UTA
Center for Advanced Construction Materials

# A convergent approach for monitoring fluid to solid transition in 3D printing using adaptive rheology and electrochemical impedance spectroscopy


**Panagiotis A. Danoglidis[1], Maria S. Konsta-Gdoutos[2]**

*[1]Assistant Professor of Research, Department of Civil Engineering, Center for Advanced Construction Materials, The University of Texas at Arlington*
*panagiotis.danoglidis@uta.edu*

*[2]Professor of Civil Engineering, Director, Tier 1 University Transportation Center for Durable and Resilient Transportation Infrastructure. Associate Director, Center for Advanced Construction Materials, The University of Texas at Arlington*
*maria.konsta@uta.edu*

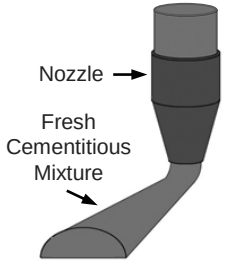



ACI Spring Convention 2024
Nanoparticles for 3D concrete printing
March 24 – 28, 2024, Hyatt Regency New Orleans, New Orleans , LA


UTA

# Characterization of Printability of Cement-Based Mixtures for Additive Manufacturing

## Flowability

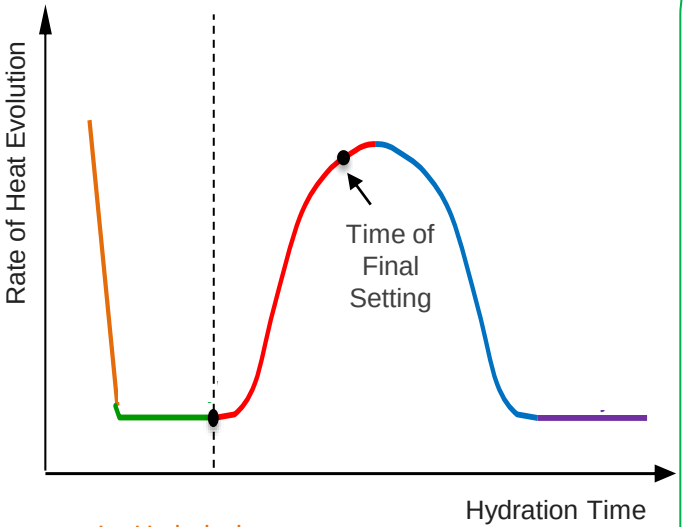



The ability of the material to be pumped and extruded, and retain its deposited shape

## Viscosity

- Material property indicating the resistance of a Newtonian fluid, i.e., fresh cementitious mixture, to flow
- Viscosity is the ratio between shear stress and shear rate

**Time of Initial Setting**




I – Hydrolysis
II – Dormancy
III – Acceleration
IV – Deceleration
V – Steady State

## Buildability

Ability of the deposited layers to gain compressive strength sufficient to withstand the weight of 3D-printed layers

*3D-printed concrete with sufficient buildability*

*3D-printed concrete structure failure due to insufficient buildability*

## Green Strength

- Compressive Strength of concrete in plastic state
- High green strength values indicate improved resistance to deformation of printed layers needed to retain their own shape after casting

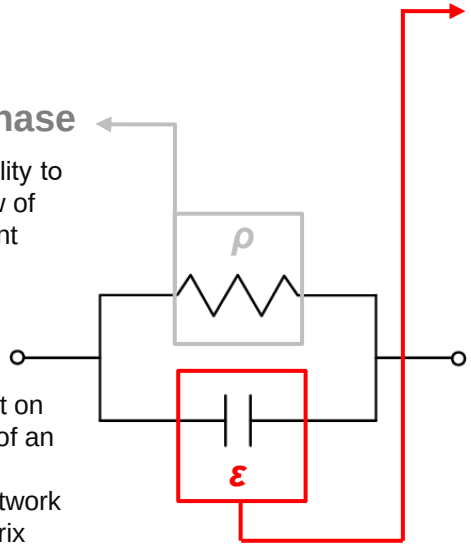
# Monitoring of Printability-Related Properties of 3D-Printed Concrete through Electrochemical Impedance Spectroscopy

## Electrochemical Phases of Concrete

### Resistive Phase

- Material's ability to resist the flow of electric current through it

- It is dependent on the formation of an electrically conducting network within the matrix




### Capacitive Phase

- Material's ability to store electrical energy
- **Independent of the need for the presence of an electrically conducting network within the matrix**

Dielectric Permittivity (***ε***): True material property indicating the electrical energy storage capacity of a material

✓ **Time of Setting** ✓ **Viscosity** ✓ **Green strength**

**Based on Dielectric Permittivity data**

*Danoglidis, P.A. and Konsta-Gdoutos, M.S., 2023. Construction and Building Materials, 390, p.131593*
*Danoglidis, P.A., et al., 2019. Carbon, 145, pp.218-228*

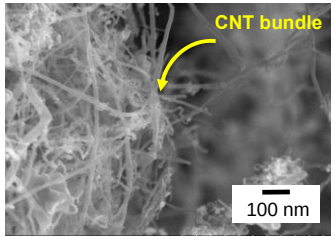
# Materials and Experimental Program

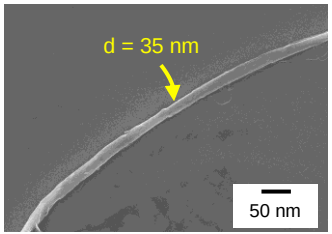
Cement Paste (CP)
w/c = 0.4

Cement Mortar (M)
w/c/s = 0.4/1.0/2.75

**OPC Type I**

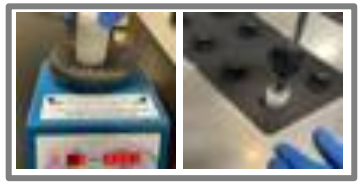
**Sand**
*(ASTM C778)*



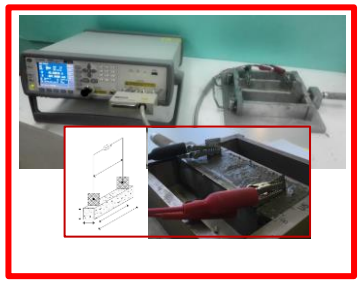

SEM of monodispersed CNT

## Hydration/Setting Time



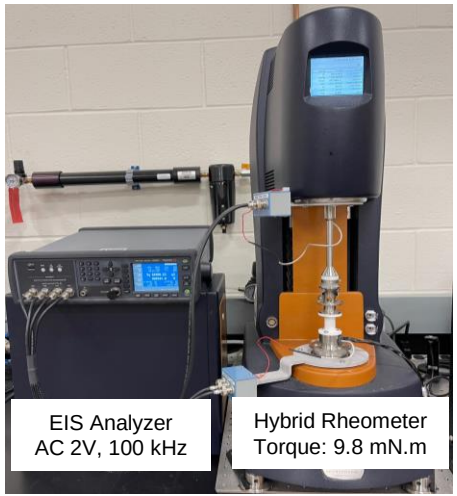
**Heat of hydration**
*ASTM C1679-22*

**Time of Setting**
*ASTM C191-21*



**Electrochemical Impedance Spectroscopy**

## Rheological Test




**Dielectric permittivity, ε**

$$\varepsilon = C\frac{d}{A\varepsilon_o}$$

C= Capacitance
d= Current depth
A= In-plane current
$\varepsilon_0$= Permittivity of vacuum

**Viscosity, η**

$$\eta = \frac{\tau}{\dot{\gamma}}$$

τ= Shear stress
γ= Shear rate

## Green Strength Test


**Green strength, $\sigma_G$**

$$\sigma_G = \frac{F}{A}$$

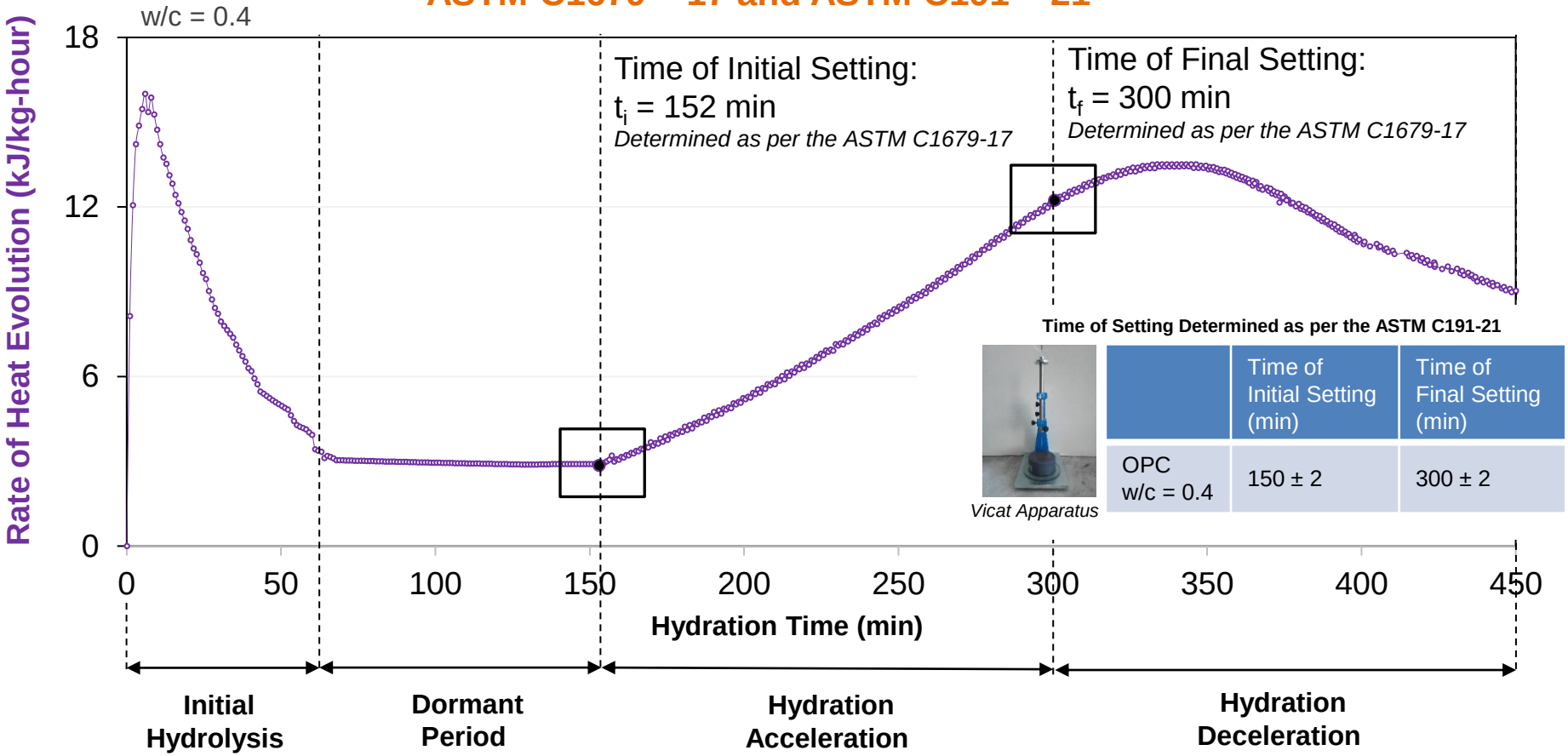
**Electrical Energy Density, *E***

$$E = \int_1^n \left(\frac{d\varepsilon_1}{dt_1} + \cdots + \frac{d\varepsilon_n}{dt_n}\right) dt$$

$d\varepsilon/dt$ = Rate of permittivity Change

n = Time of initial setting

# Time of Initial and Final Setting of OPC Cement Paste
## ASTM C1679 – 17 and ASTM C191 – 21




**Time of Setting Determined as per the ASTM C191-21**

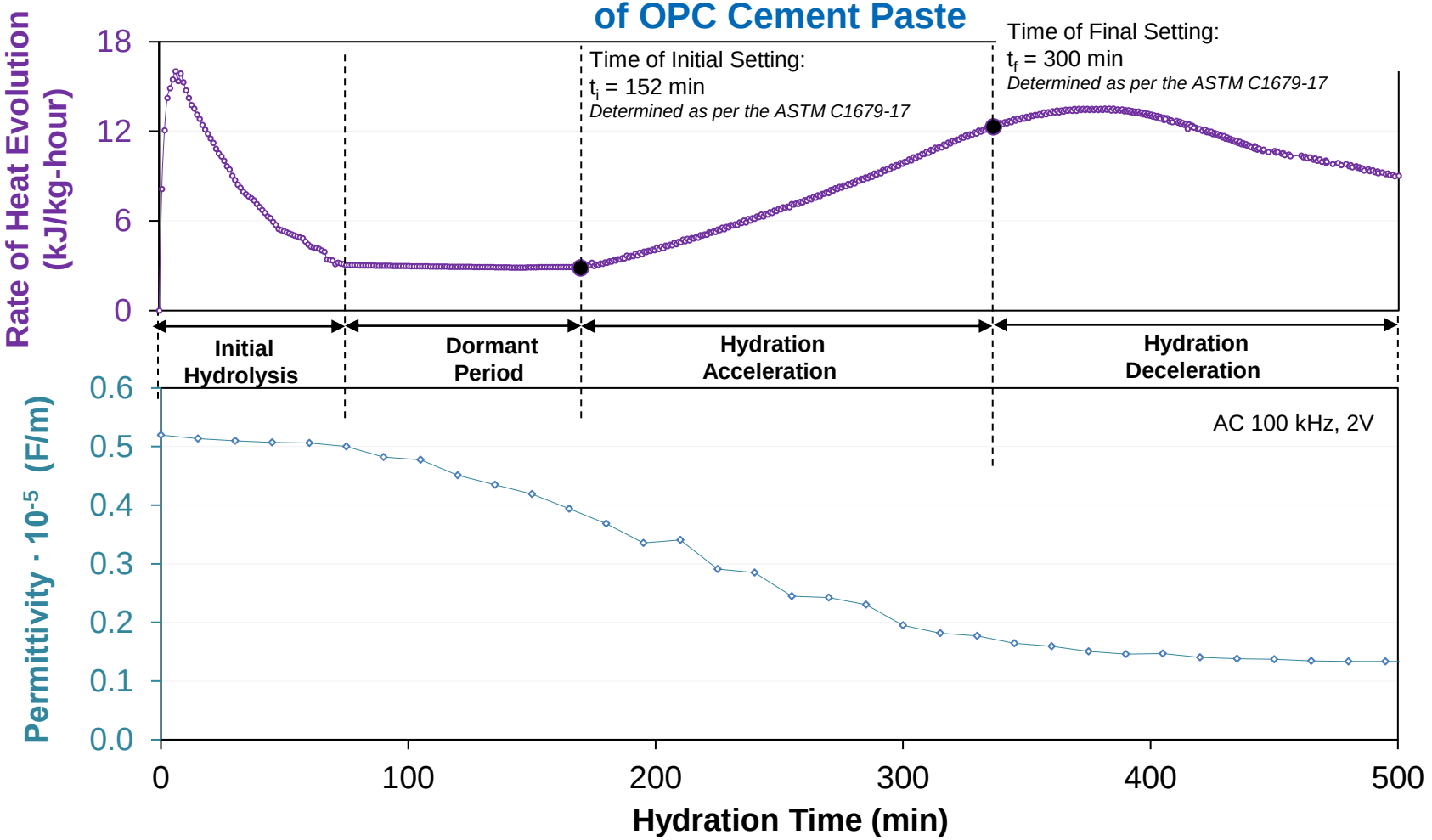


*Vicat Apparatus*

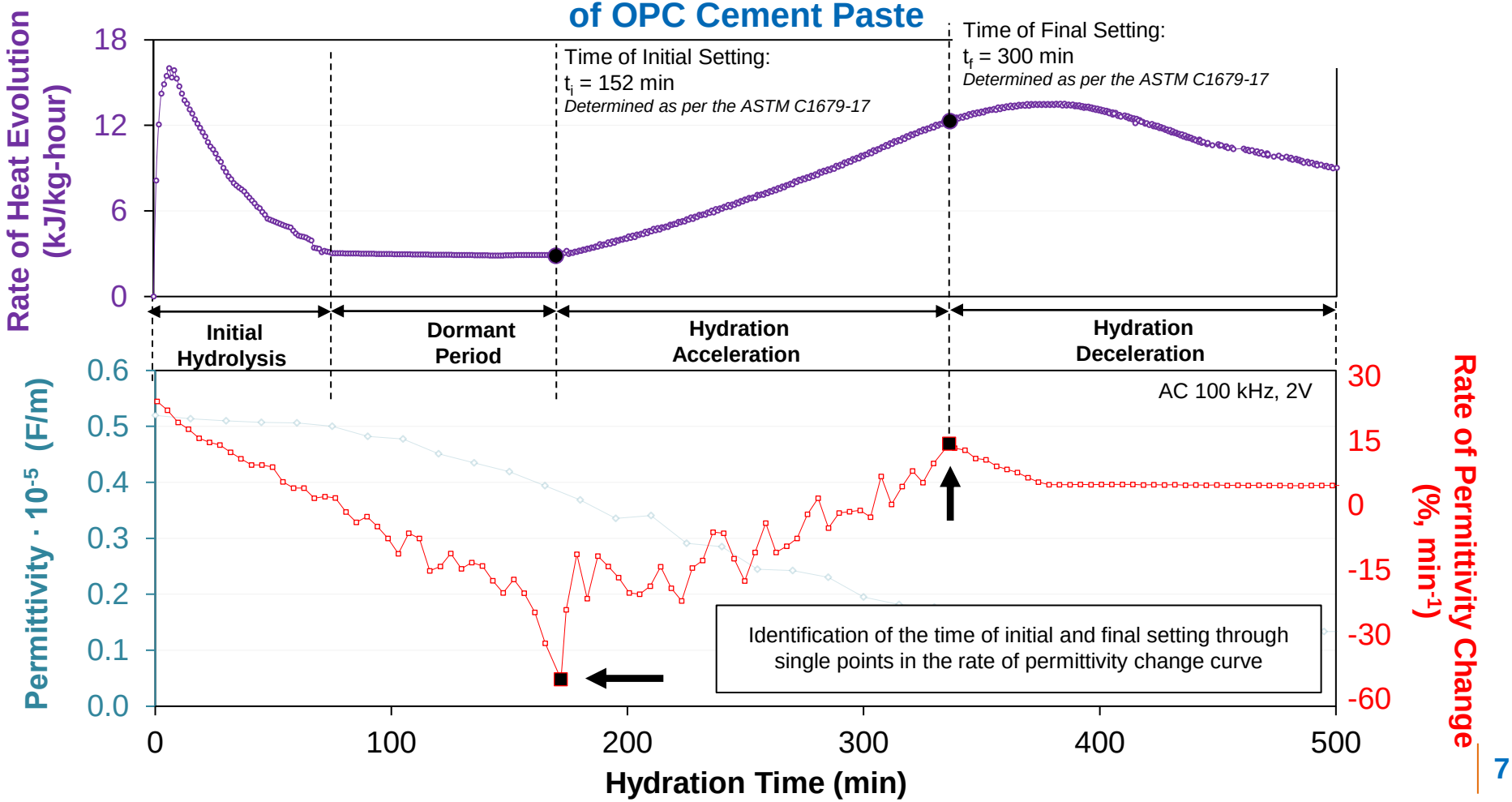
| | Time of Initial Setting (min) | Time of Final Setting (min) |
|---|---|---|
| OPC w/c = 0.4 | 150 ± 2 | 300 ± 2 |


*Konsta-Gdoutos, M.S. and Shah, S.P., 2003. Cement and Concrete Research, 33(8), pp.1269-1276*
*Konsta-Gdoutos, M.S. et al 2002. Materials Journal, 99(2), pp.173-179*

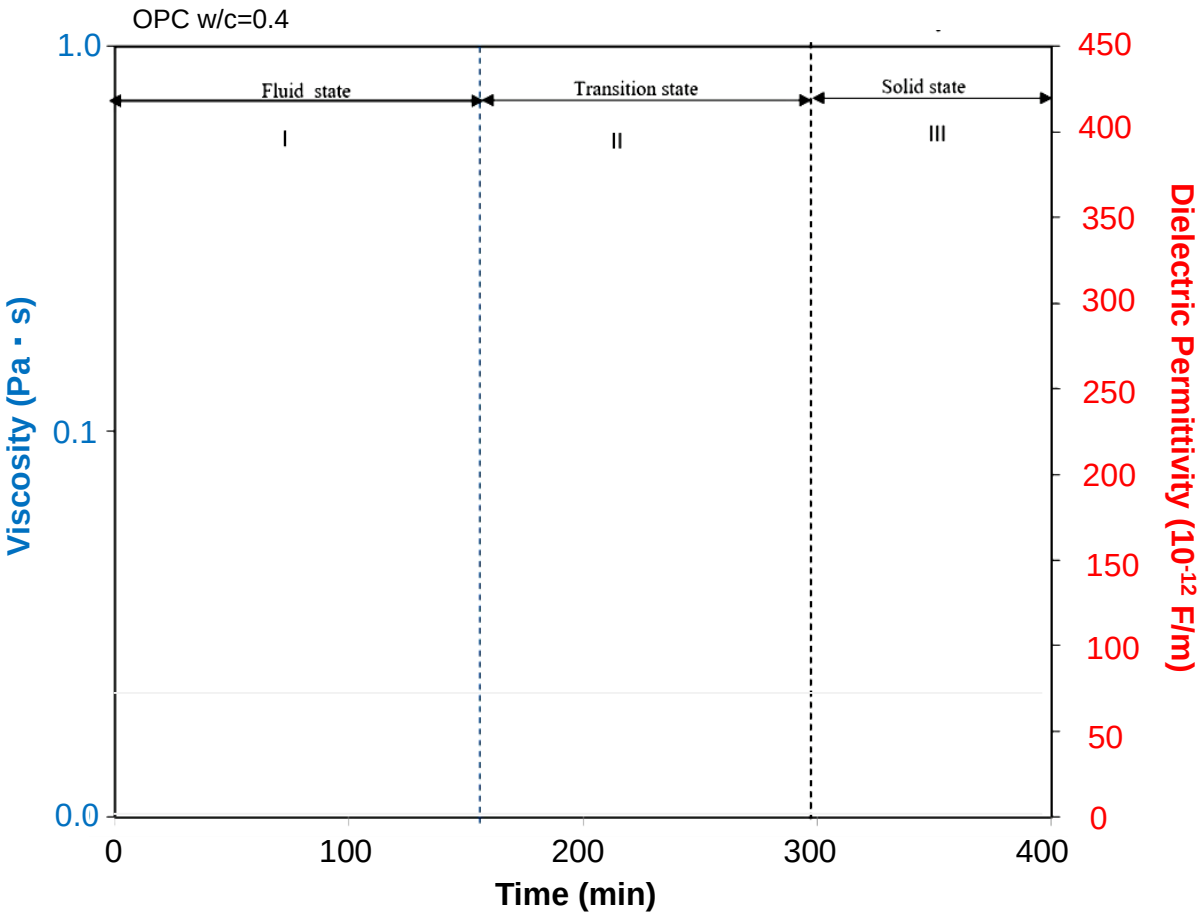
# Relationship between Rate of Heat Evaluation and Permittivity of OPC Cement Paste

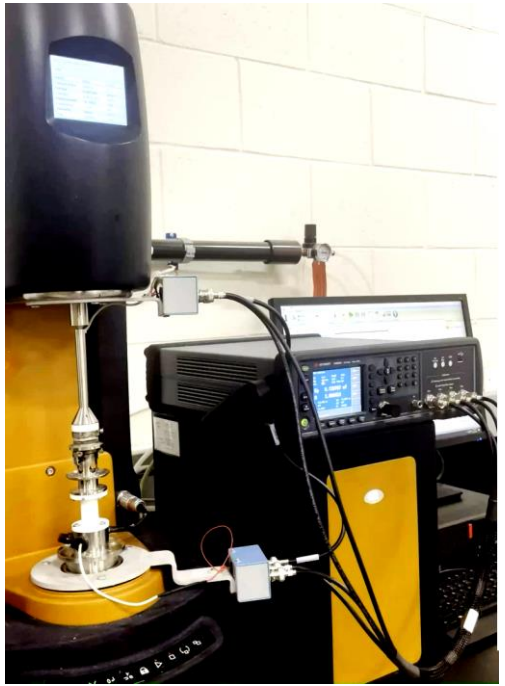


# Relationship between Rate of Heat Evaluation and Rate of Permittivity Change of OPC Cement Paste

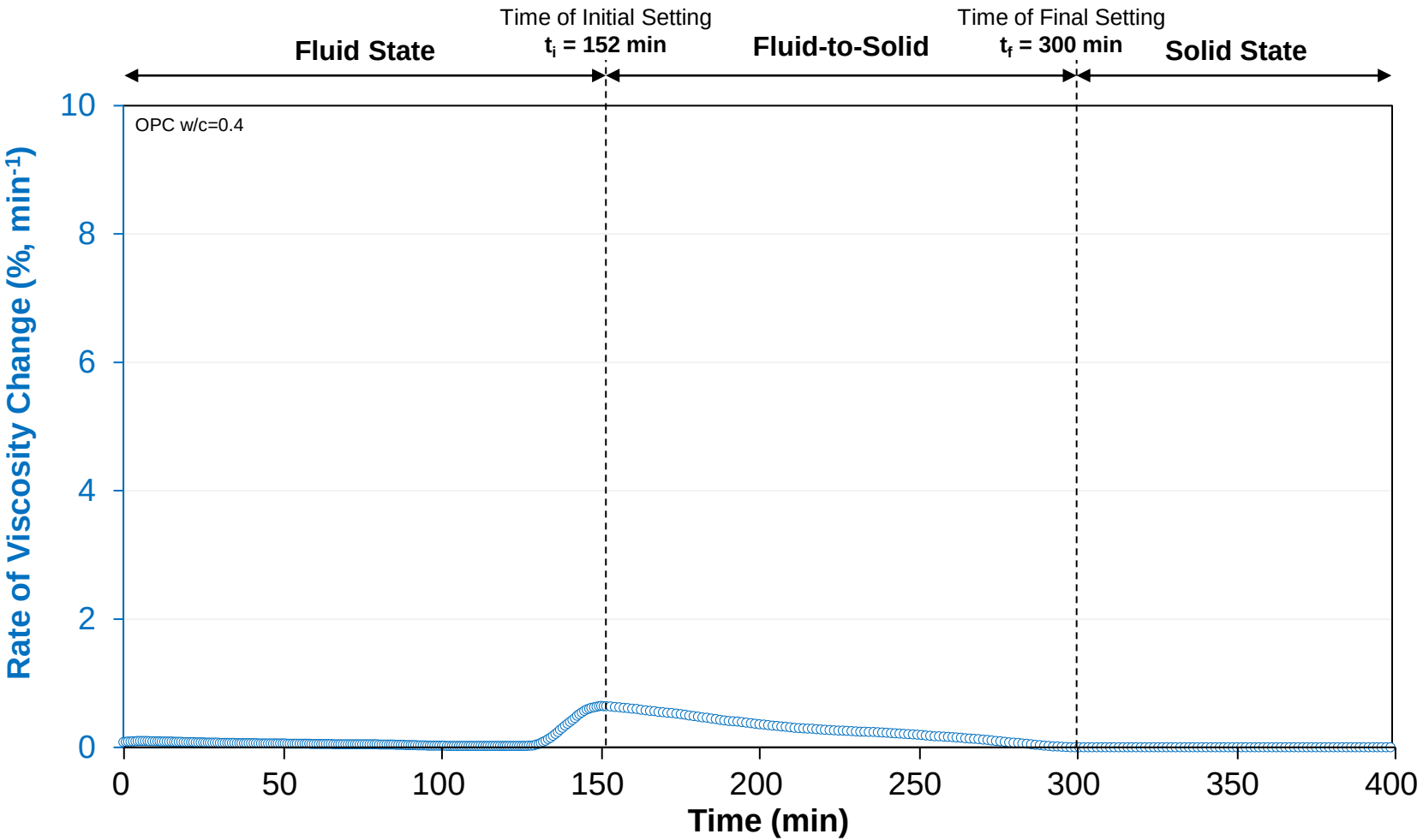


# Rheo-Impedance Spectroscopy Test in OPC Cement Paste
## Viscosity and Dielectric Permittivity

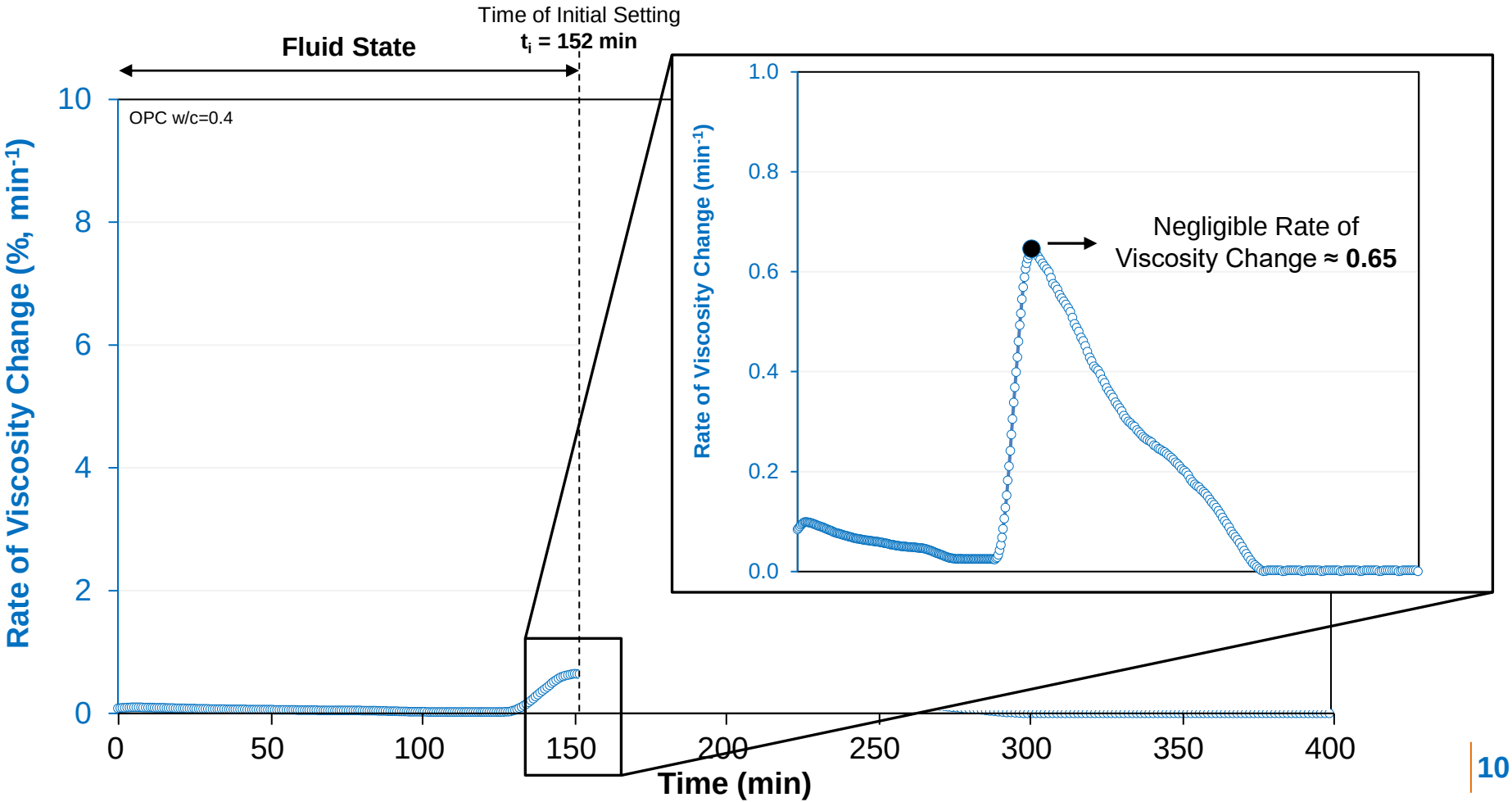


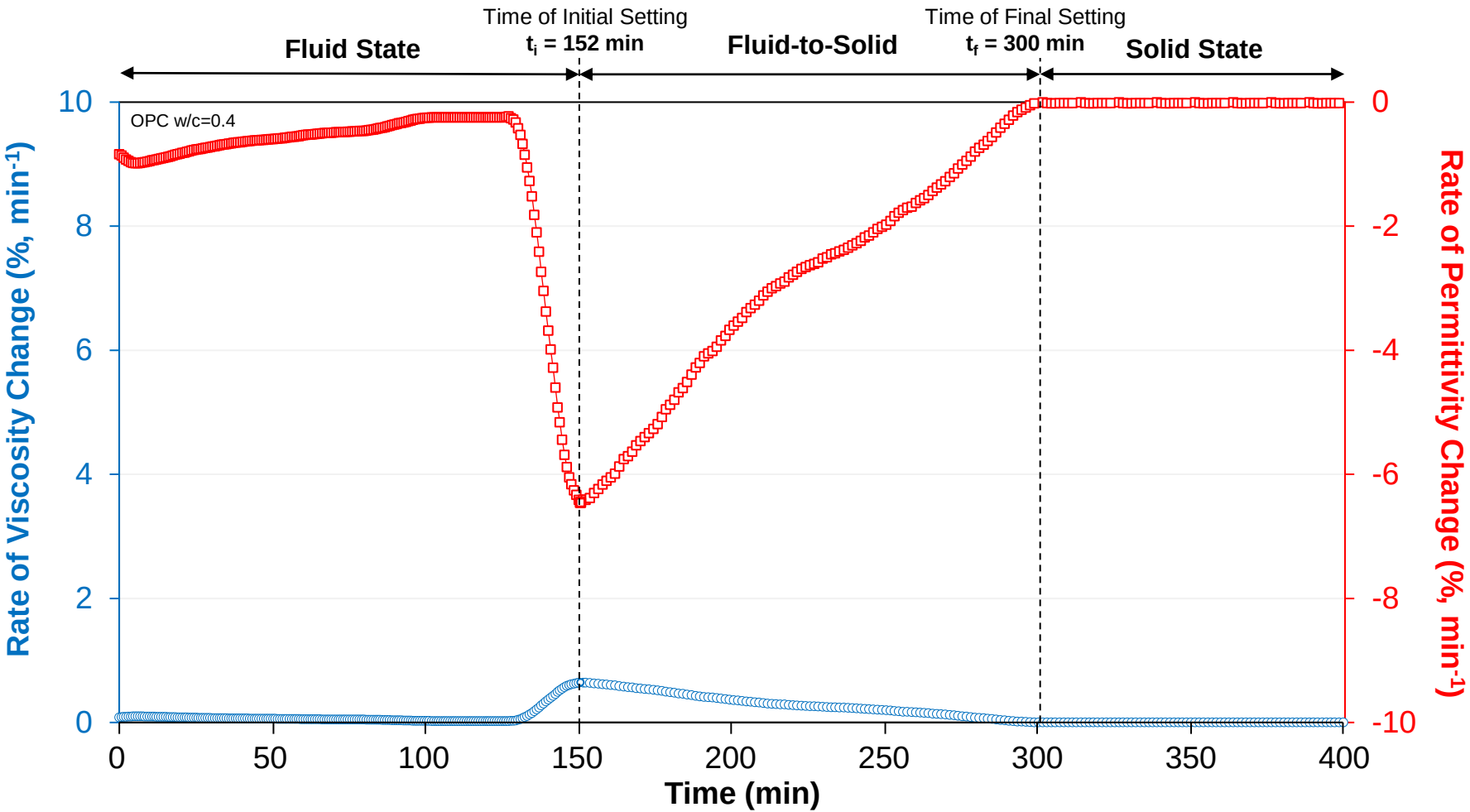
# Rate of Viscosity Change of OPC Cement Paste



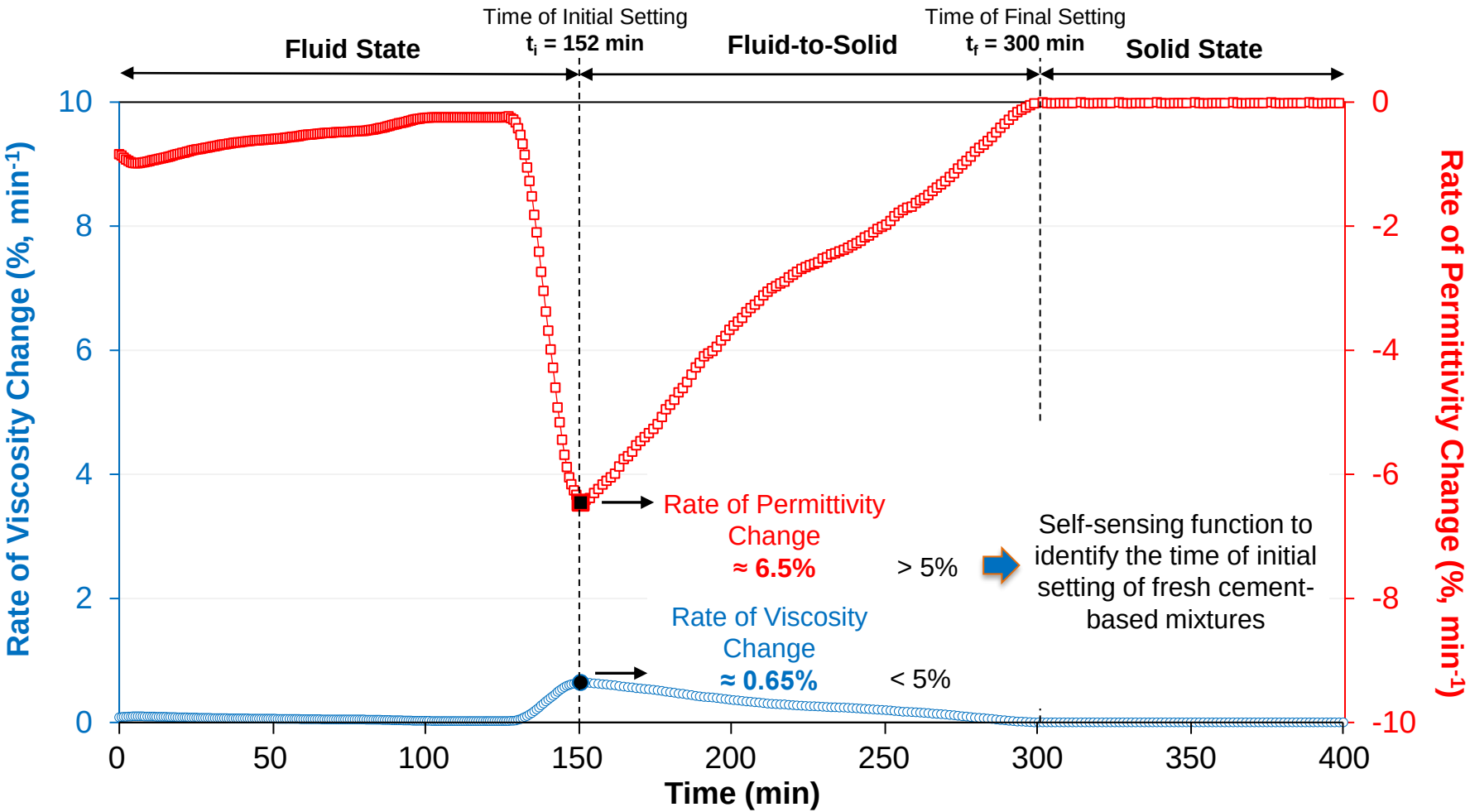
# Rate of Viscosity Change of OPC Cement Paste



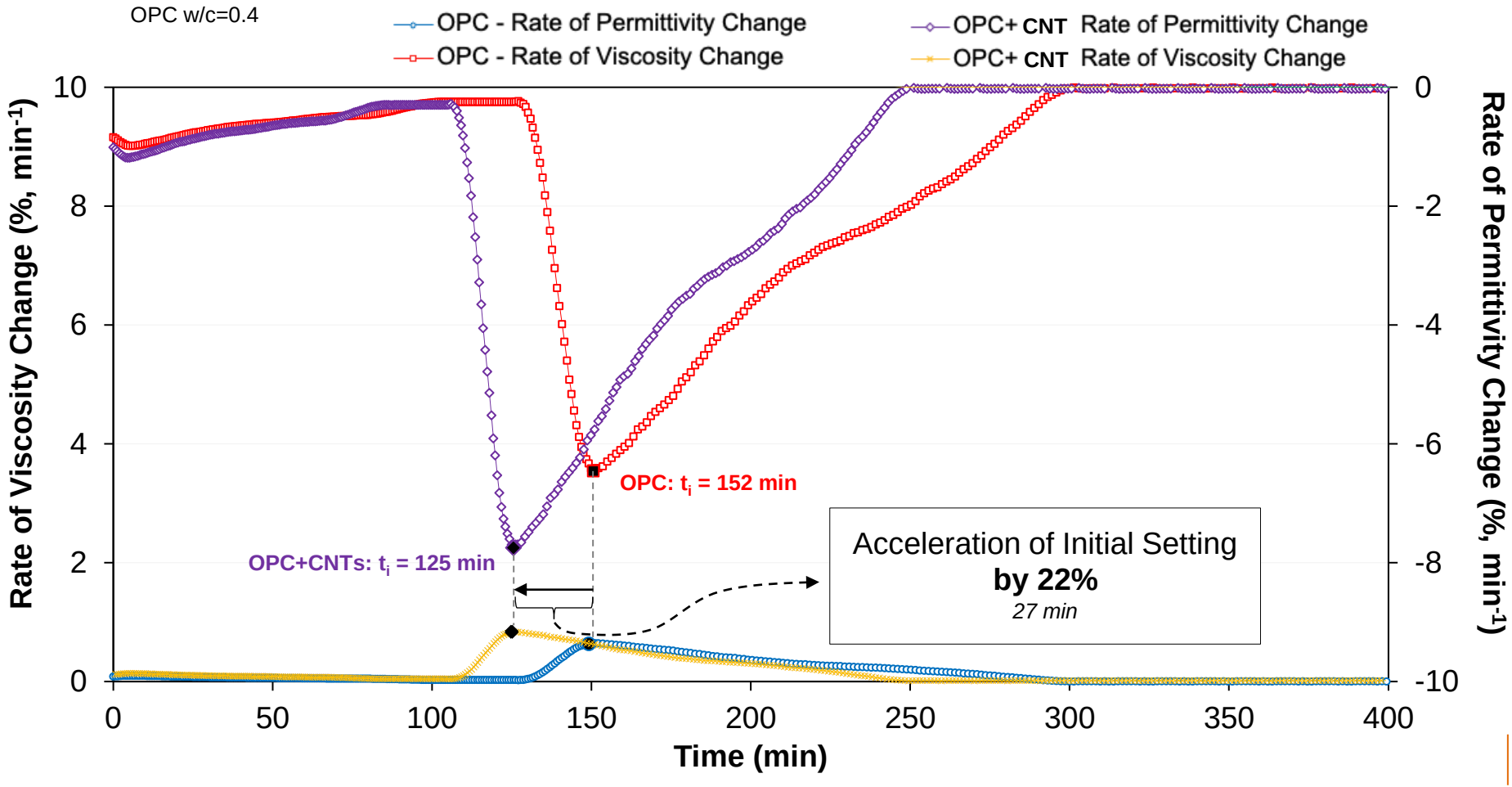
# Rate of Viscosity and Permittivity Change of OPC Cement Paste



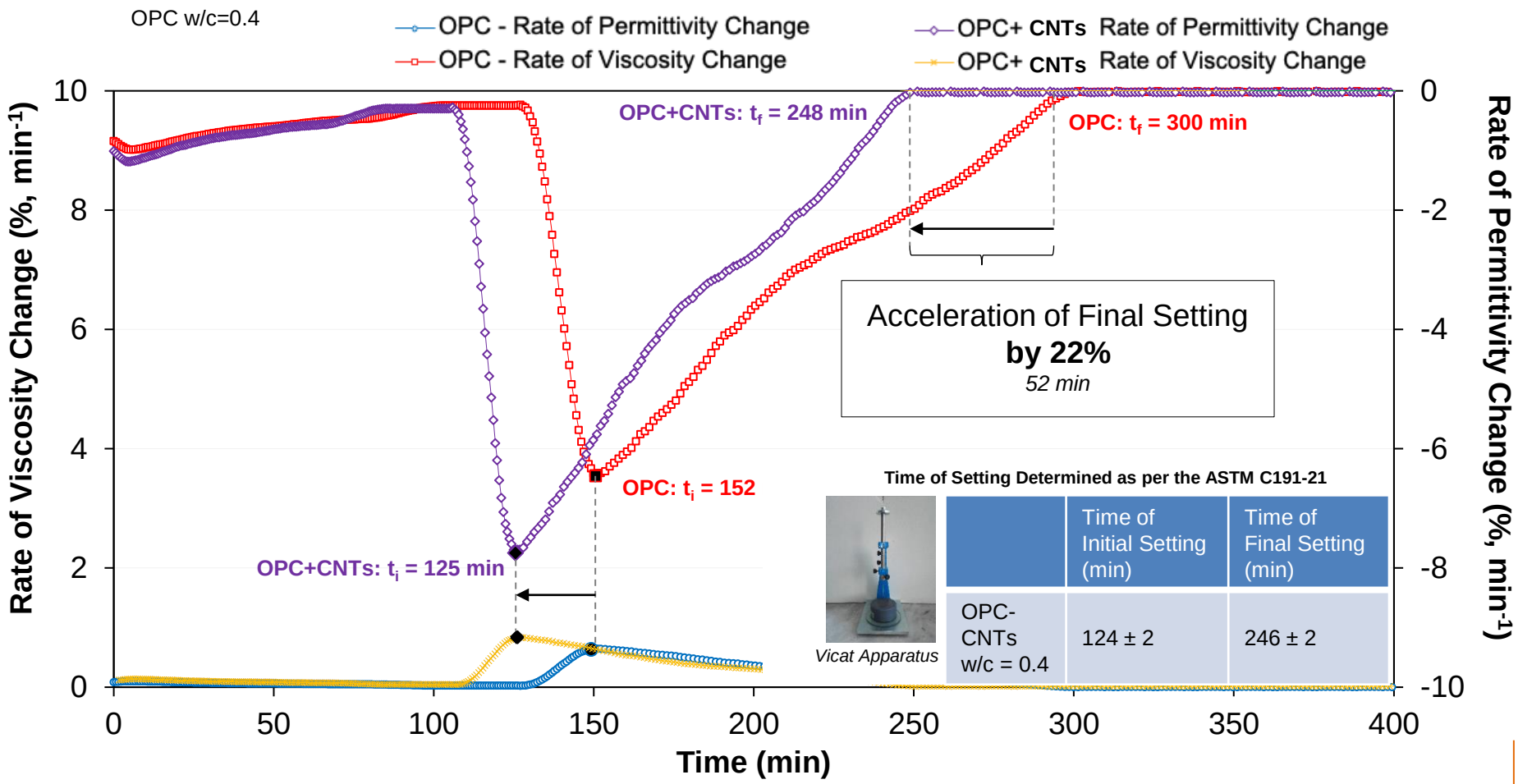
# Rate of Viscosity and Permittivity Change of OPC Cement Paste



# Effect of Monodispersed CNTs on the Time of Initial Setting of OPC Cement Paste



# Effect of Monodispersed CNTs on the Time of Initial Setting of OPC Cement Paste



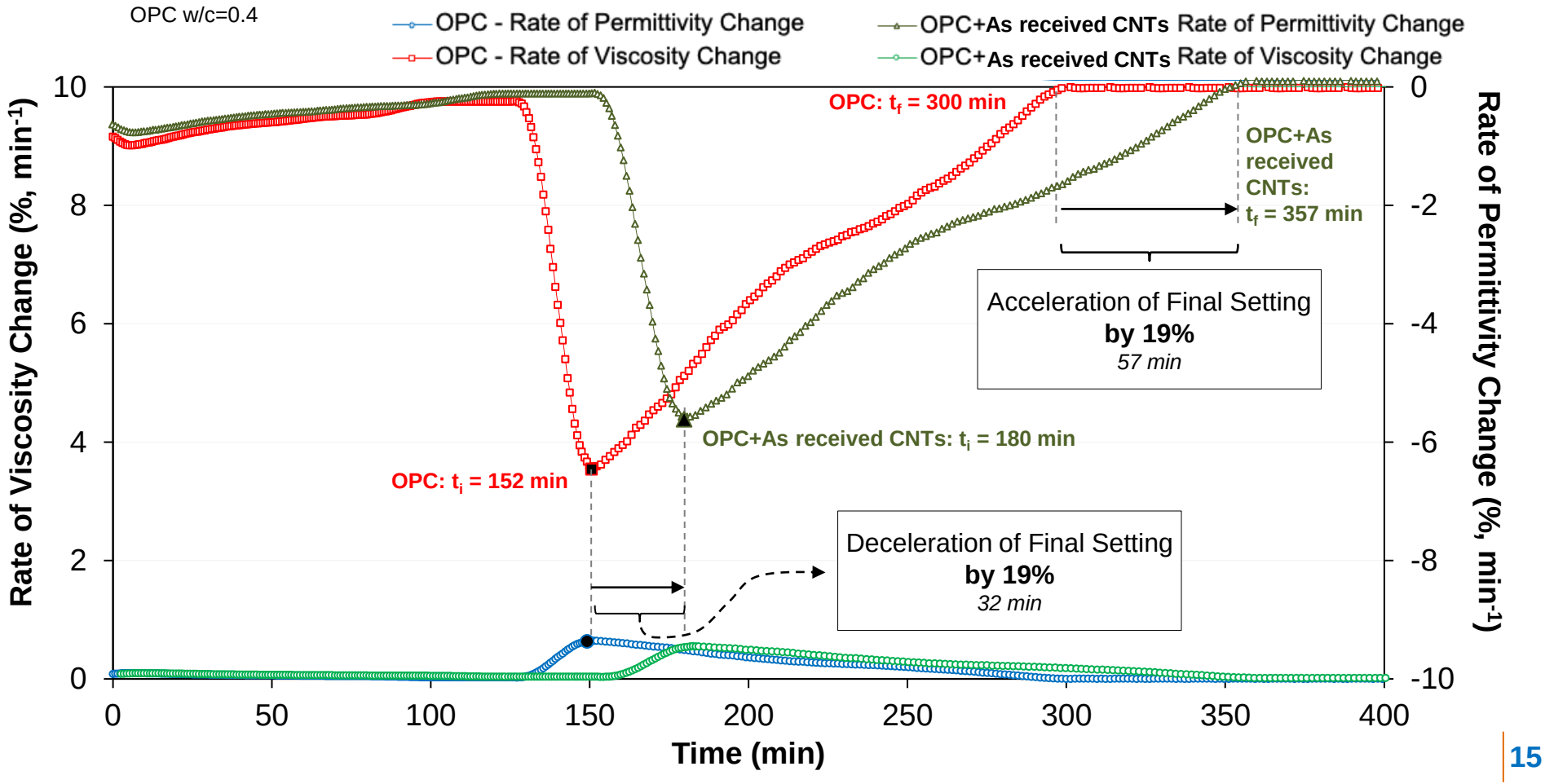

Time of Setting Determined as per the ASTM C191-21



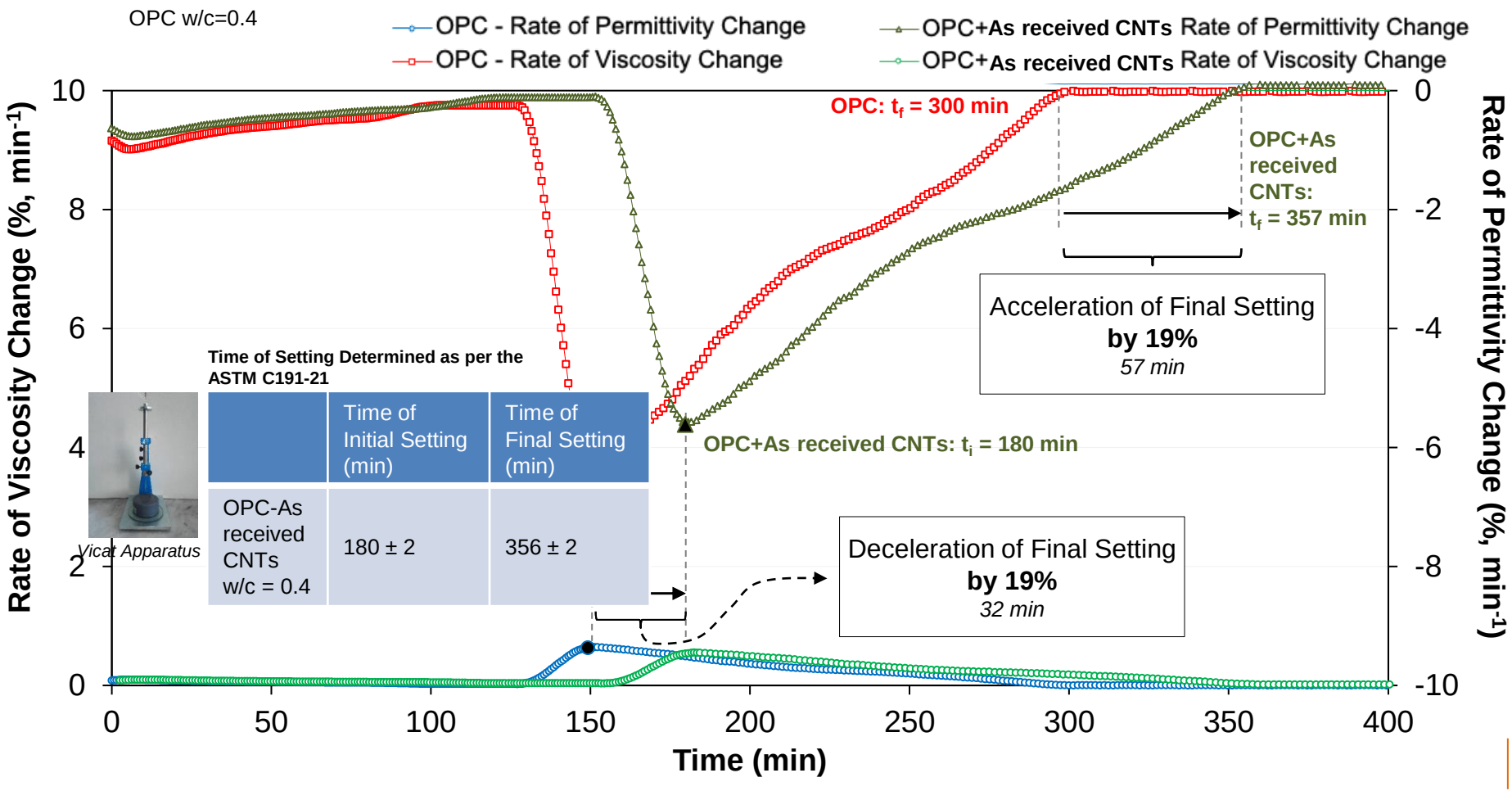
*Vicat Apparatus*

| | Time of Initial Setting (min) | Time of Final Setting (min) |
|---|---|---|
| OPC-CNTs w/c = 0.4 | 124 ± 2 | 246 ± 2 |

# Effect of As received CNTs on the Time of Setting of OPC Cement Paste



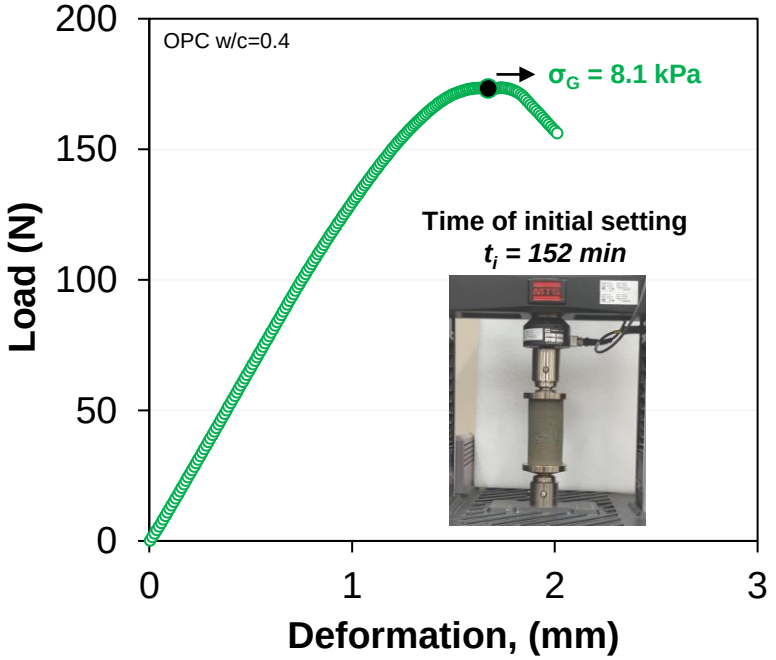
# Effect of As received CNTs on the Time of Setting of OPC Cement Paste



# Relationship Between Green Strength and Electrical Energy Density of OPC Mortars

## Green Strength, $\sigma_G$

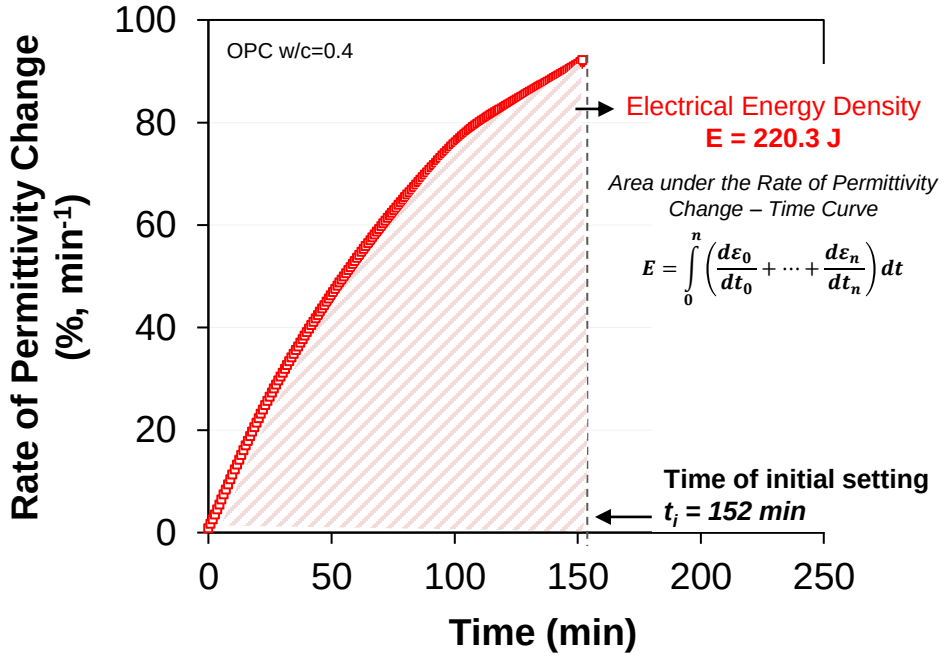
Compressive Strength of concrete in plastic state




High green strength values

## Electrical Energy Density, *E*

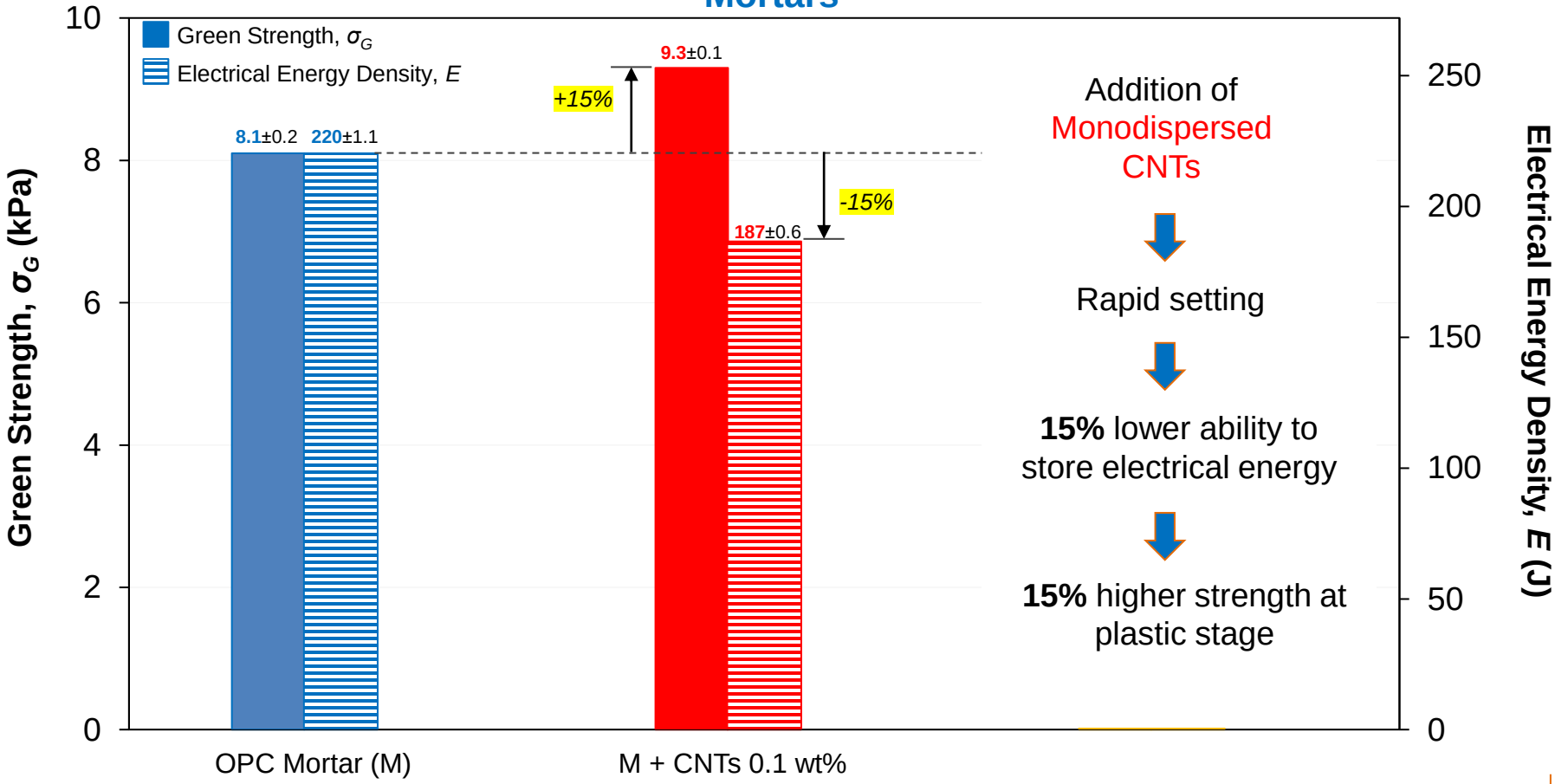
Amount of electrical energy stored in concrete's volume




High Electrical Energy Density values

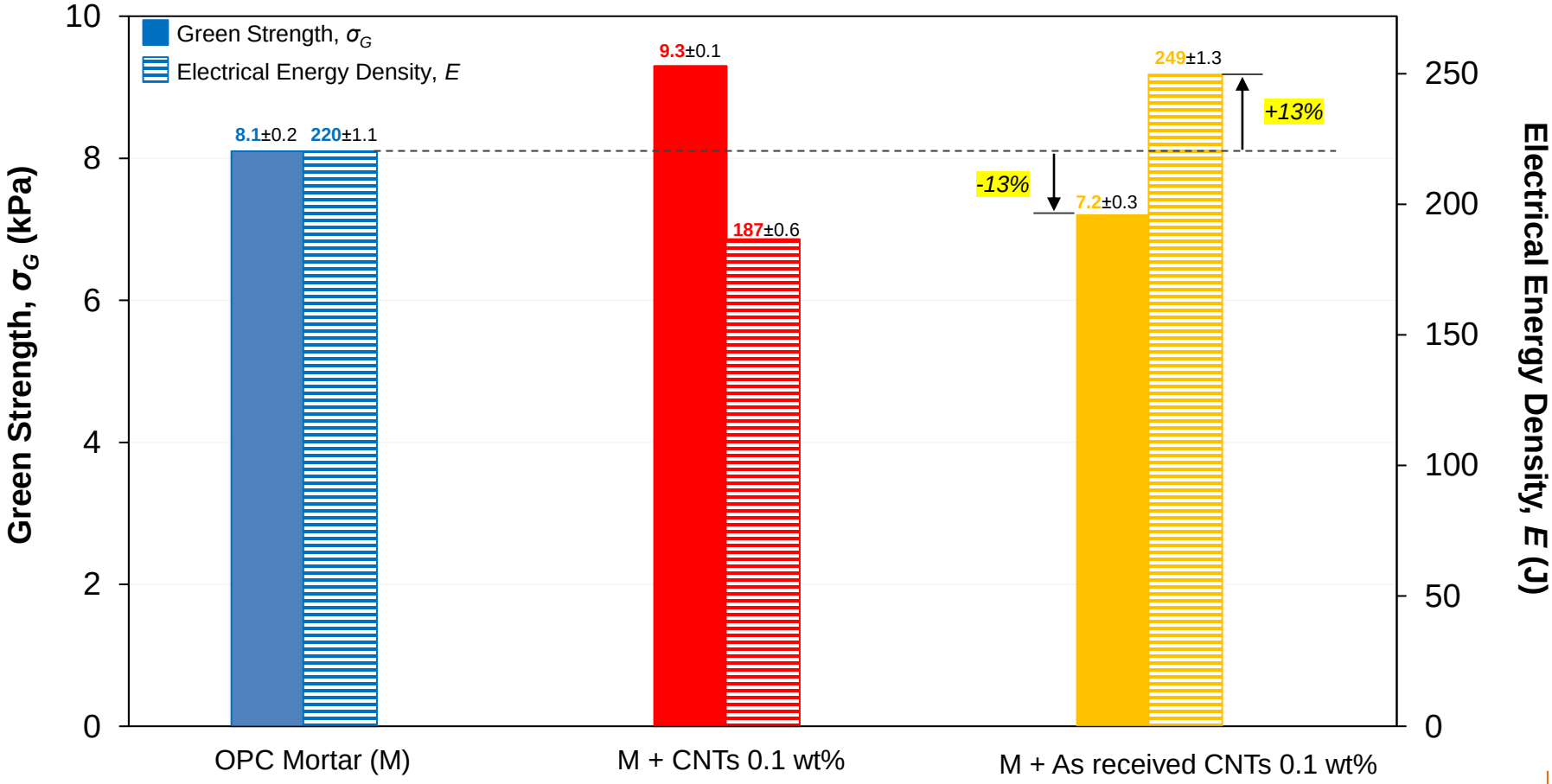
**Improved ability of concrete to retain its own shape after casting**

# Effect of Monodispersed CNTs on the Green Strength and Electrical Energy Density of Mortars




**Initial Setting Time** $t_i$ = 152 min $t_i$ = 125 min

# Effect of As received CNTs on the Green Strength and Electrical Energy Density of Mortars



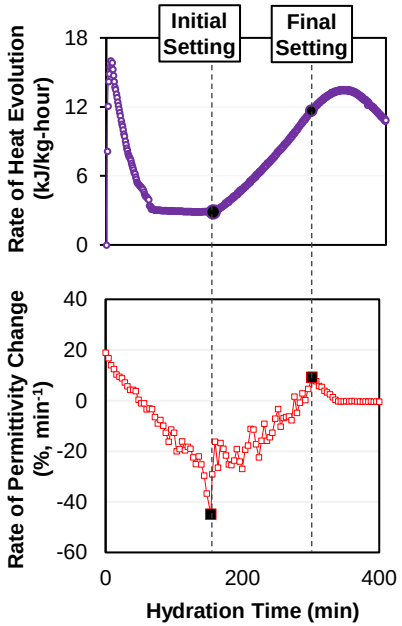

**Initial Setting Time** $t_i$ = 152 min | $t_i$ = 125 min | $t_i$ = 180 min

# Conclusions

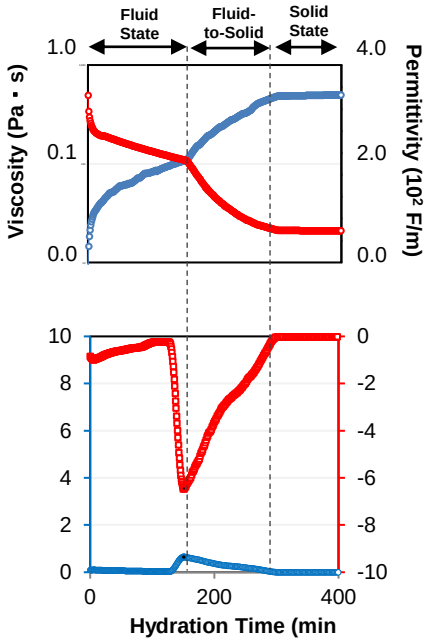
Electrochemical Impedance Spectroscopy: Dielectric Permittivity

Self-Sensing Function for monitoring fluid to solid transition of cementitious materials during the 3D printing process

**Setting Time**

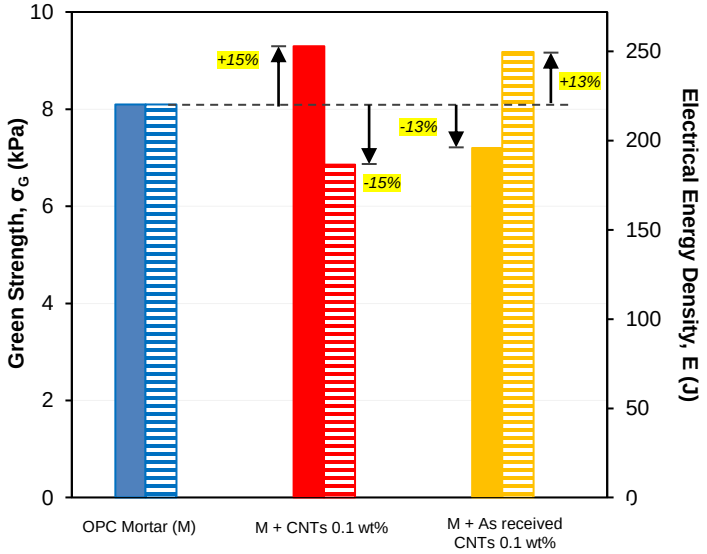



**Viscosity**



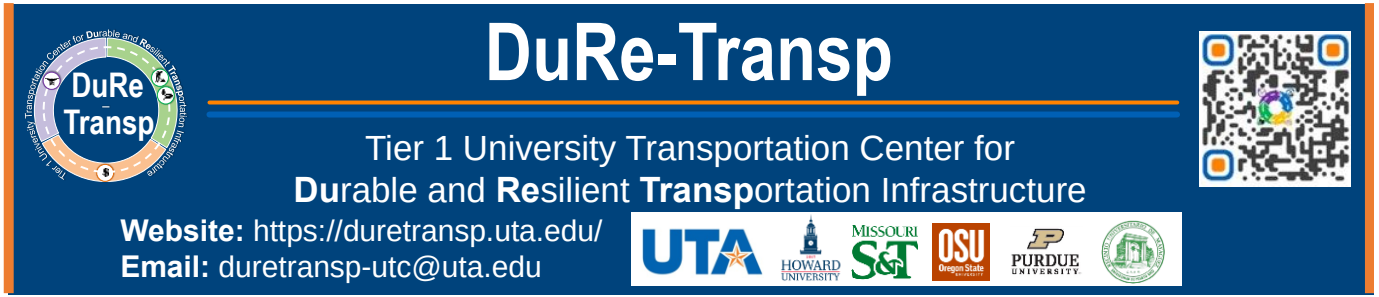

Relationship between Green Strength and Electrical Energy Density

- *Monodispersed CNTs* Acceleration of Hydration Kinetics → 15% Higher $\sigma_G$, 15% Lower *E*
- *As received CNTs* Deceleration of Hydration Kinetics → 13% Lower $\sigma_G$, 13% Higher *E*



## Acknowledgements

The authors would like to acknowledge the financial support of the U.S. Department of Transportation - University Transportation Centers Program “Tier 1 University Transportation Center for Durable and Resilient Transportation Infrastructure (DuRe-Transp)” (69A3552348339).



# Thank you!


DuRe Transp
DuRe-Transp
Tier 1 University Transportation Center for
Durable and Resilient Transportation Infrastructure
UTA
0:33.31