# ENHANCING AMBIENT-CURED GEOPOLYMER-BONDED LIGNOCELLULOSIC COMPOSITES (GLC) WITH DIRECT BINDER ADDITIONS OF AMINOSILANE AND COLLOIDAL NANO-SILICA:

## Workability, Setting Time, and Reactivity.


By

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# Lignocellulosic fillers in geopolymer composites

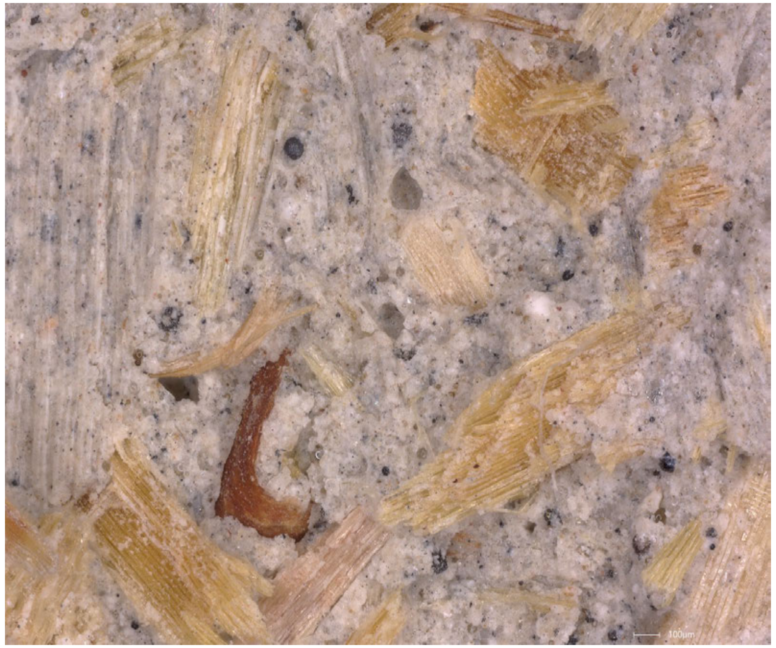
**Definition**: Natural, plant-based materials (e.g., wood fibres, agricultural residues such as coir fibre, hemp etc.).

## Advantages:

✓ Low-cost

✓ Abundant

✓ High strength-to-weight ratio



## Challenges:

- Matrix compatibility
- Polar-hydrophilic behavior
- Durability concerns?
- Microstructural disturbances.

## Applications:

- Construction
- Insulation
- Lightweight composites

**Research focus: Interfacial bonding | Fibre treatments | Matrix modifications**

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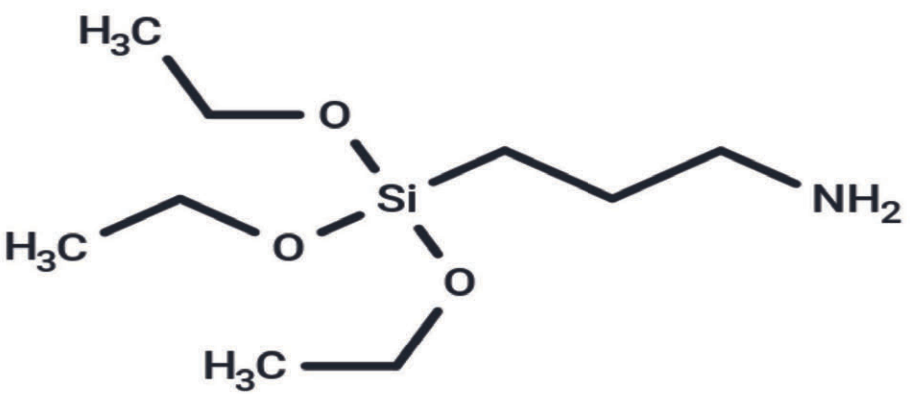
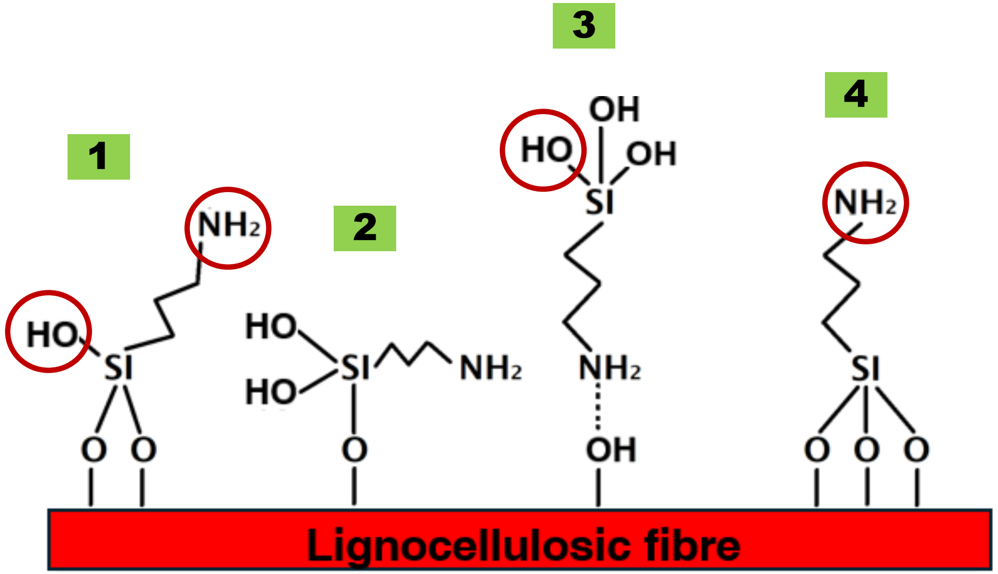
# Enhancing Ambient-Cured GLC: Aminosilane (APTES)

## 3-Aminopropyltriethoxysilane (APTES):

- Improves interfacial bonding
- Enhances filler-matrix compatibility

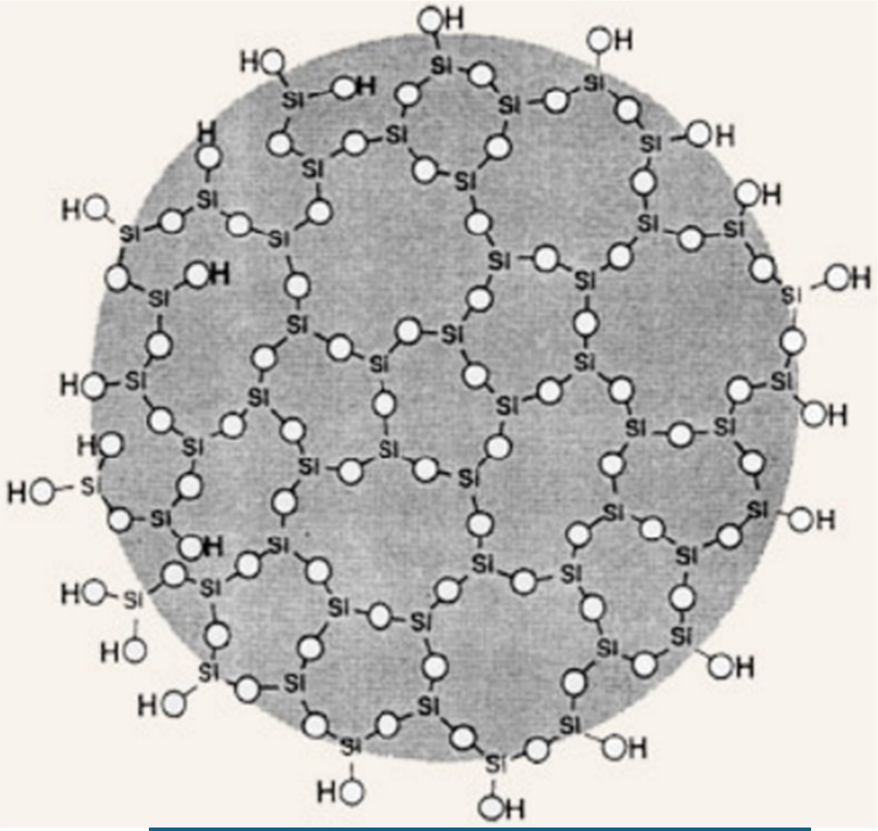



$$H_2N(CH_2)_3Si(OC_2H_5)_3 + 3\ H_2O \rightarrow H_2N(CH_2)_3Si(OH)_3 + 3\ C_2H_5OH$$

APTES   Water   Hydrolysed APTES   Ethanol

# Enhancing Ambient-Cured GLC: Colloidal Nano-Silica (CNS)



Credit: Vafaei *et al.* 2022 [1]

## CNS (Colloidal Nano-Silica)

- Strengthens geopolymer matrix
- Increases reactivity

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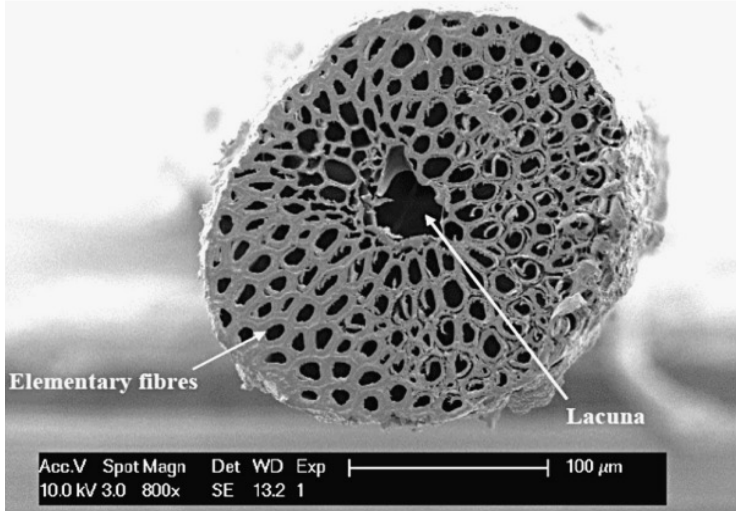
# Fibre Characteristics




SEM image of the cross-section of a typical coir technical fibre with the presence of lacuna and elementary fibres.

Credit: Tran *et al.* 2015 [2]

**Unique properties for elaboration in composite materials viz:**

- **High ductility with the highest failure strain among known plant fibres.**
- **Virtually available all year round.**
- **Ability to form semi-protective char with increasing temperature.**
- **Remarkable low density due to the sieve cells (high porosity of 22 to 50%).**
- **Unaffected by moisture and dampness (i.e., decay resistant).**

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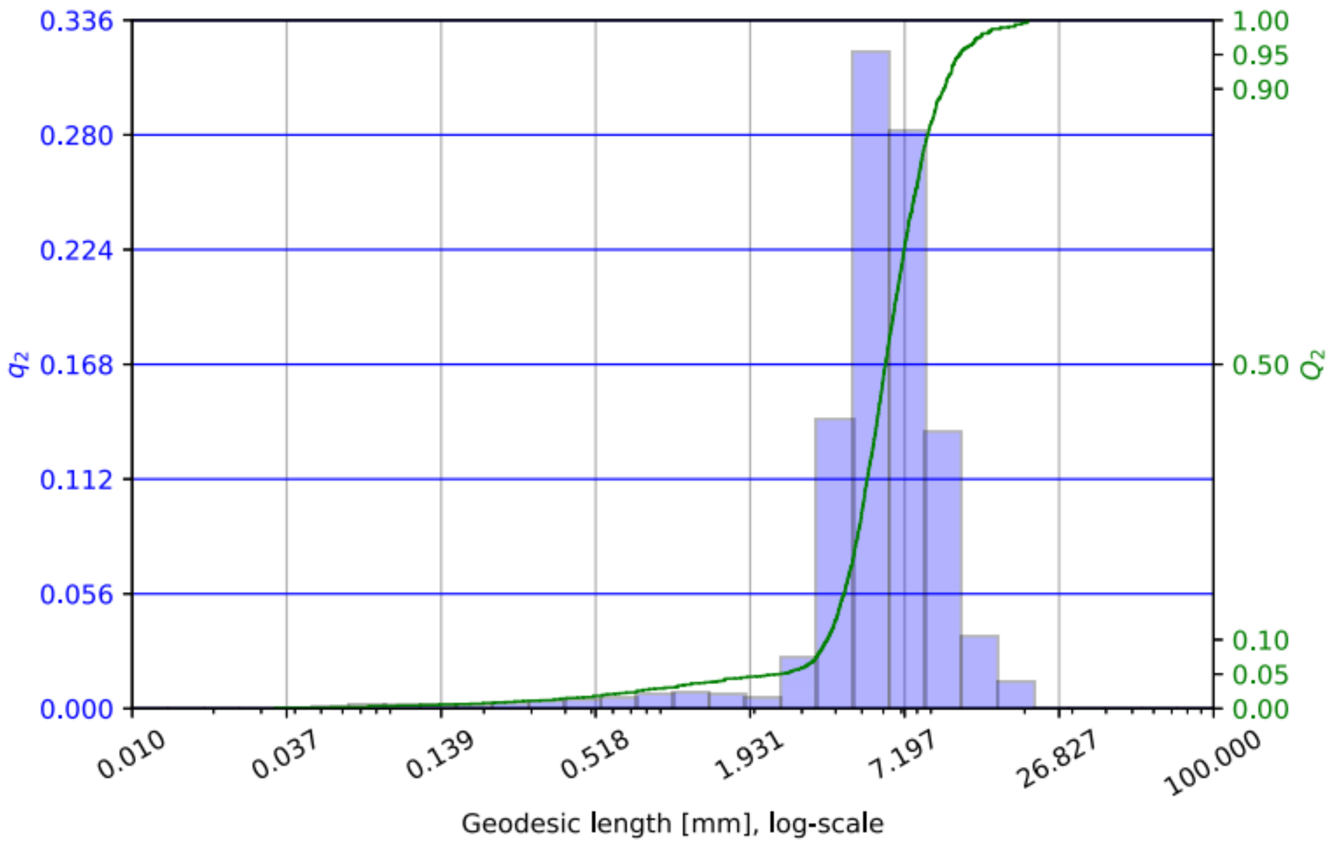
# Fibre Characteristics

**Table 4.** Chemical composition and microfibrillar angle of coir and some commonly known fibres.

| Fibre | Cellulose (wt.%) | Hemicellulose (wt.%) | Lignin (wt.%) | Microfibrillar angle [°] |
|---|---|---|---|---|
| Coir | 32 – 43 | 0.15 – 0.25 | 40 – 45 | 30 – 31 |
| Ramie | 62 – 85 | 3 – 8 | 0.5 – 9 | 69 – 83 |
| Jute | 61 – 71 | 13 – 20 | 5 – 13 | 8 |
| Hemp | 68 – 75 | 15 – 22 | 4 – 10 | 2 – 6 |
| Kenaf | 31 – 72 | 20 – 34 | 8 – 21 | 2 – 6 |
| Flax | 64 – 75 | 10 – 20 | 2 – 5 | 5 – 10 |
| Sisal | 60 – 78 | 10 – 38 | 8 – 14 | 10 – 25 |

# Fibre Characteristics

### Graphical representation




### Percentiles

| % | Geodesic length [mm] |
|---|---|
| 0.00 | 0.034 |
| 5.00 | 2.505 |
| 10.00 | 3.734 |
| 50.00 | 6.124 |
| 90.00 | 10.066 |
| 95.00 | 11.495 |
| 100.00 | 20.641 |

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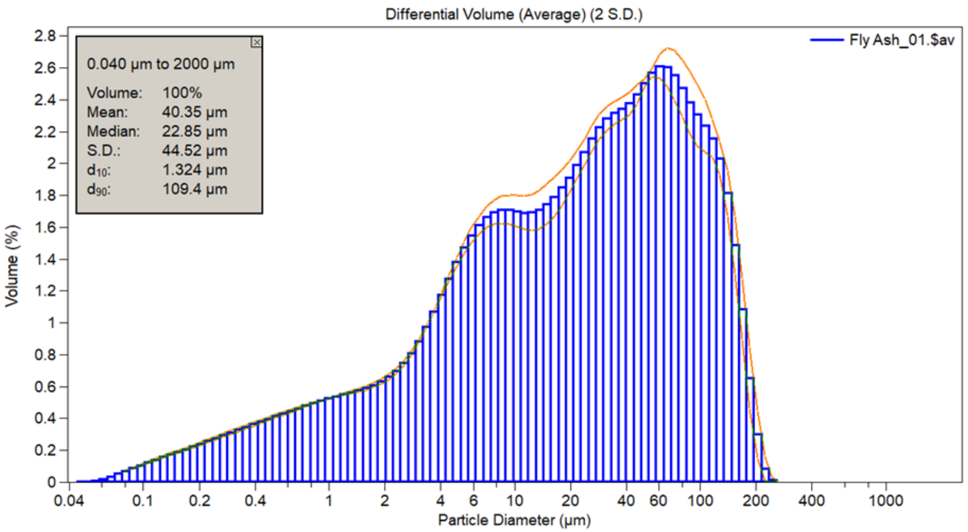
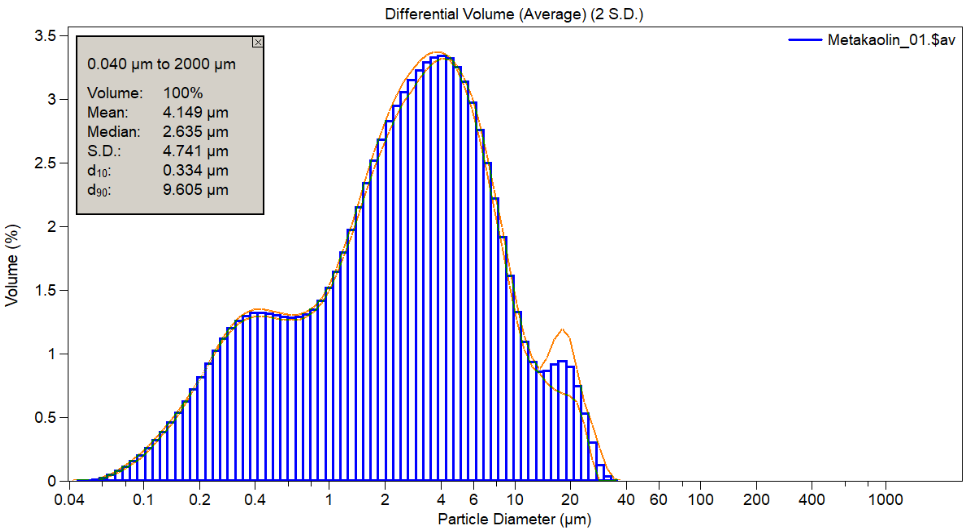
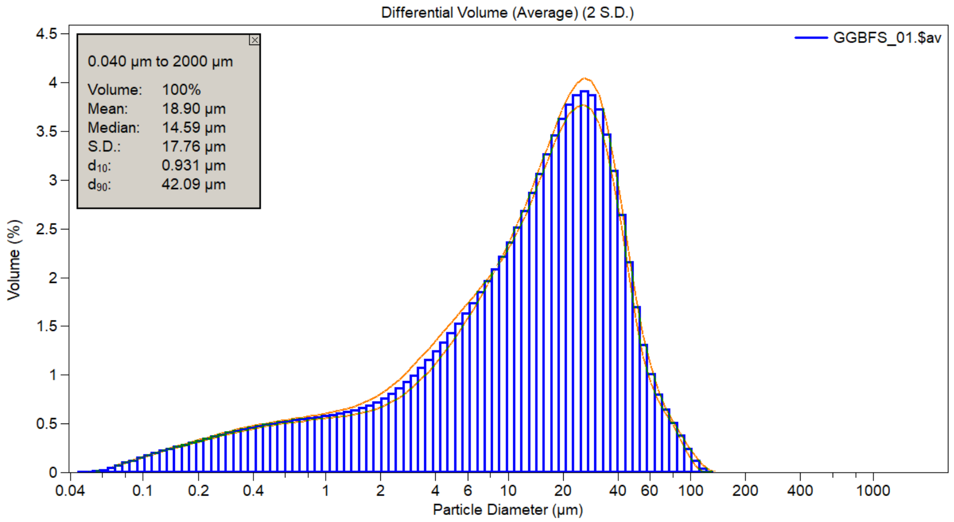
# Particle Size Distribution of Precursors (Granulometry)




| Parameter | Fly ash | Metakaolin | GGBFS |
|---|---|---|---|
| **Mean (µm)** | 40.4 | 4.15 | 18.9 |
| **Median (µm)** | 22.9 | 2.64 | 14.6 |
| **Standard Deviation S.D. (µm)** | 44.5 | 4.74 | 17.8 |
| **$d_{10}$ (µm)** | 1.32 | 0.33 | 0.93 |
| **$d_{90}$ (µm)** | 109.0 | 9.6 | 42.1 |

****NB: only formulations incorporating MK will infiltrate the fibres.**

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# Enhancers (additives)

**APTES**:

Levasil® CB8 (Nouryon, Sundsvall, Sweden)

| γ-Aminopropyltriethoxysilane | Property* |
|---|---|
| Physical Form | Liquid |
| Colour | Clear, colourless |
| Specific Gravity at 25 °C | 0.95 |
| Boiling Point at 760 mm Hg | 220 °C |
| Refractive Index, $n_D$ at 25 °C | 1.420 |
| Flash Point, Pensky-Martens Closed Cup[a] | 96 °C |

**CNS**:

Siliquest A-1100™ (Momentive Performance Materials GmbH, Leverkusen, Germany)

| Colloidal silica | Property* |
|---|---|
| Density at 20 °C | 1.39 – 1.40 g $cm^{-3}$ |
| $SiO_2$ content | 49.9 – 51.5 wt.-% |
| pH at 20 °C | 9.1 – 9.9 |
| Viscosity at 20 °C | ≤15 cP |

"*": Information provided by supplier/Manufacturer, "a": According to ASTM Method D 93

# Ambient Cured Geopolymer Cement Composition

## (K, Ca)-poly(sialate-siloxo)

**Table 1.** Stoichiometric ratios of geopolymer cement variants

| Stoichiometric ratio | Control | Aminosilane Mod. | CNS Mod. |
|---|---|---|---|
| $SiO_2:Al_2O_3$ | 4.37 | 4.37 | 4.47 |
| $K_2O:Al_2O_3$ | 1.01 | 1.01 | 0.95 |
| $K_2O:SiO_2$ | 0.10 | 0.10 | 0.09 |
| $H_2O:K_2O$ | 27.72 | 27.72 | 27.72 |
| $SiO_2:H_2O$ | 0.35 | 0.35 | 0.38 |
| Additive content | - | 0.10 wt.% | 1.45 wt.% |

# Mixing Process

Fly Ash

+

GEOSIL 14517 (MR 1.7)

10 Min.

+

Metakaolin

5 Min.

60 Min.

Ageing

+

GGBFS

3 Min.

+

**Enhancers: APTES, CNS**

Coir fibre

Planetary mixer

Casting + curing (240 Min.)

Geopolymer bonded coir fibre composite

**Mould dimensions**: 450 × 450 × 16 $mm^3$ (L×W×T)

**Pressure:** 15 Mpa

**Temp:** 20 ± 2 °C

**RH :** 55 ± 5 %

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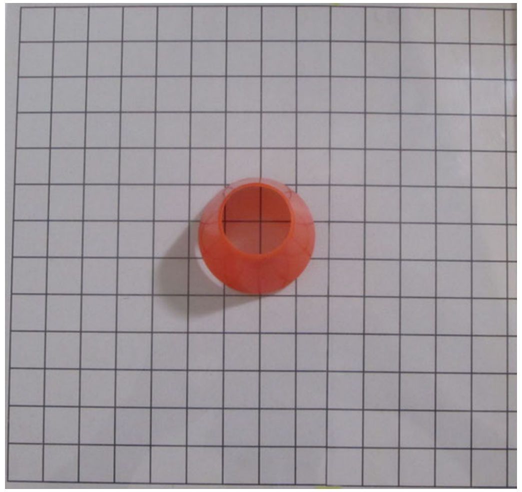
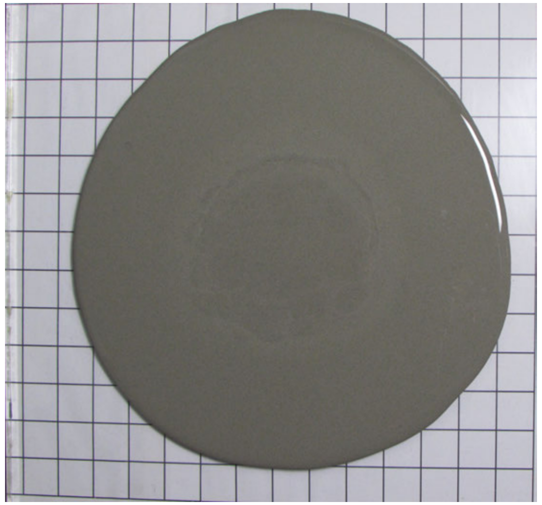
# Results: Workability

**Mini-cone Spread Test**

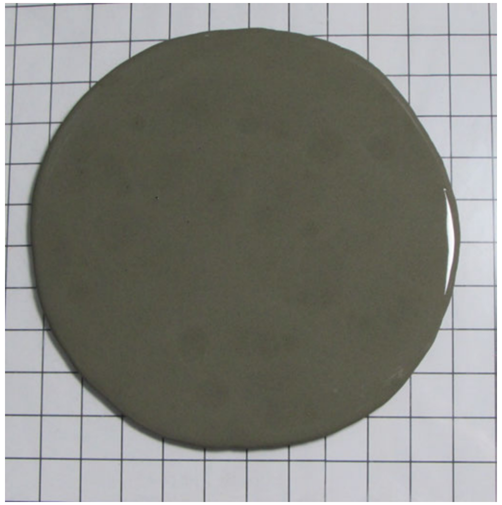


**Control**



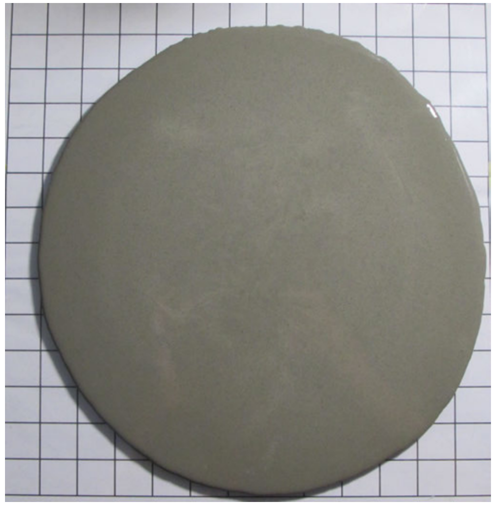
**256.7 ± 13.4[a]**

**Aminosilane Mod.**



**200.5 ± 10.8[c]**

**CNS Mod.**



**249.1 ± 8.3[b]**

**Cone Dimensions:**

$U_D$ = 36 mm

$L_D$ = 60 mm

h = 60 mm

**Grid Dimensions:**

2 × 2 $cm^2$

**NB: Spread diameters above 220 mm (240 – 260 mm) provides the best workability.

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# Results: Setting Behaviour

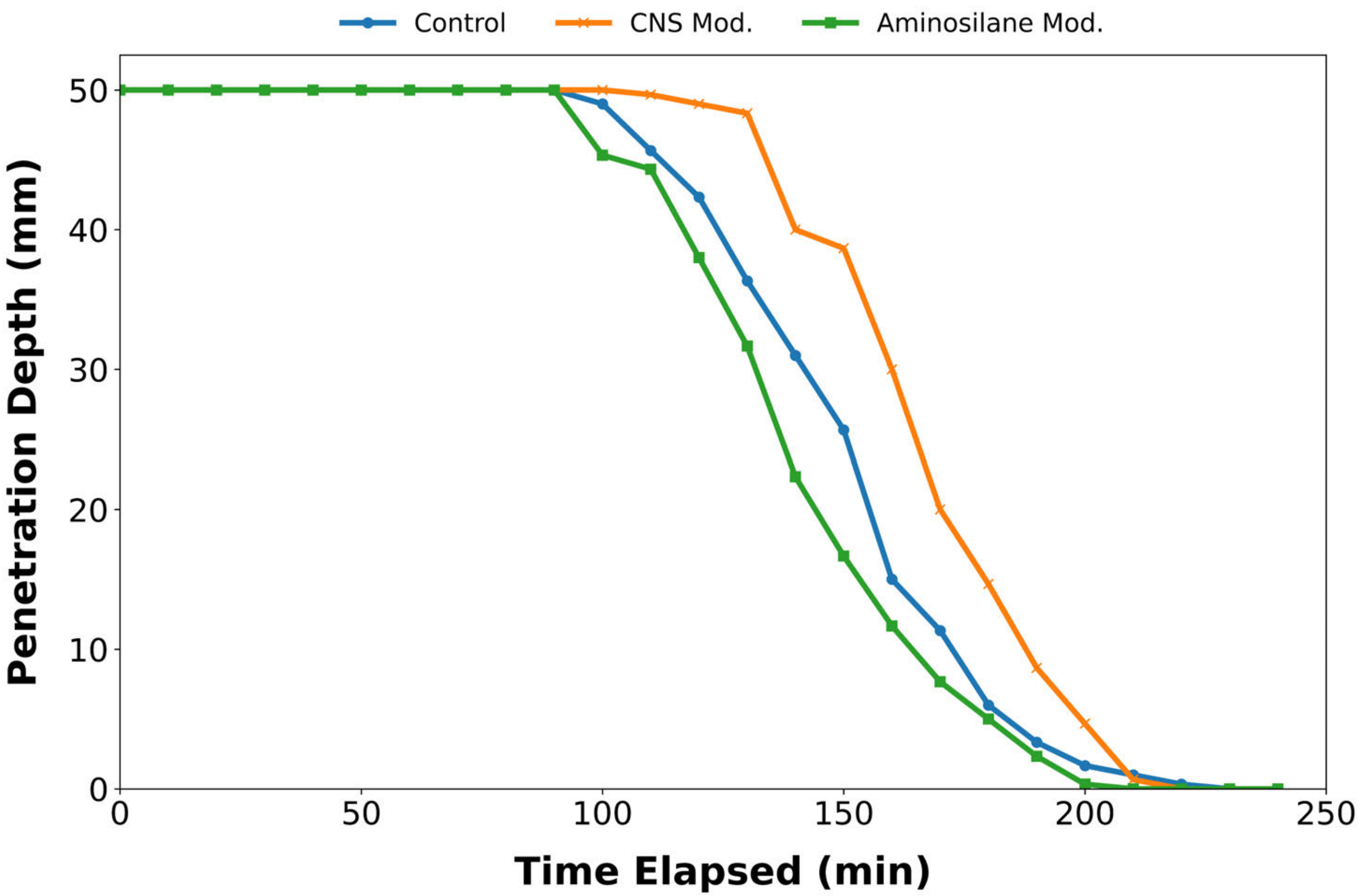



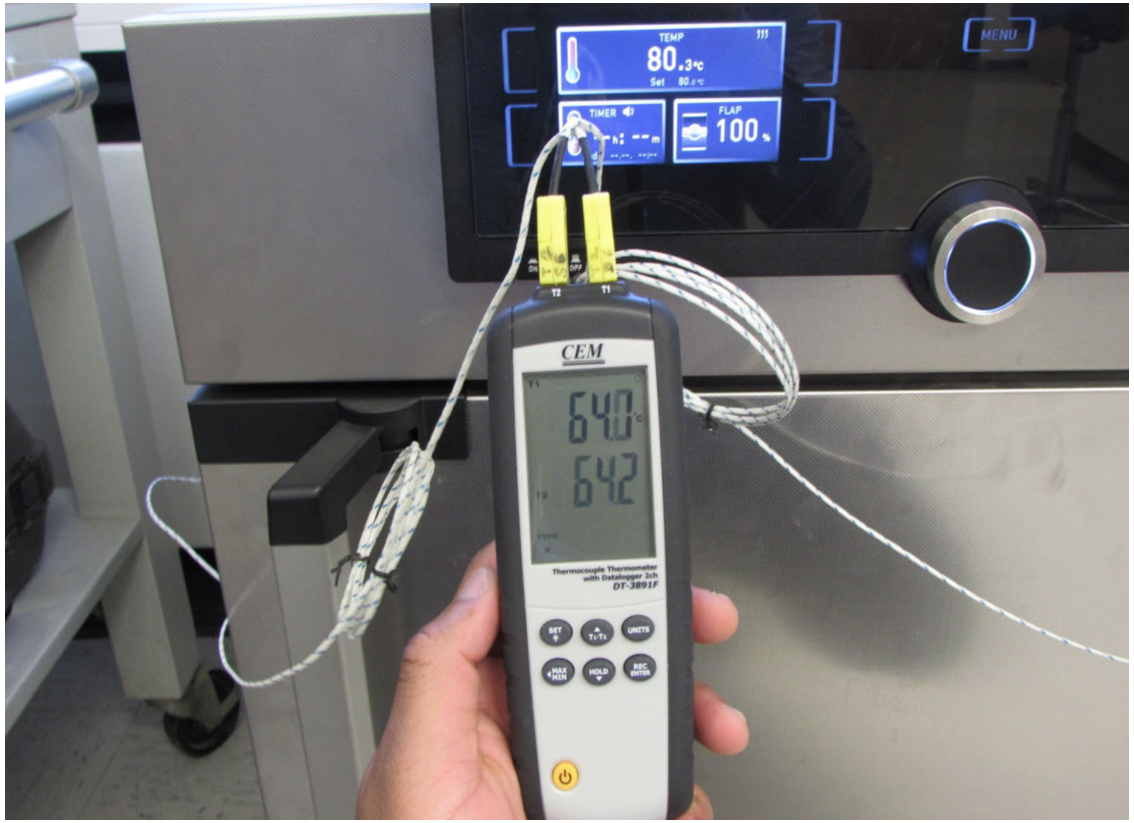

**Table 5.** Summary of setting times

| Variant | IST (min) ± SD | FST (min) ± SD |
|---|---|---|
| Control | 101.7 ± 9.4 | 203.3 ± 18.9 |
| Aminosilane Mod. | 96.7 ± 8.5 | 193.3 ± 17.0 |
| CNS Mod. | 106.7 ± 2.4 | 213.3 ± 4.7 |

"**IST**": Initial Setting Time, "**FST**": Final Setting Time.

# Results: Reactivity Test

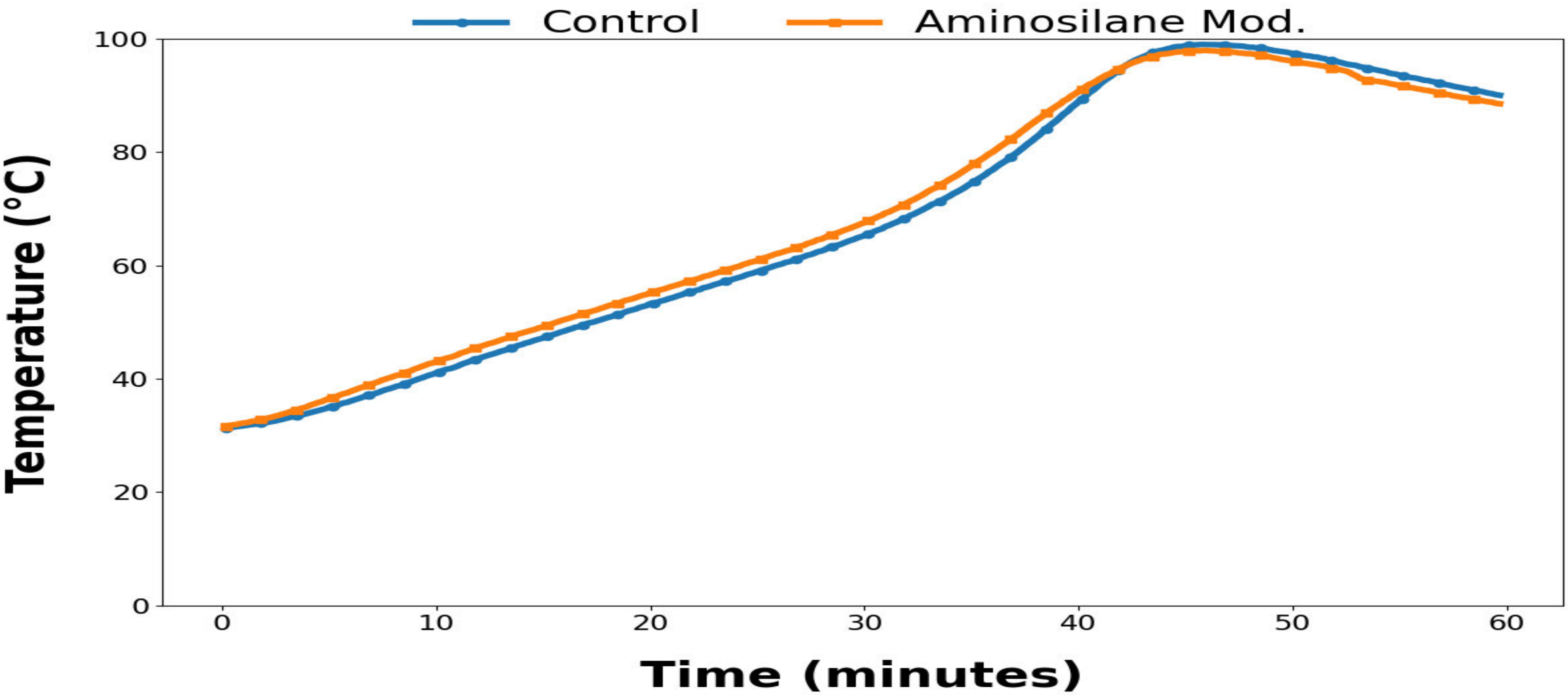
## Cumulative exothermal heat flow (80 °C)





**NB: For guide on this method, consult Technical Paper #26 (2019), geopolymer.org**

# Results: Reactivity Test_Control vs Aminosilane Mod.

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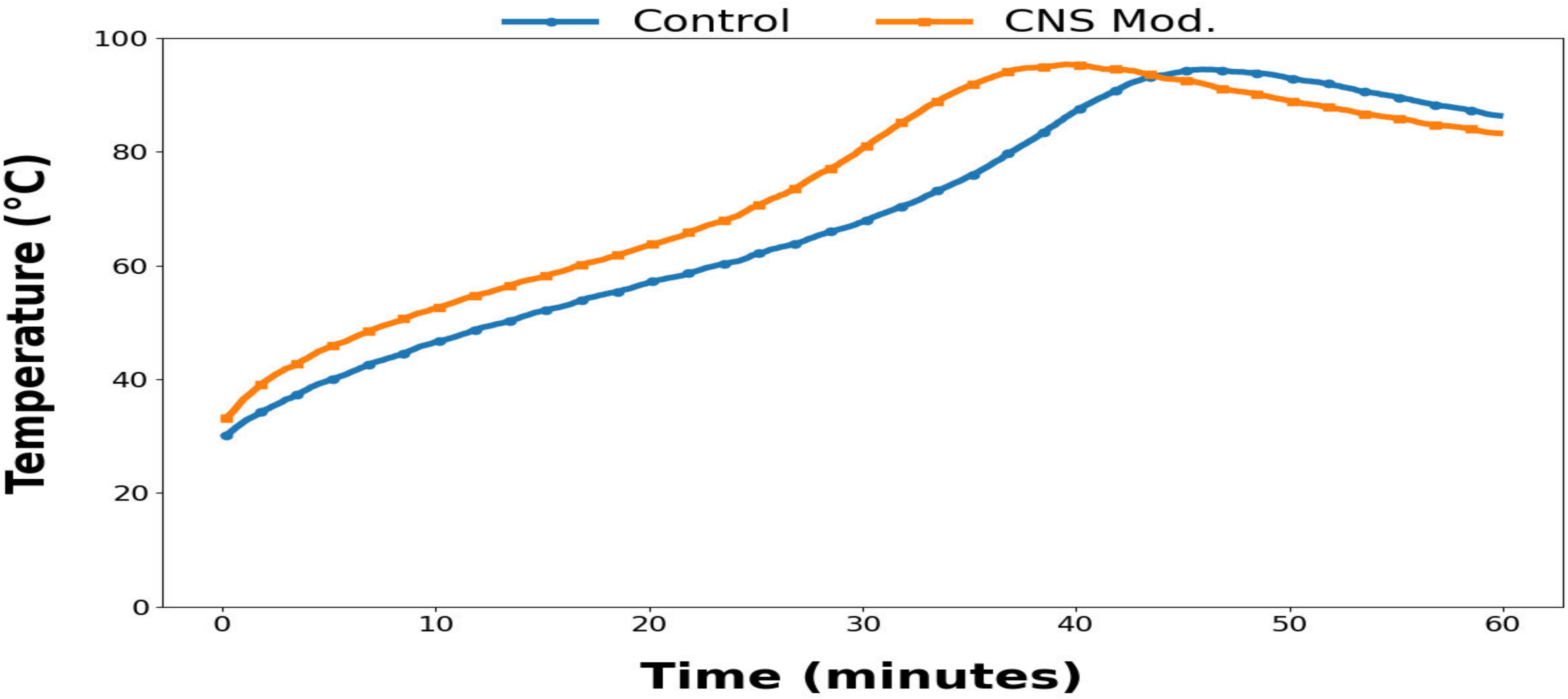
# Results: Reactivity Test_Control vs CNS Mod.

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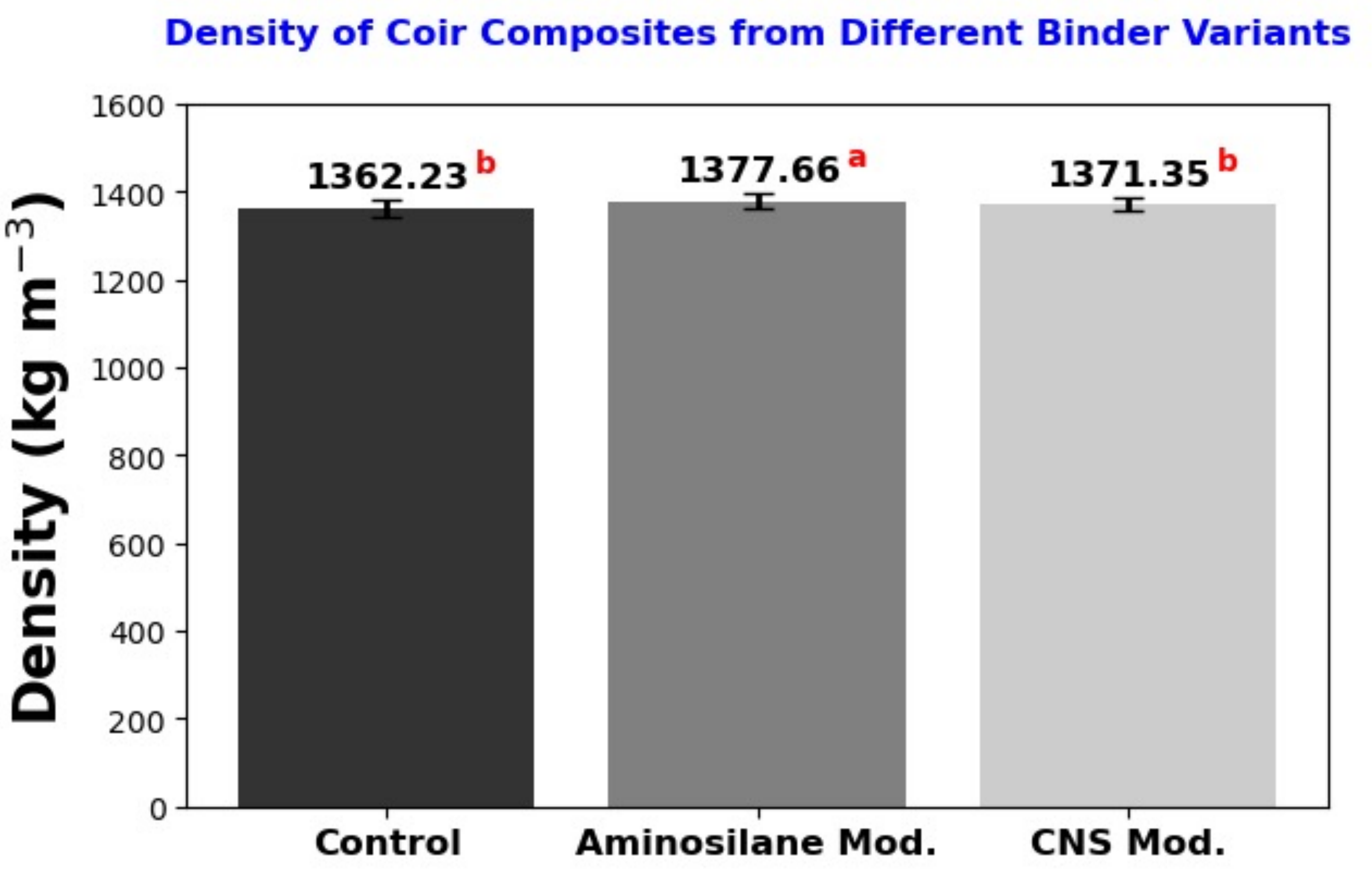
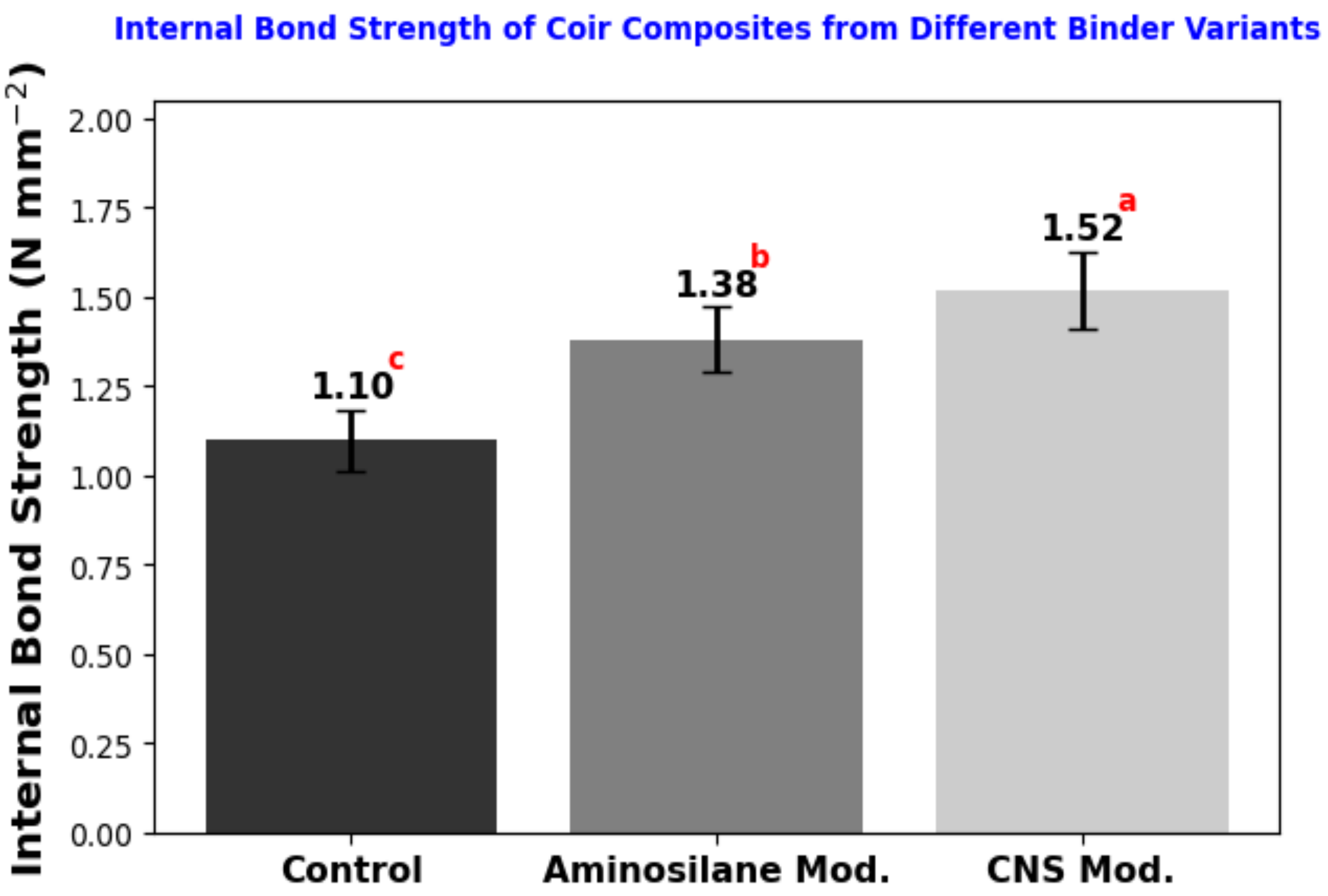
# Coir Composite Properties: Density




***f* = 20.58, *p* = 0.00**

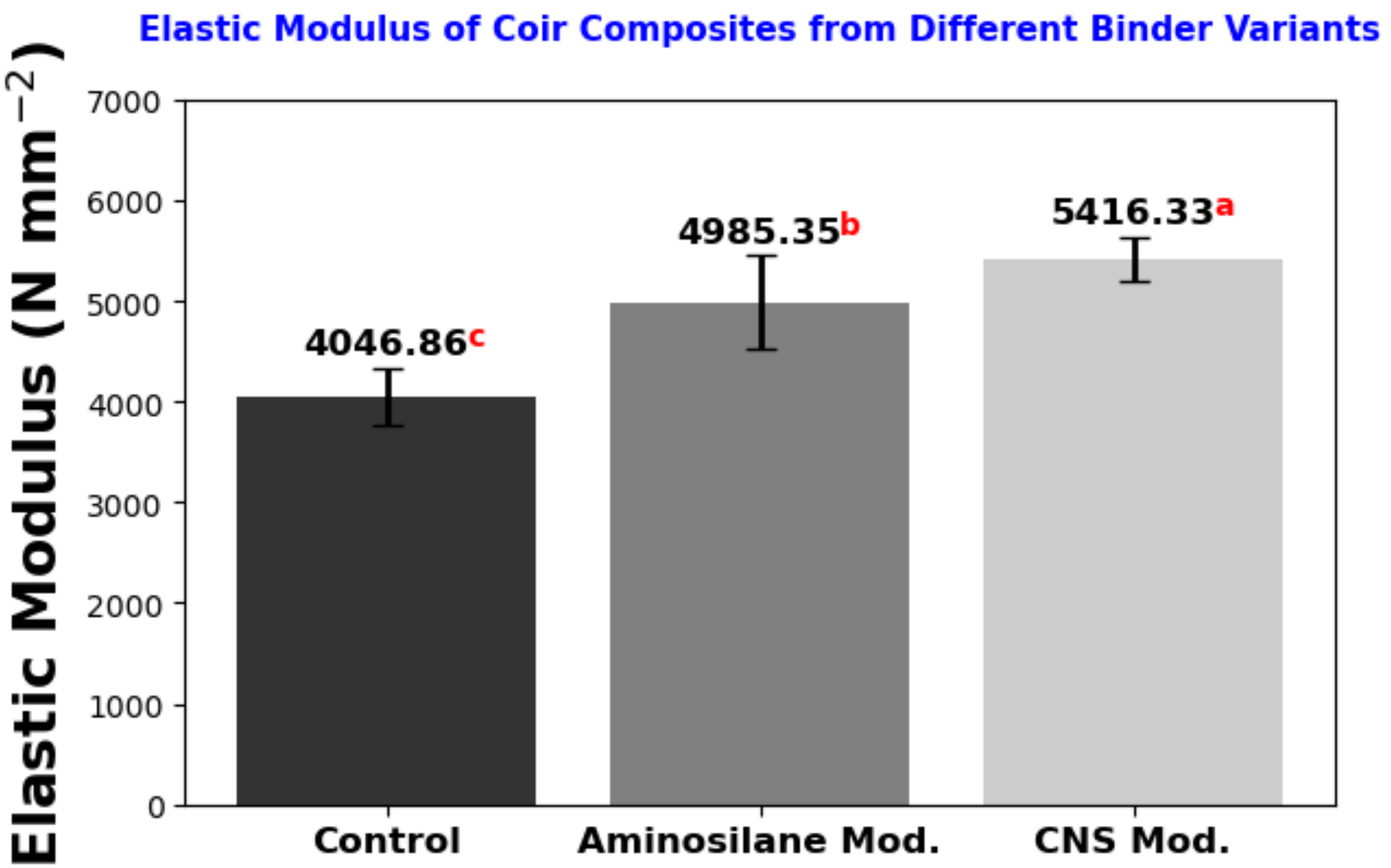
**NB: Evidence of direct correlation between cooling rate and density of composites.**

# Internal Bond Strength (IBS)




$f = 50.24$, $p = 0.00$

# Elastic Modulus ($E_m$)




$f$ = 32.95, $p$ = 0.00

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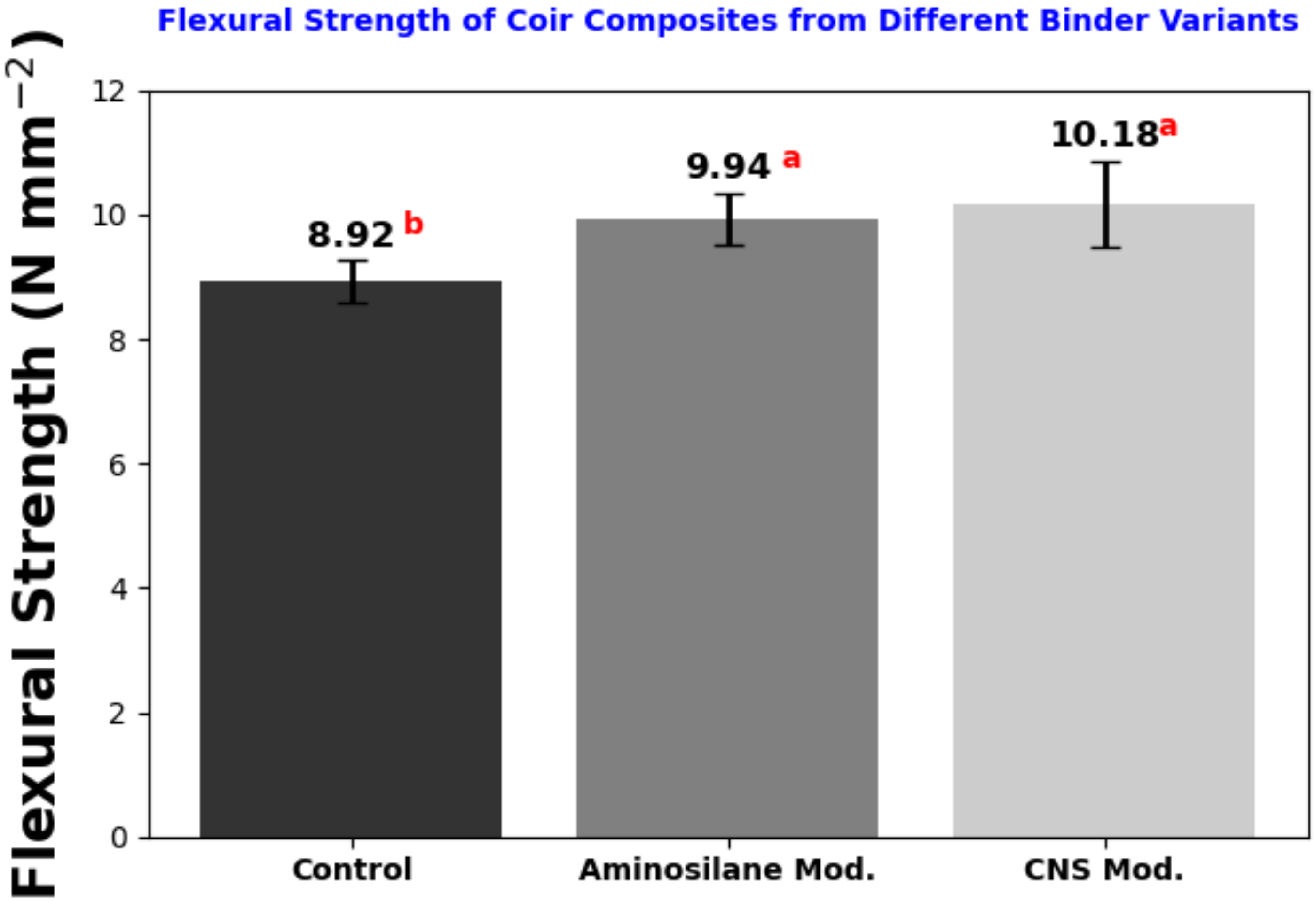
# Flexural Strength ($f_m$)




$f$ = 33.76, $p$ = 0.00

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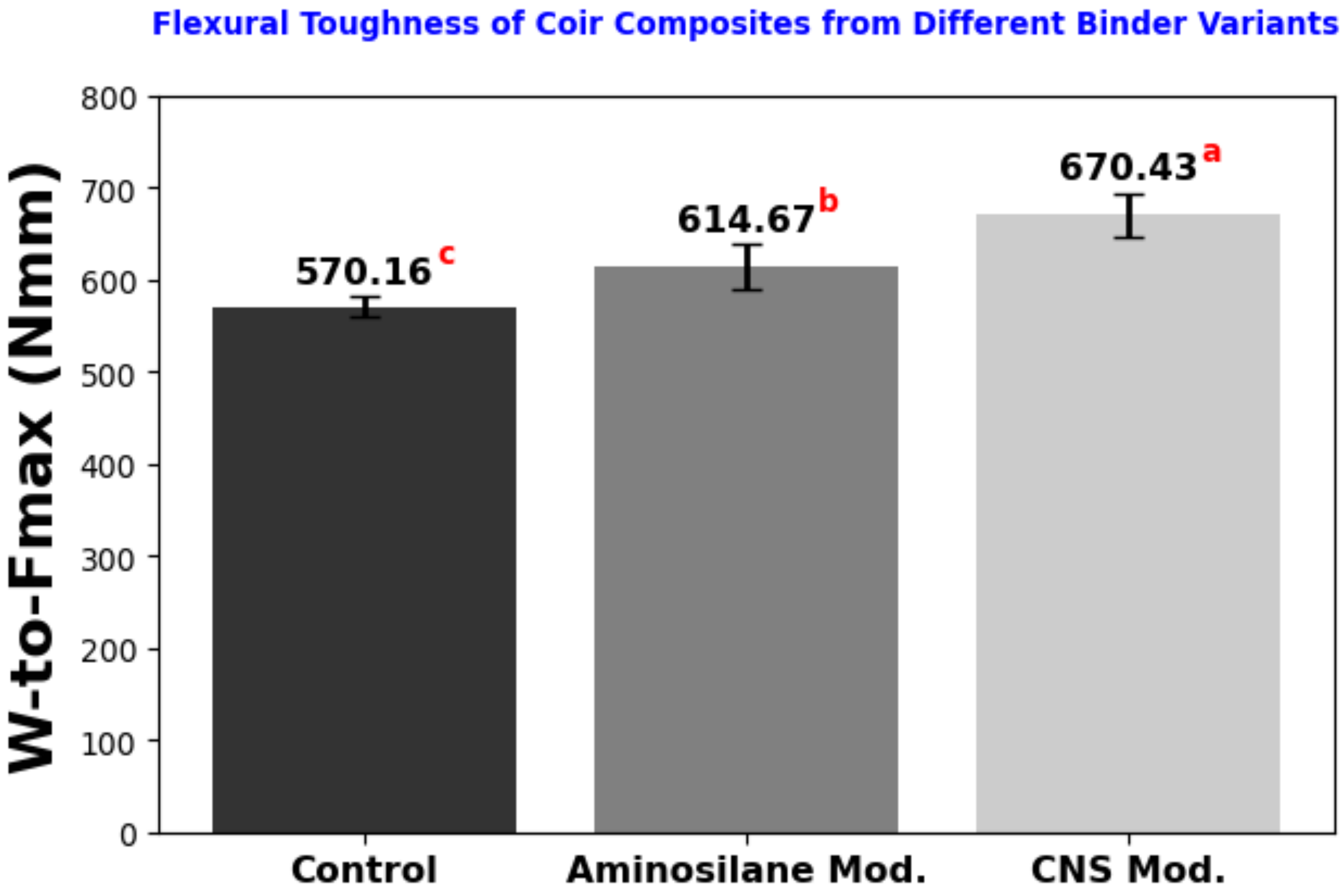
# Flexural Toughness (W-to-Fmax)




***f*** **= 35.18,** ***p*** **= 0.00**

# Conclusion

- Direct binder modification with additives enhances composite properties effectively.
- Geopolymer-bonded lignocellulosic composites with desirable properties can be achieved through ambient curing.
- Potential for further optimization (Future work).

# Acknowledgements

# Contact Information

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**https://www.uni-goettingen.de/en/643916.html**

# Cited Literature

[1] Vafaei, A., Choobbasti, A.J., Afrakoti, M.P. et al. (2022). The presence of colloidal nano silica in sandy soils: a review. Arab J Geosci 15, 582. DOI: 10.1007/s12517-022-09908-4

[2] Tran, L., Minh, T. N., Fuentes, C., Chi, T. T., Van Vuure, A., & Verpoest, I. (2015). Investigation of microstructure and tensile properties of porous natural coir fibre for use in composite materials. *Industrial Crops and Products*, 65, 437-445. DOI: 10.1016/j.indcrop.2014.10.064

# THANKS FOR LISTENING

Please Feel Free to ask Questions.