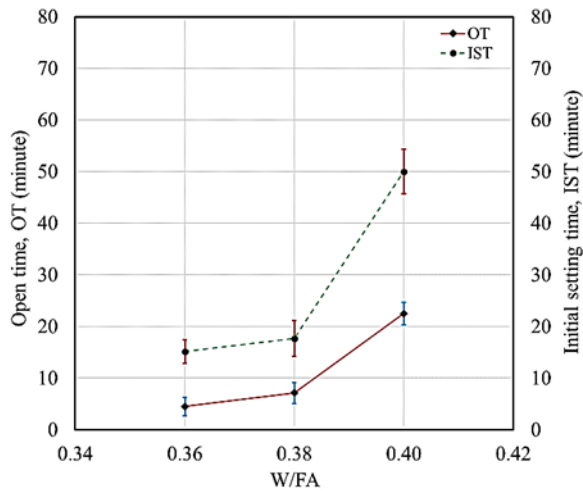
- Initial setting time (IST)

# Investigating the fresh properties

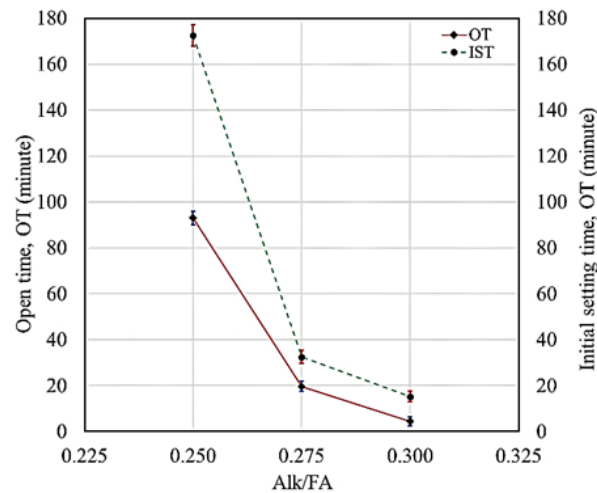
## ❖ Results

- Open time ranged from 3 to 90 minutes.
- Initial setting time ranged from 7 to 170 minutes.

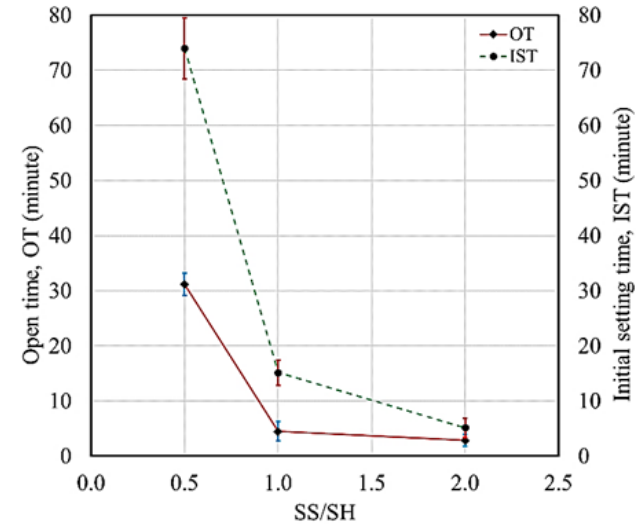
Alk/FA= 0.30, SS/SH= 1.0



W/FA= 0.36, SS/SH= 1.0



W/FA= 0.36, Alk/FA= 0.30

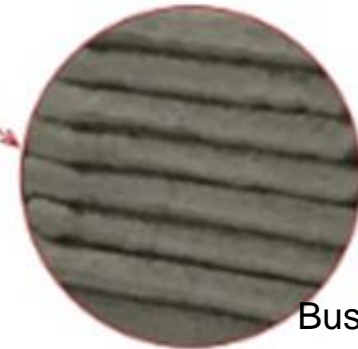
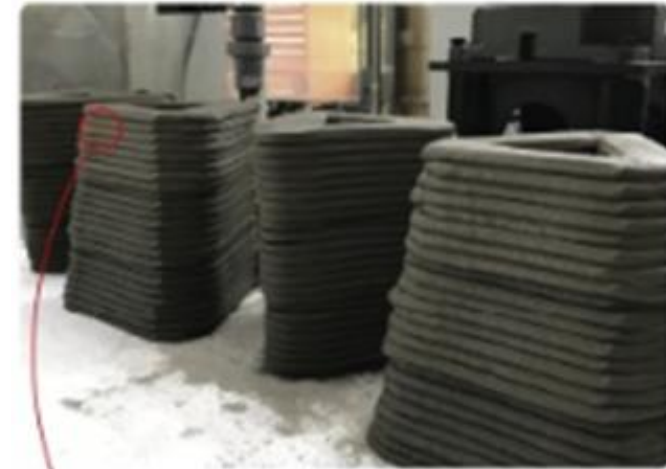
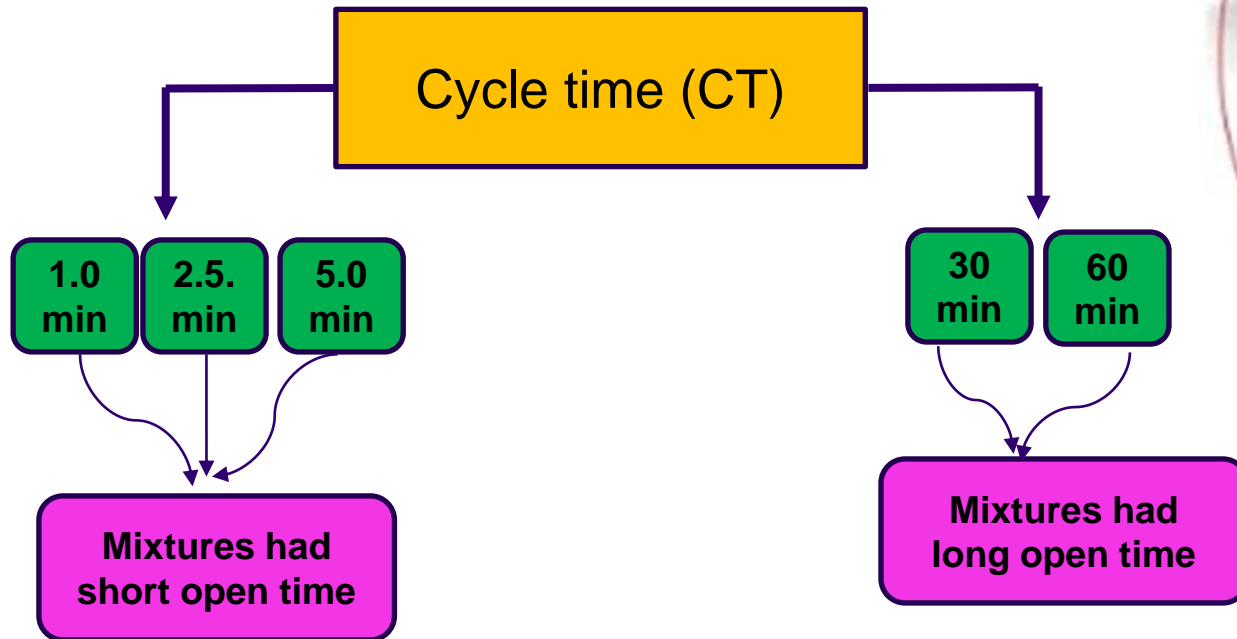


Effect of W/FA, Alk/FA, and SS/SH on the OT and IST

# Investigating the fresh properties

## ❖ Axial deformation test

➤ Axial deformation test → Buildability

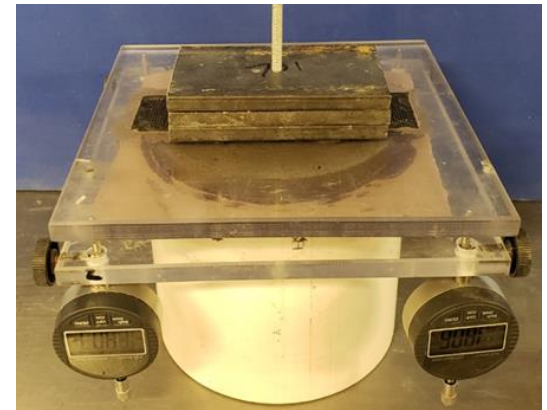
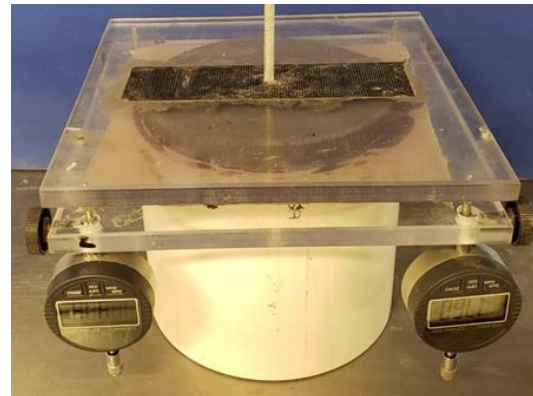
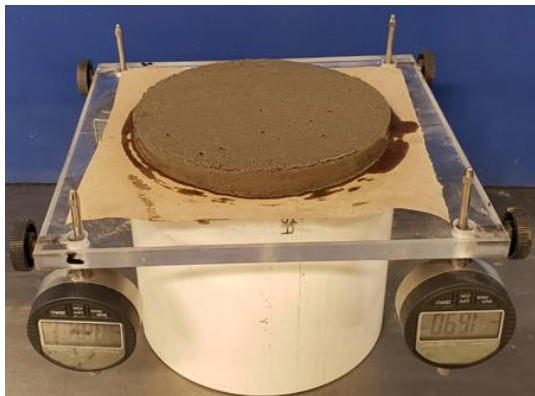
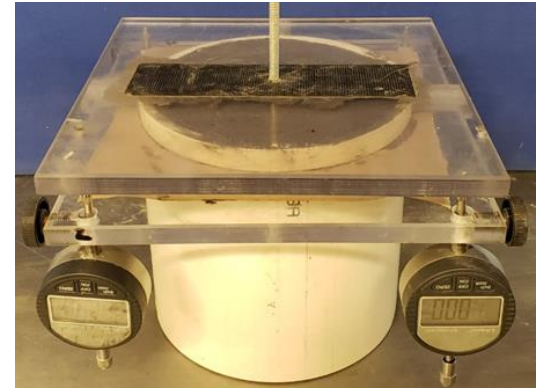
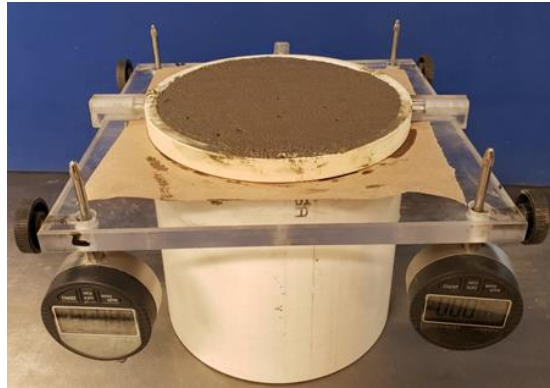
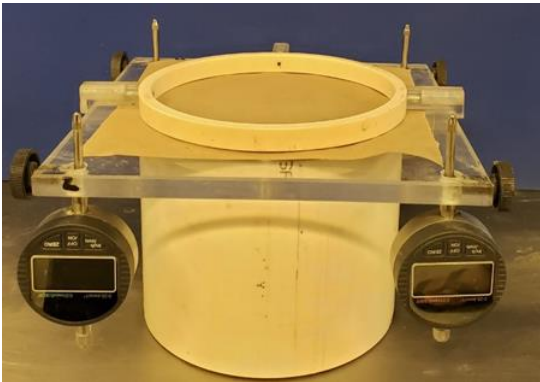


Buswell, 2018

# Investigating the fresh properties

## ❖ Axial deformation test

- New test setup was proposed



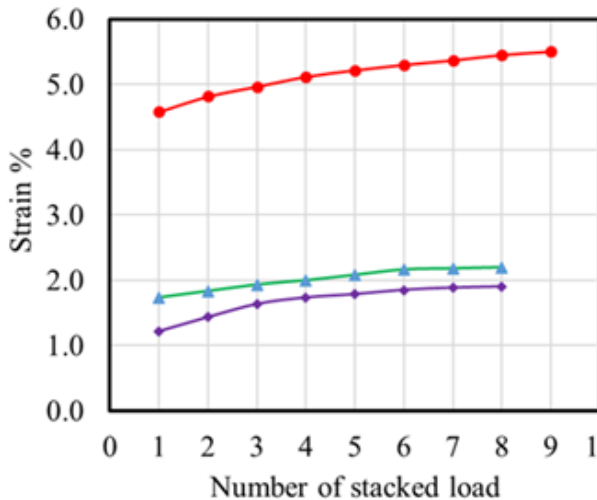
# Investigating the fresh properties

## ❖ Results

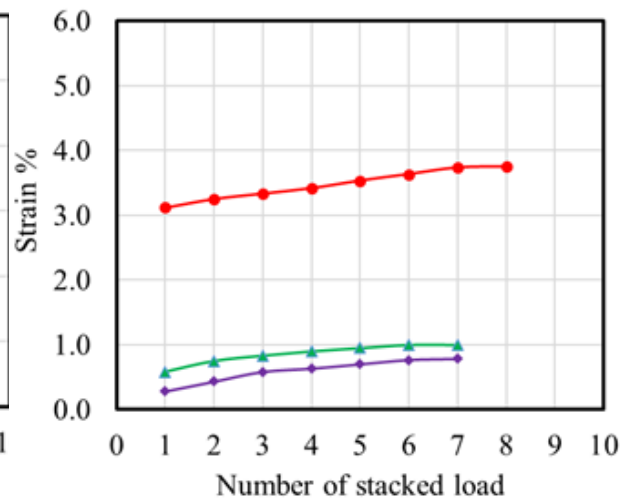
### ➤ Effect of W/FA

—●— W/FA=0.36    —▲— W/FA=0.38    —●— W/FA=0.40

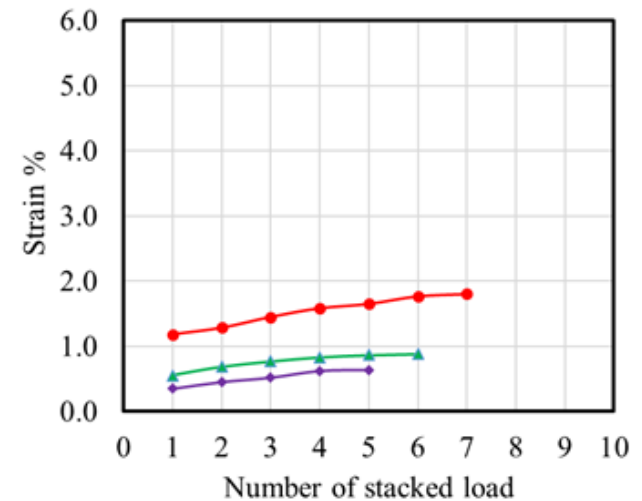
1.0 min



2.5 min



5.0 min

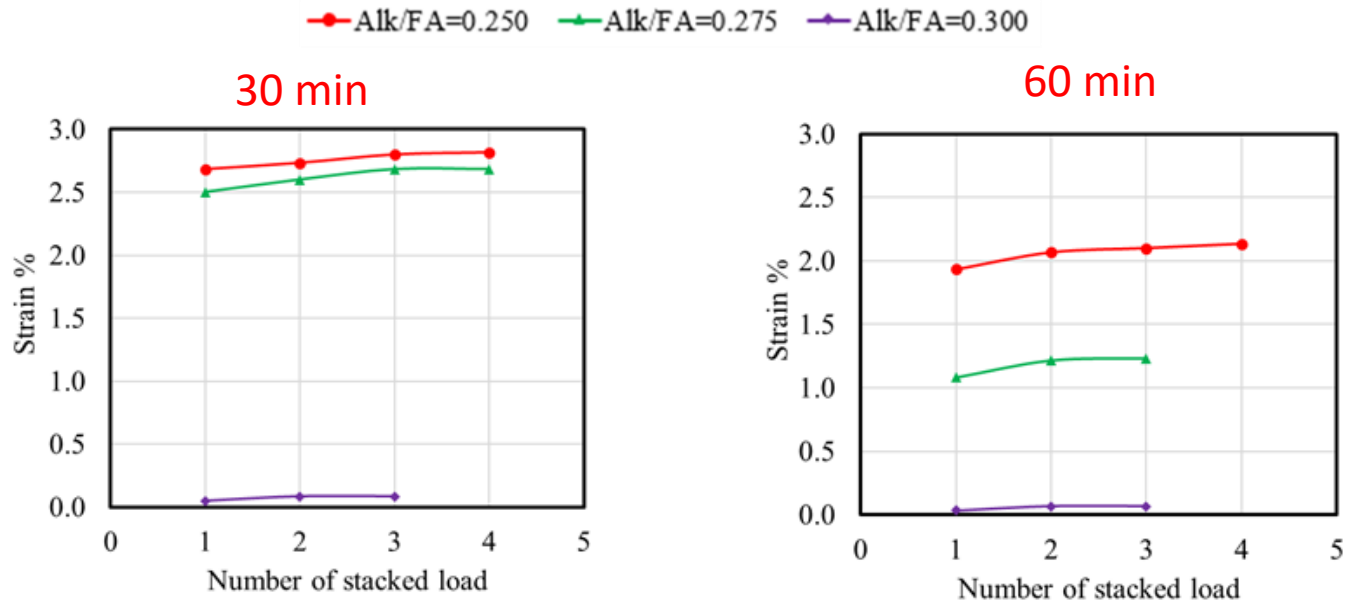


Effect of W/FA on the buildability in terms of **STRAIN**, Alk/FA=0.30, SS/SH= 1.0

# Investigating the fresh properties

## ❖ Results

### ➤ Effect of Alk/FA



Effect of Alk/FA on the buildability in terms of STRAIN, W/FA=0.36, SS/SH= 1.0



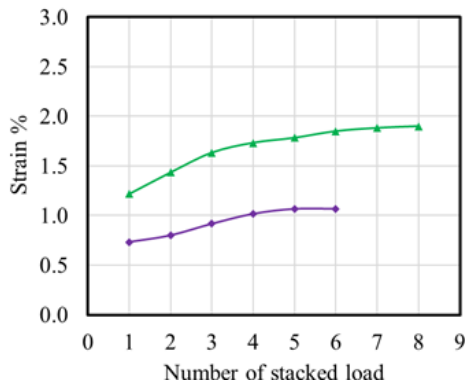
# Investigating the fresh properties

## ❖ Results

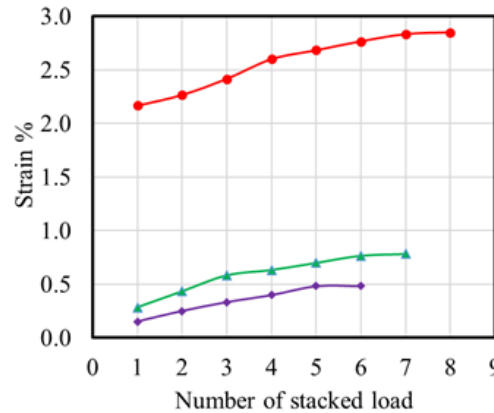
### ➤ Effect of SS/SH

● SS/SH=0.5    ● SS/SH=1.0    ● SS/SH=2.0

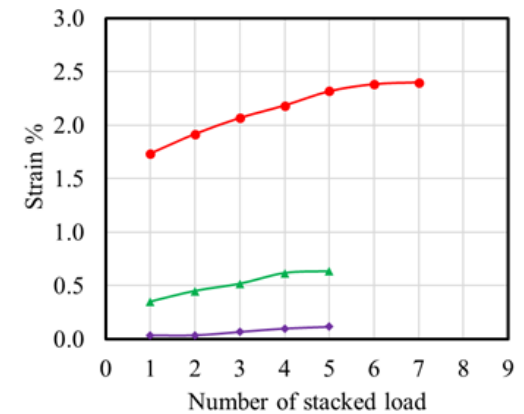
1.0 min



2.5 min



5.0 min



Effect of SS/SH on the buildability in terms of STRAIN, W/FA=0.36, Alk/FA= 0.30

# Investigating the fresh properties

## ❖ Yield stress properties

➤ Penetration test (PT) → Static yield stress ( $\tau_o$ ) → Buildability

- ✓ Penetration depth (h) of the cone plunger in the fresh AAM was measured.
- ✓ The  $\tau_o$  evolution was measured at rest time of 5, 10, 15, and 20 minutes.

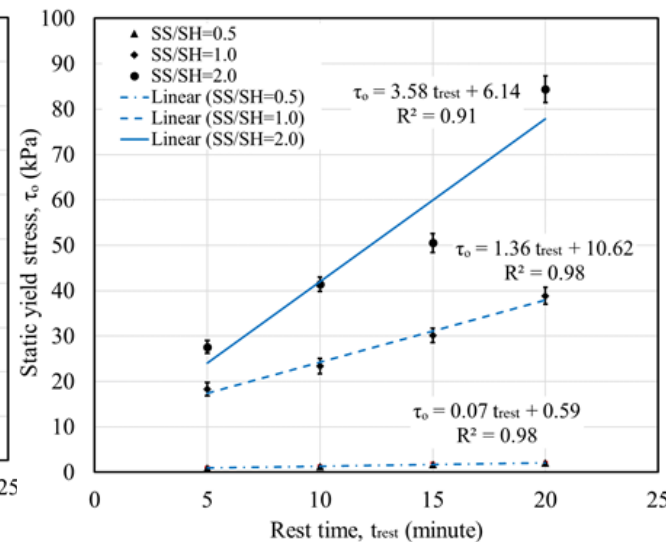
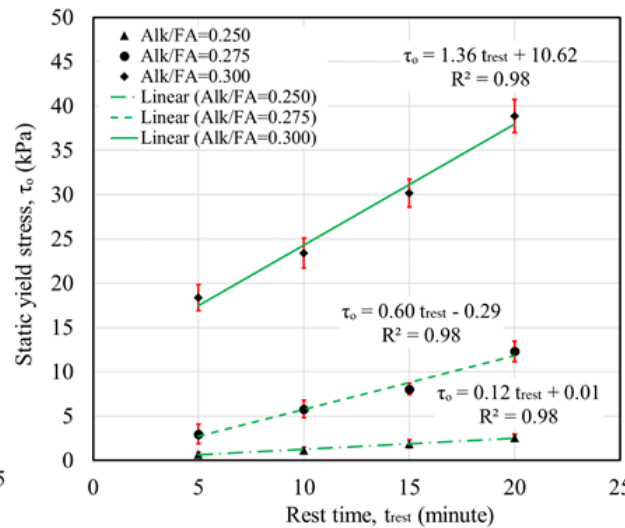
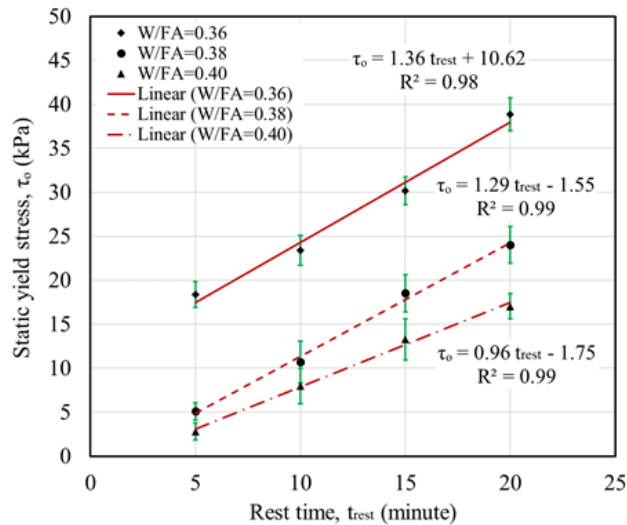


Penetrometer

# Investigating the fresh properties

## ❖ Results

➤ Static yield stress evolution rate ( $A_{thix}$ ) ranged from 0.07 to 3.58 kPa/min



Effect of W/FA, Alk/FA, and SS/SH on the static yield stress

# Investigating the fresh properties

## ❖ Yield stress properties

➤ Viscometer 6 → Static yield stress → Buildability

- ✓ A bucket serving as the outer cylinder, rotating on a base plate, and a stationary inner cylinder.
- ✓ Inner cylinder is 50 mm, while the outer cylinder is 61.9 mm.
- ✓ The AAM is housed in the outer cylinder and the inner cylinder is lowered into the material.
- ✓ The outer cylinder rotates, and measures the torque needed to maintain the set velocity.

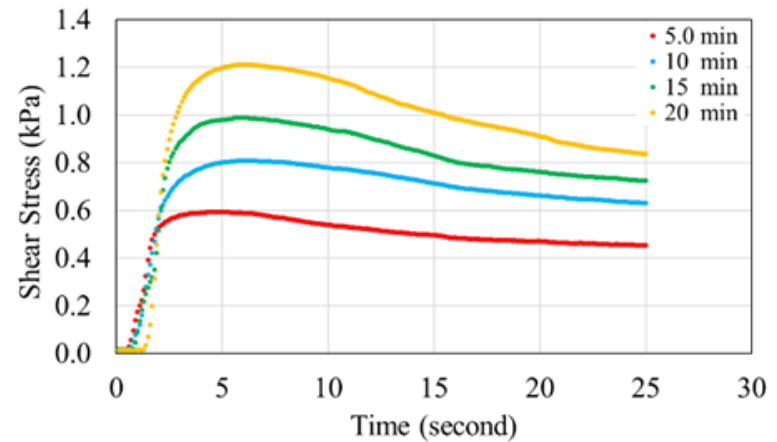
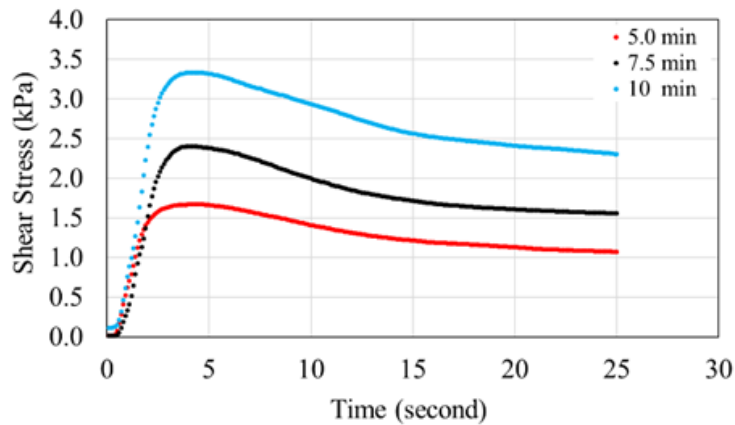


Viscometer 6

# Investigating the fresh properties

## ❖ Results

### ➤ Static yield stress

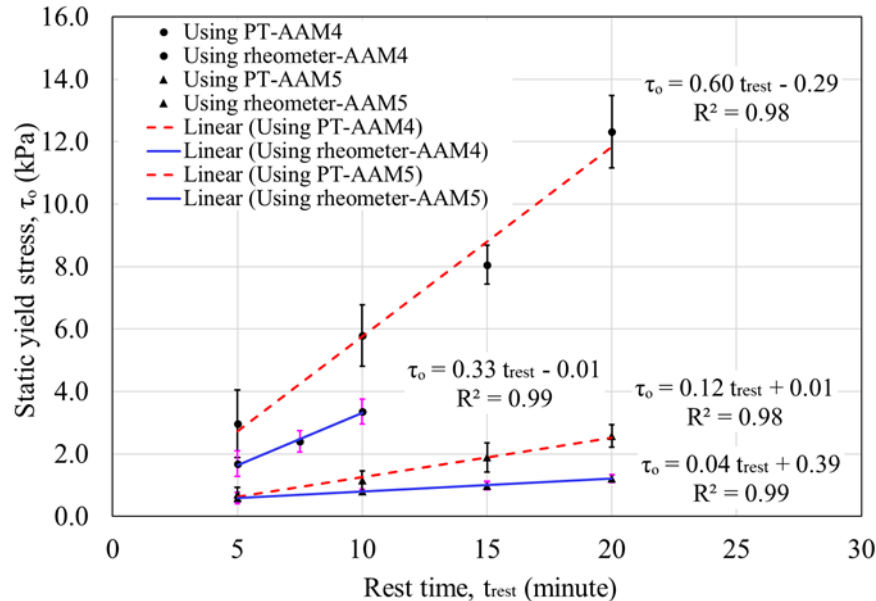


Shear stress curves at different test of: (a) AAM4, and (b) AAM5

# Investigating the fresh properties

## ❖ Results

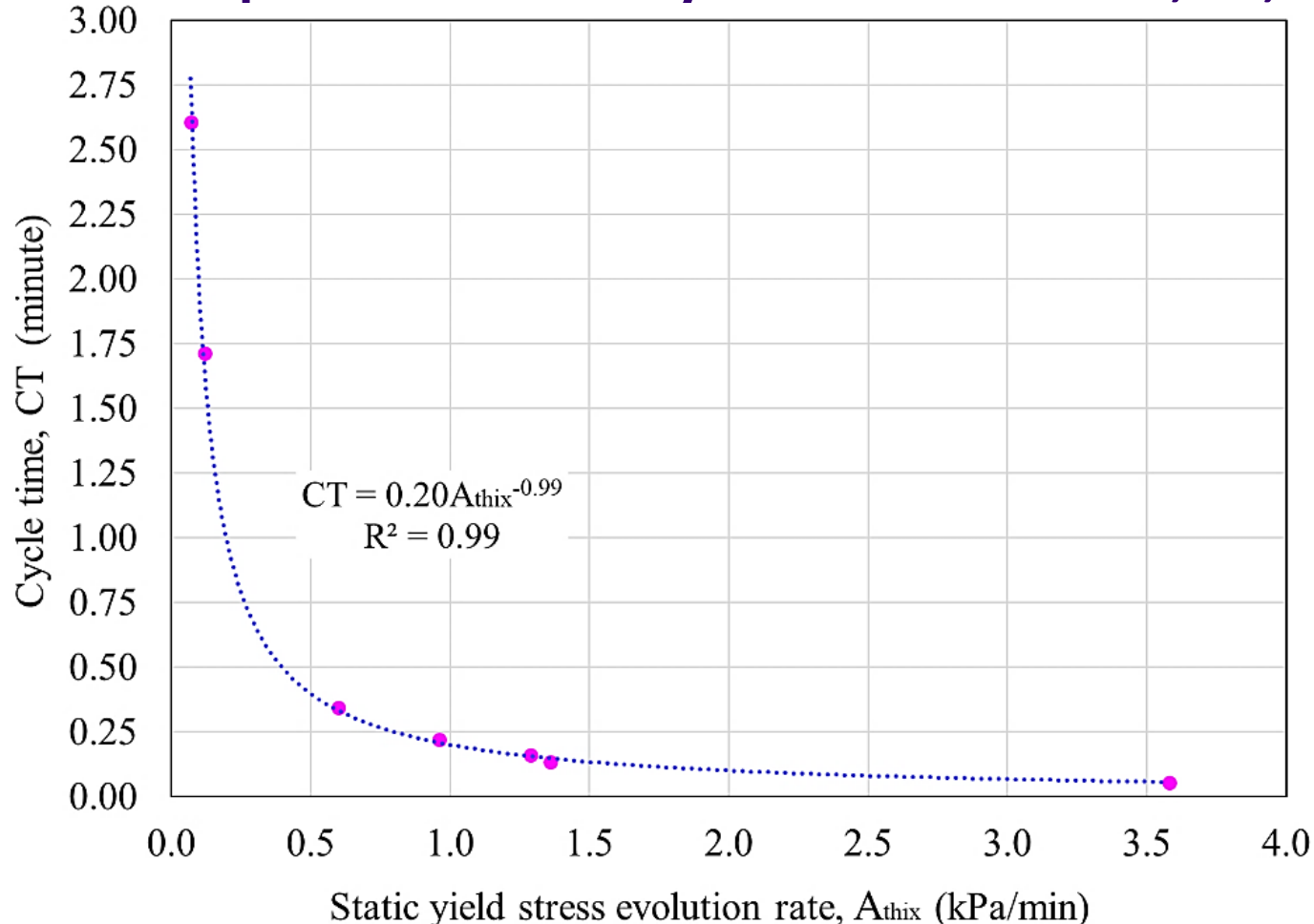
- The static yield stress values obtained from the rheometer were about 17-53% lower than the  $\tau_0$  values obtained from the PT.



Correlation between  $\tau_0$  determined using PT and ConTec6 rheometer

# Investigating the fresh properties

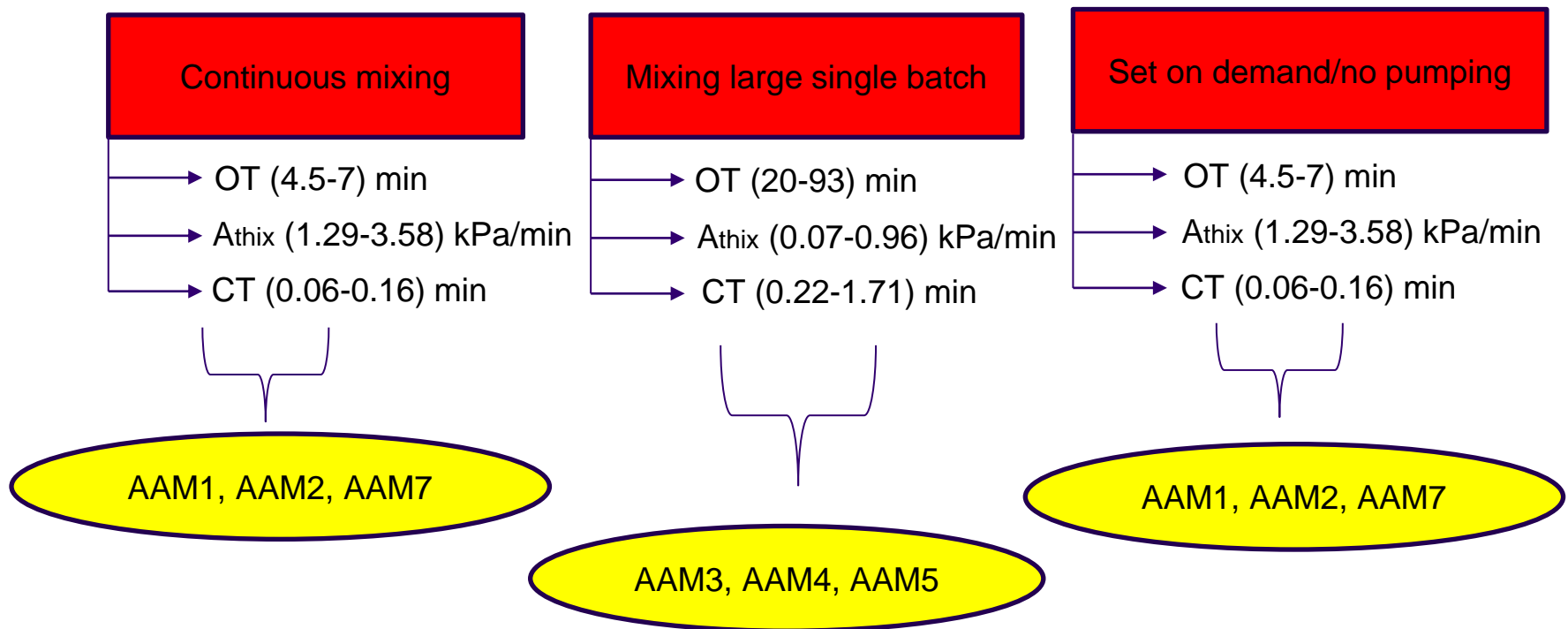
## ❖ Relationship between static yield evolution rate, CT, and OT





# Discussion

## ❖ 3DPC approach recommended for each AAM



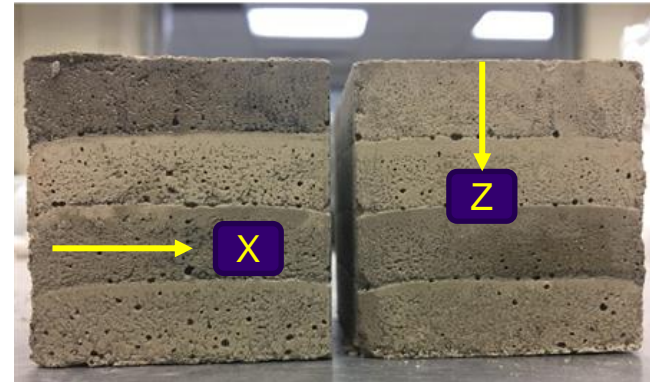
# Investigating the mechanical properties

## ❖ Compressive strength

- Two types of specimens were cast



Full cube casting on one pouring



3DPC specimens with different CTs

- Specimens cured at ambient temperature for 24 hrs and then a moisture room for 28 days

# Investigating the mechanical properties

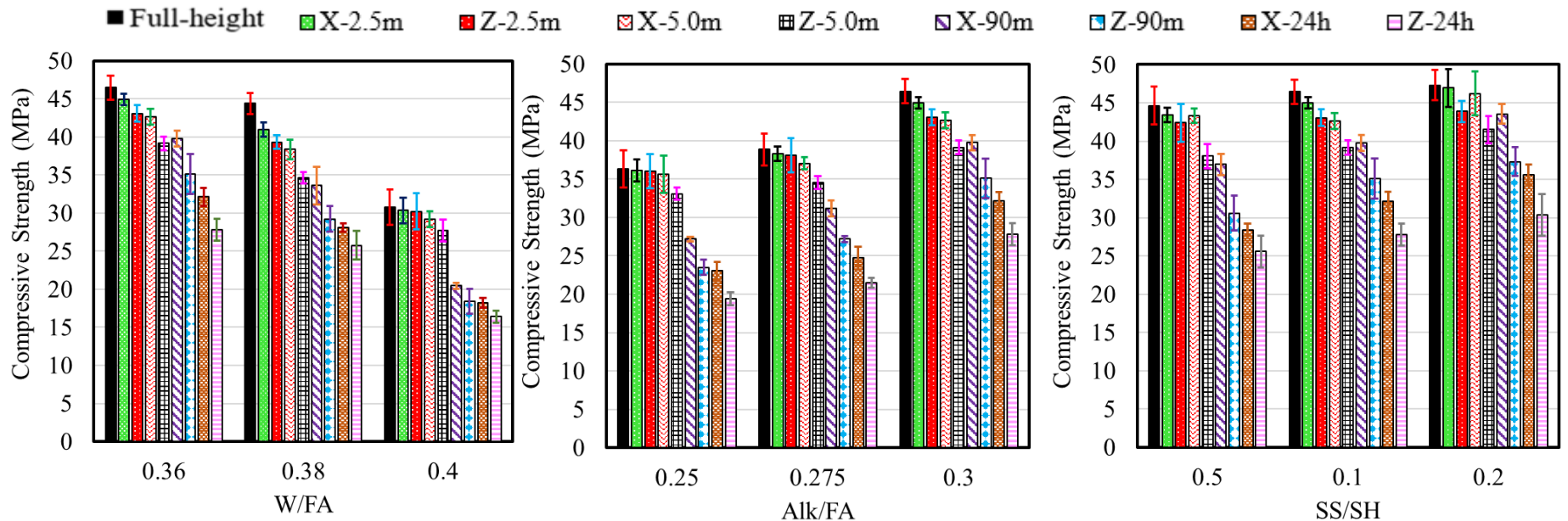
## ❖ Compressive strength

➤ Effect of 3DPC (0.3-50%)

➤ Effect of loading direction (0.3-21%)

➤ Effect of the CT (0.6 to 47%)

➤ Effect of W/FA, Alk/FA, and SS/SH



Compressive strength of full-height and 3DPC specimens

# Investigating the mechanical properties

## ❖ Pull-off test (direct tension)

- Two types of specimens were cast for all mixtures

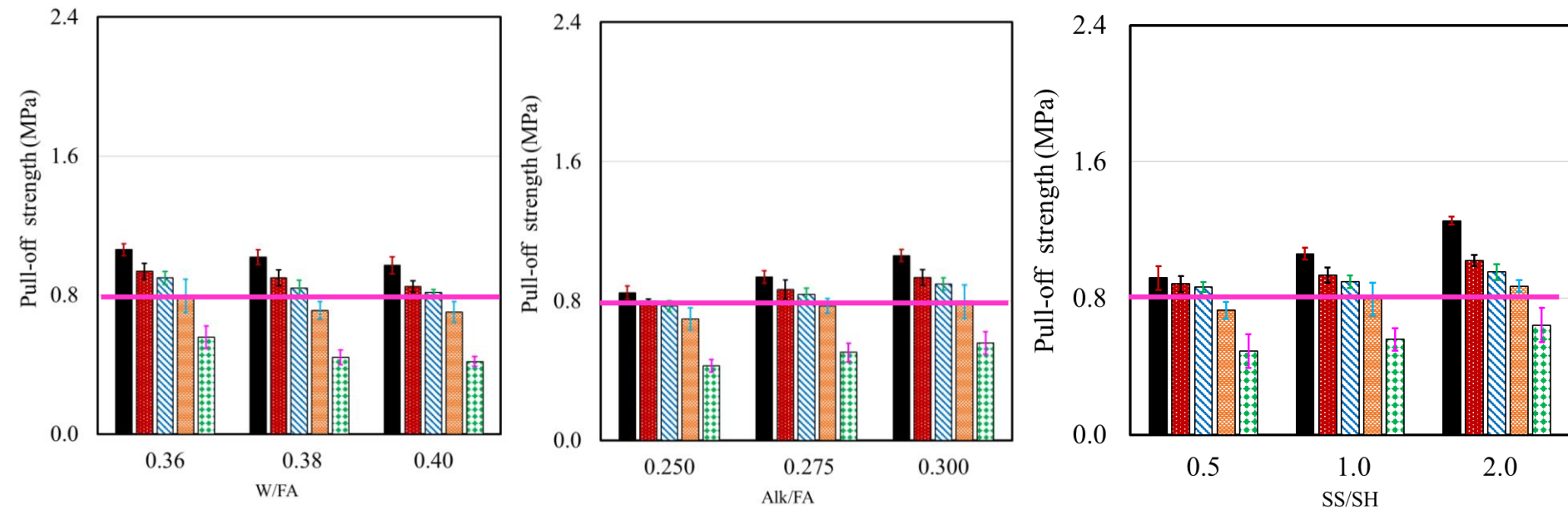


# Investigating the mechanical properties

## ❖ Pull-off results

- Effect of 3DPC
- Effect of the CT
- Effect of W/FA, Alk/FA, and SS/SH

■ Full-height ■ 2.5m ■ 5.0m ■ 90m ■ 24h — EN-1504



Pull-off strength of full-height and 3DPC specimens

# Investigating the mechanical properties

## ❖ Pull-off results

### ➤ Failure mode



Full-height



2.5 min



90 min



24 hr



# Investigating the mechanical properties

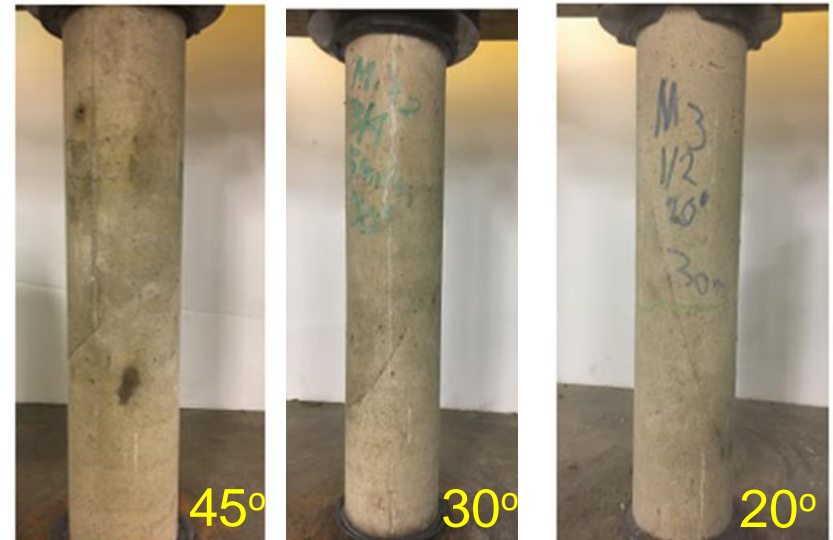
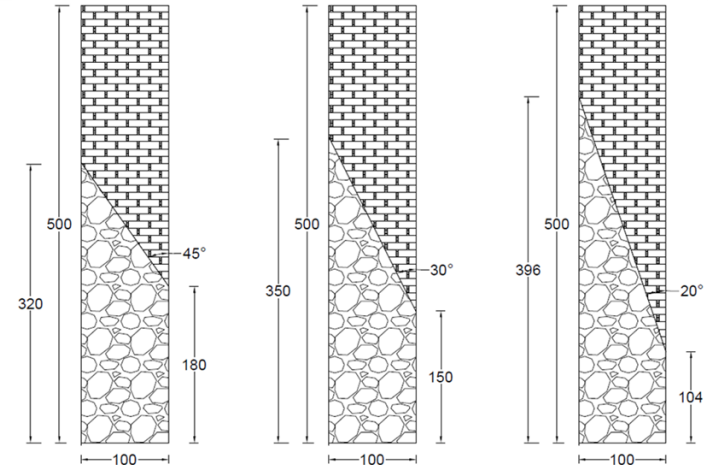
## ❖ Slant shear test

### ➤ Test setup

$$\tau_n = \mu \sigma_n + c$$

AAM mixtures of slant shear test

AAM no.	W/FA	Alk/FA	SS/SH	C.T (minute)
AAM1	0.36	0.300	1.0	5.0
AAM2	0.38	0.300	1.0	5.0
AAM3	0.40	0.300	1.0	30.0
AAM4	0.36	0.275	1.0	30.0
AAM5	0.36	0.250	1.0	30.0
AAM6	0.36	0.300	0.5	30.0
AAM7	0.36	0.300	2.0	5.0

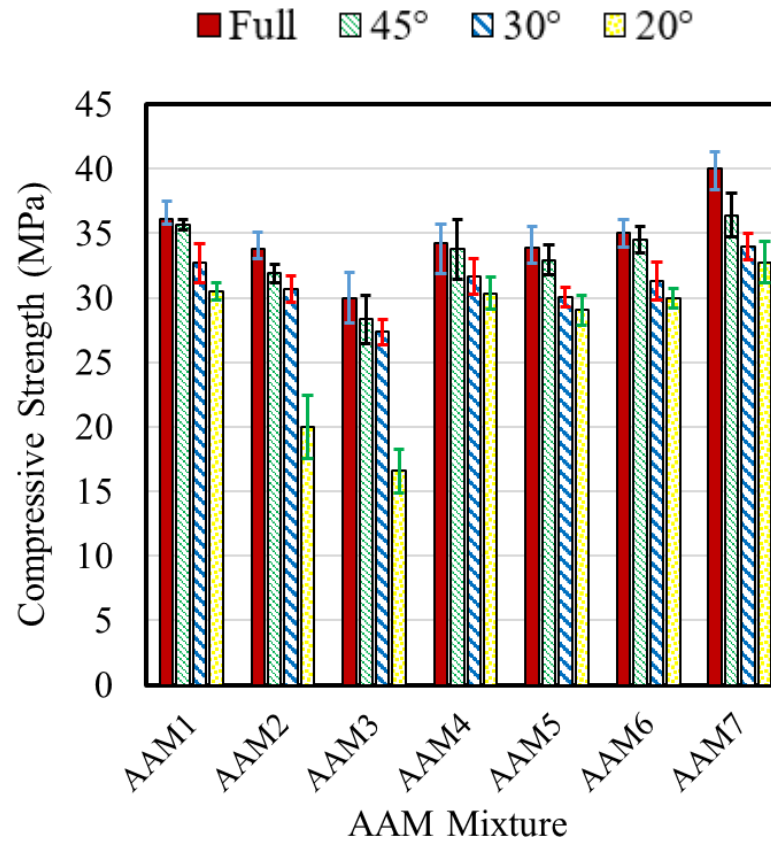


Slant shear specimens having interface angles of 45°, 30°, and 20°



# Task 2: Investigating the mechanical properties

## ❖ Slant shear strength results

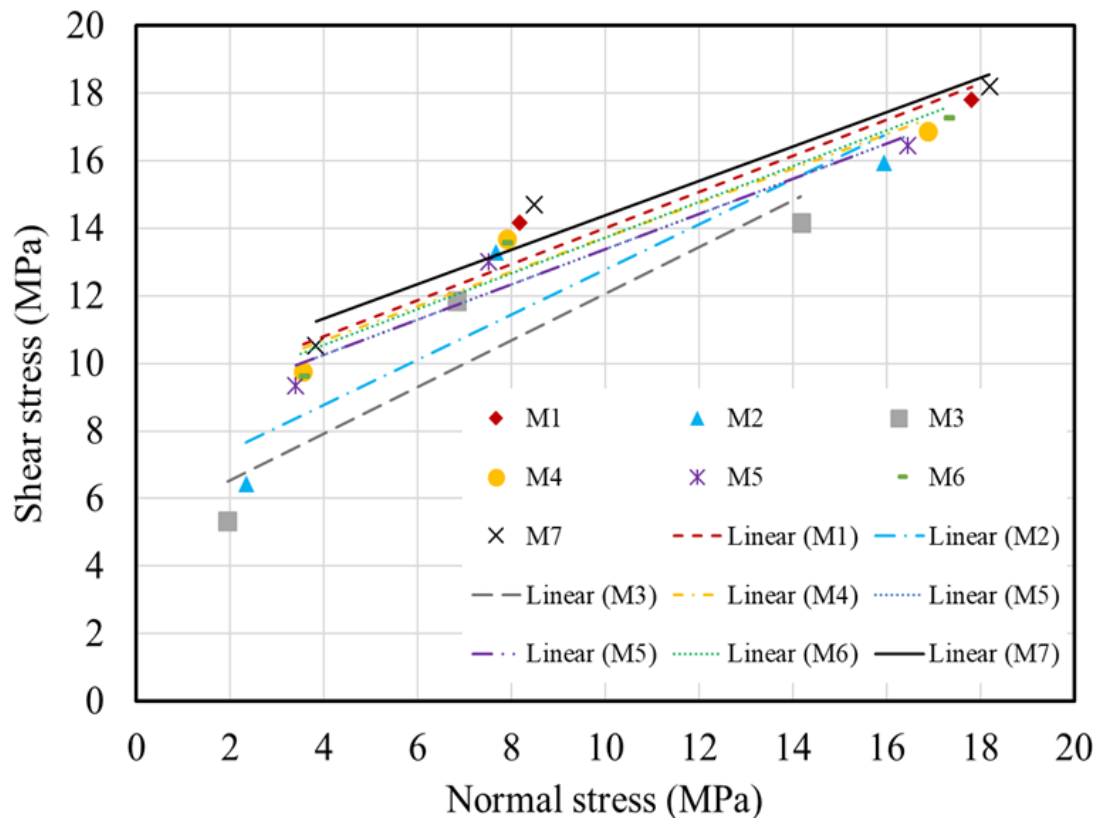


Compressive strength of the full-height and two layered specimens

# Investigating the mechanical properties

## ❖ Slant shear strength results

➤ Cohesion ranged from 5.3 to 9.3 MPa



$$\tau_1 = 0.5345\mu + 8.6598$$
$$R^2 = 0.9377$$

$$\tau_2 = 0.6667\mu + 6.1169$$
$$R^2 = 0.8665$$

$$\tau_3 = 0.6914\mu + 5.158$$
$$R^2 = 0.8616$$

$$\tau_4 = 0.5077\mu + 8.6468$$
$$R^2 = 0.9348$$

$$\tau_5 = 0.5198\mu + 8.1933$$
$$R^2 = 0.9481$$

$$\tau_6 = 0.5286\mu + 8.4413$$
$$R^2 = 0.9484$$

$$\tau_7 = 0.5074\mu + 9.3149$$
$$R^2 = 0.9377$$

# Using 3D-printer machine



# Using 3D-printer machine

## ❖ Adjusting the extrusion rate (ER)

- Nozzle diameter is 30 mm
- Layer thickness is 15 mm

**ER=1.5**

**Layer width=65.9**



**ER=1.0**

**Layer width= 64.1**



**ER=0.7**

**Layer width=32.5**





# Using 3D-printer machine

## ❖ Example of 3D-printed AAMs using single batch

### Poor extrudability



### Failure



### High quality 3D structure



# Conclusions

- OT, ranged from 3 to 93 minutes, was obtained by optimizing the W/FA, Alk/FA, and SS/SH.
- The strain ranged from 0.11 to 8.9% depending on the proportions of AAMs and CTs.
- The static yield stress evolution rate ranged from 0.07 to 3.58 kPa/min.
- The lowest estimated CT possible while avoiding collapse of the 3DPC structure was based on the static yield stress evolution rate and shear stress induced from gravity, and it ranged from 0.06 to 2.61 minutes.
- The reduction in the compressive strength of 3DPC specimens having CT ranging from 2.5 mins to 24 hrs ranged from 0.6 to 47% than full-height specimens.
- The reduction in the bond strength of 3DPC specimens having CT ranging from 2.5 mins to 24 hrs ranged from 3.8 to 50% by pill-off test.

# Conclusions

- The average cohesion between the layers was found to be 7.7 MPa with a slight increase when either increasing SS/SH or decreasing W/FA.
- The bond strength values of 3DPC by pull-off and direct shear strength exceeded the minimum recommended values by standards of 0.8 and 0.9 Mpa, respectively.

# Acknowledgement



**MISSOURI**  
DEPARTMENT OF  
NATURAL RESOURCES



# Thank You