Natural Fiber Reinforced Geopolymer Composites

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Supported by
US Air Force Office of Scientific Research,
Army Research Office, US Army Corps of Engineers and
Dow Chemical Company

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Keramos Mug Drop Competition - ACERS 2005

Testing for leaks ………

The 5 storey plummet ………

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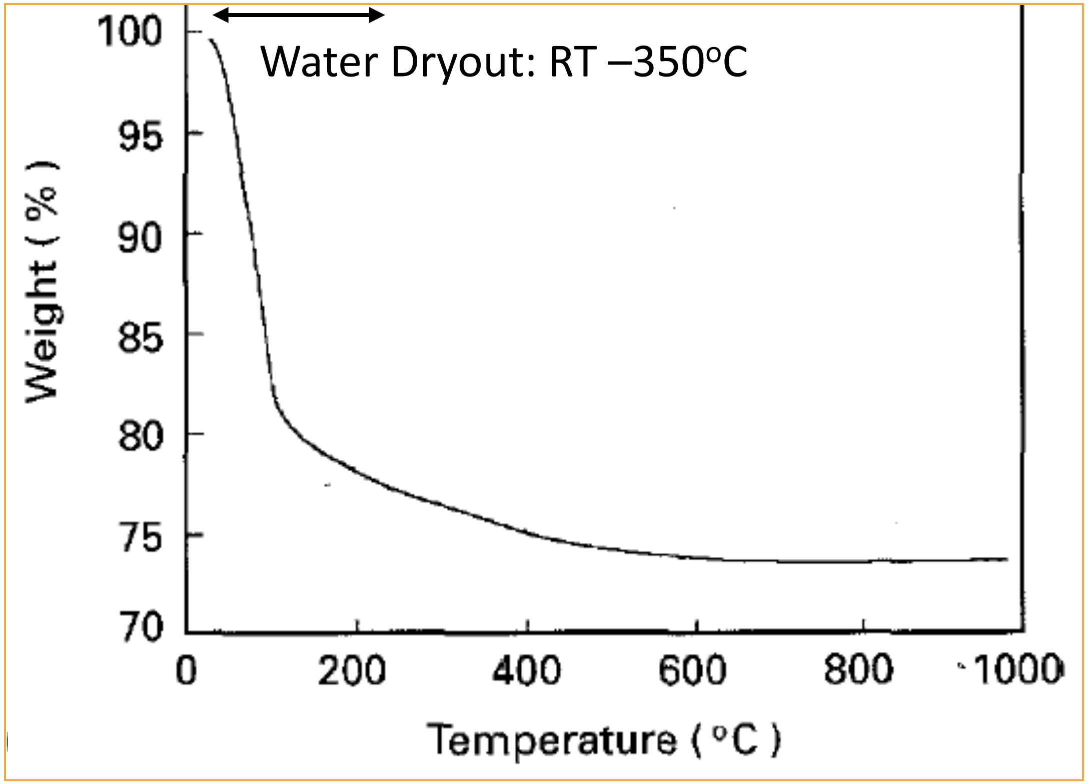
WATER REMOVAL

TGA Results*



Most of water evaporated below 250ºC leading to a 5% shrinkage*

This includes free water at surface/pores, adsorbed water, and chemically bound water*

*Rahier, H., B. VanMele, and J. Wastiels, Low-temperature synthesized aluminosilicate glasses: Part II: Rheological transformations during low-temperature cure and high-temperature properties of a model compound. Journal of Materials Science, 1996. **31** 80-85, (1996)

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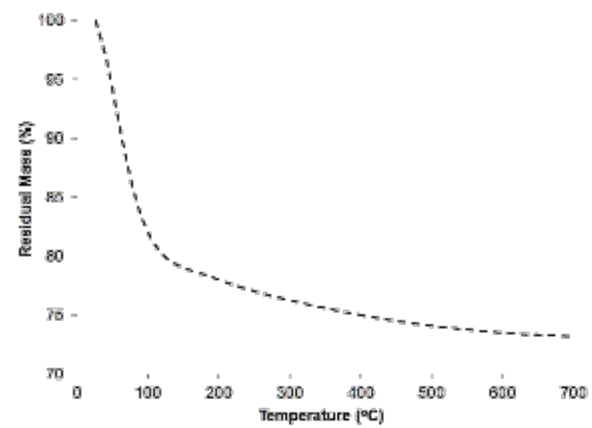
Dehydration Cracking

- Geopolymers undergo shrinkage upon heating due to water loss[1]
  - RT to 100°C: Dehydration of physically bonded (free) water
  - 100 to 300°C: Dehydration of chemically bonded (interstitial) water
  - >300°C: Dehydroxylation of OH groups
- This is described by the Young and La Place Equation: $\Delta P = (2\gamma \cos \phi) / r$
- Dehydration shrinkage causes cracking of monolithic geopolymer
- Reinforcing or filler phases can be utilized to maintain structural integrity during shrinkage by crack-bridging and offering pathways for more graceful dehydration



[1]J. Davidovits, *Geopolymer Chemistry and Applications*, 2008.

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Reinforcements

- Chamotte/mullite particulates, granite, dolomite sediment
- Chopped fibers
  - Alumina (13 μm D x 100 μm long)
  - Basalt (50 μm D x ¼", ½" long)
  - Carbon (7 μm D x 60 or 100 μm long)
- Alumina platelets (D = 50 μm)
- Woven fabric
  - Carbon fiber
  - Nextel 610 alumina, 720 mullite + alumina, 550 mullite
  - Basalt weaves and felts
- Mullite single crystal fibers (Moscow)
- Polymeric chopped fibers – polyproplyene (½", 1", 2")
- Biological fibers
  - Corn husk fiber bundles - Cordgrass (Illinois)
  - Jute (China, India) - Abaca (Manila hemp) and Hemp
  - Fique (Colombia) - Malva (Amazon)
  - Cork - Curaua (Amaon)

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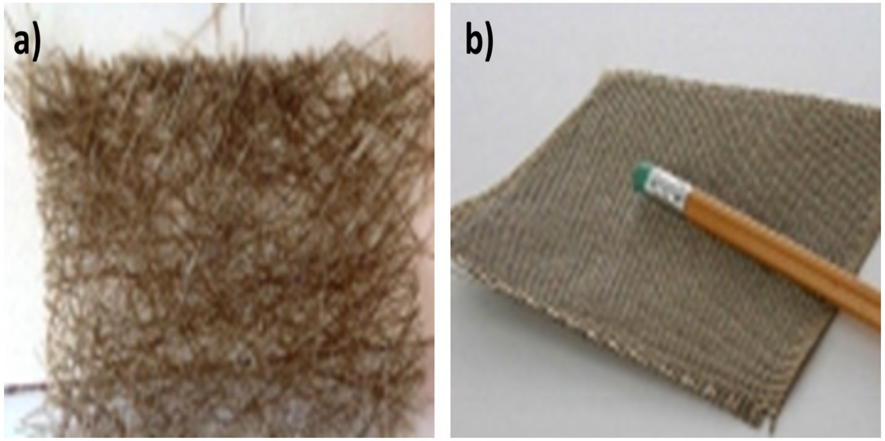
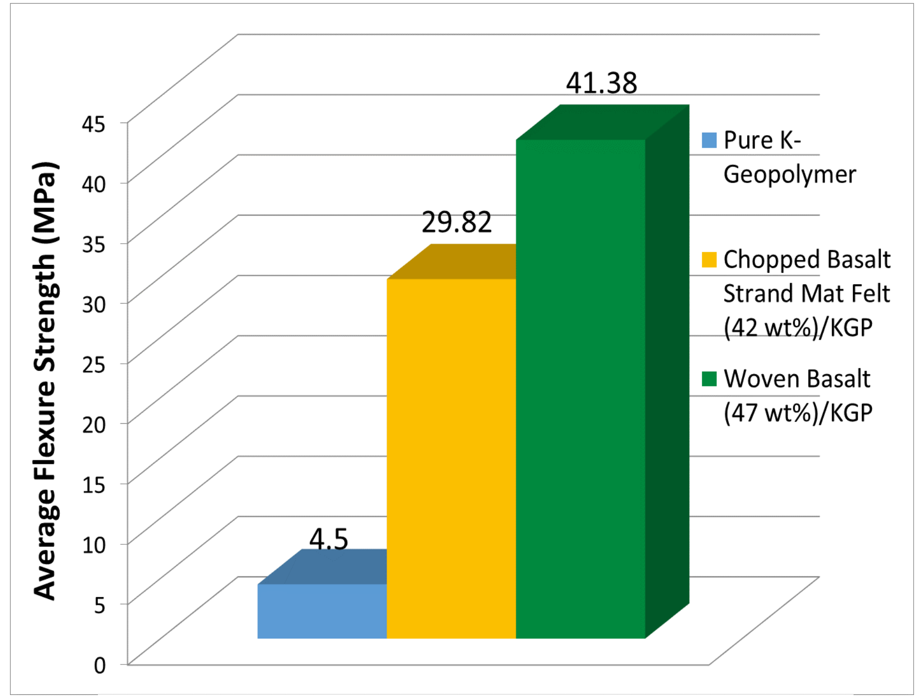
BENCHMARK STRUCTURAL PROPERTIES of GP CERAMIC COMPOSITES

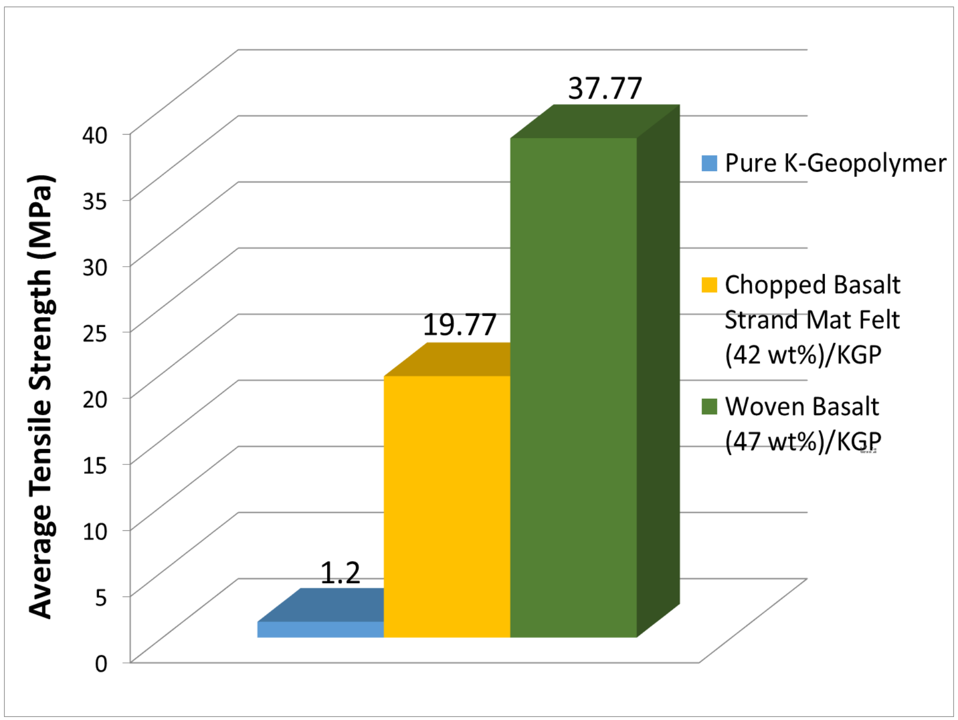
Material	Flexure strength (MPa)
KGP-chamotte particulate	2.1 → 15.3 for 50 wt%
Alumina platelets	2.0 → 20 (or 40 at HT)
KGP-chopped basalt ¼" ϕ =13 μm ½" ϕ=13 μm	1.7 → 19.5 for 10 wt% 2.2 → 27 for 10 wt%
Chopped C fiber 60 μm ϕ =7 μm	8.8 → 14.1 for 10 wt%
Chopped Al_2O_3 fiber CsGP ϕ = 3 μm	10 → 20 for 20 wt%
Nextel 610 weave	8.7 → 50 for 33 vol%
Nextel 720 weave	8.7 → 40 for 27 vol %
Basalt plain weave	4.5 → 41.4 for 30 vol%
Chopped strand mat	4.5 → 31.7 for 20 vol %

Material	Tensile strength (MPa)
Nextel 610 weave in KGP	2.5 → 205
Nextel 720 weave in KGP	2.5 → 125
Plain basalt weave in KGP	1.2 → 38
Nextel 610/monazite in alumina	0.25 → 117

Basalt chopped strand mat reinforced GP Composites







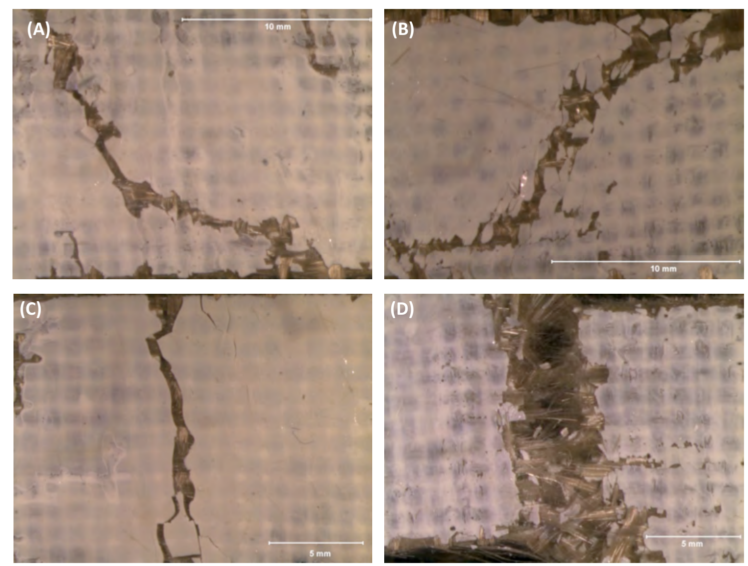


Table 5. Summary of flexure strengths of geopolymer composites (GPCs) with various inorganic reinforcements

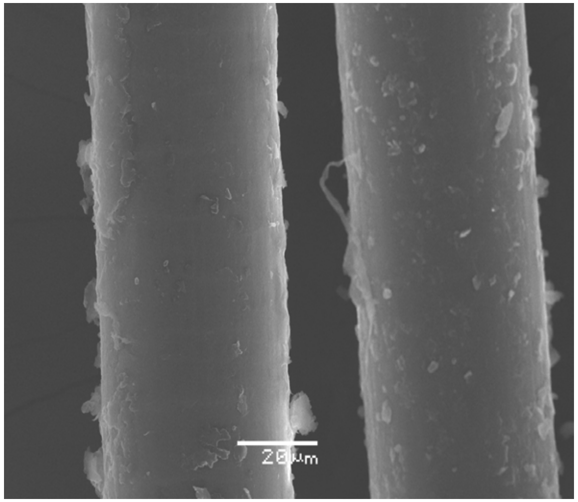
Reinforcement	Wt % Additions	Flexure strength (MPa)
Chamotte (25 μm)[16]	50	15.33
Dolomite (45 μm)[94]	20	15.92
Mica phlogopite platelets (50-100 μm)[88]	20	11.4
Granite powder (≤ 90 μm)[17]	55	10.3
Dicalcium phosphate (DCP)[91]	15	9.8
Hydroxyapatite bone ash[91]	15	9.5
Alumina platelet grinding media (50 μm)[97,98]	70	20 (RT) to 40 (at 1200°C)
Alumina chopped Saffil® fibers (ϕ = 3 μm)[99]	20	20
Carbon chopped fibers (60 μm x 7 μm ϕ)[49-51]	20	22.2
Carbon chopped fibers (100 μm x 7 μm ϕ)[49-51]	20	29.9
Basalt chopped fibers (1/4”)[14,15]	10	19.5
Basalt chopped fibers (1/2”)[14,15]	10	27
Graphene nanoplatelets	3	12
Basalt felt[18]	10	22.2
Fiberglass felt[18]	10	5.6
Basalt 4” chopped strand mat[19]	20	31
Basalt fiber weave[19]	30	41
E-glass Leno weave[89]	25	25.6
Carbon unidirectional fiber[53,54]	20	269
Nextel 610 alumina (8 satin weave)[36]	50	45.8
Nextel 720 (mullite +15 vol % alumina)[36]	50	46

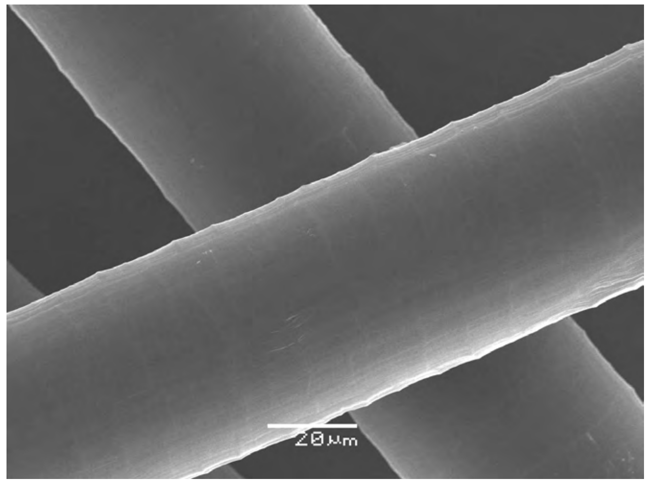
Synthetic and Biological Fiber Reinforcements

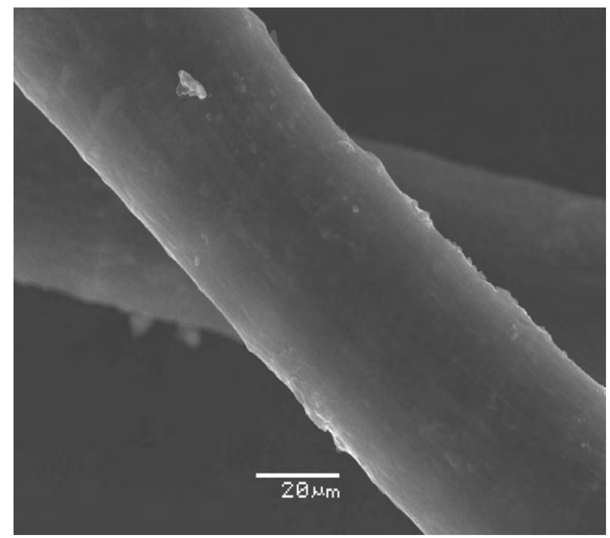
<u>Material</u>

- NaGP-polypropylene
- ½”, 1”, 2”
- Corn husk fibers
- China jute weave
- Colombian fique
- Brazilian malva
- Brazilian curaua
- Abaca (Manila hemp)

SEM of Polyproplyene Fibers







Innegrity Polypropylene (PP)

(a) 0.5" fibers with diameter 47±1 µm

(b) 1" fibers with diameter 50±1.5 µm

(c) 2" fibers with diameter 48±1 µm.

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Optical micrograph of PP-geopolymer composite broken in 3-point flexure (side view)

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Optical micrograph of PP-geopolymer composite broken in 3-point flexure (top-side, hairy view)

Optical micrograph of PP-geopolymer composite broken in 3-point flexure (bottom view)

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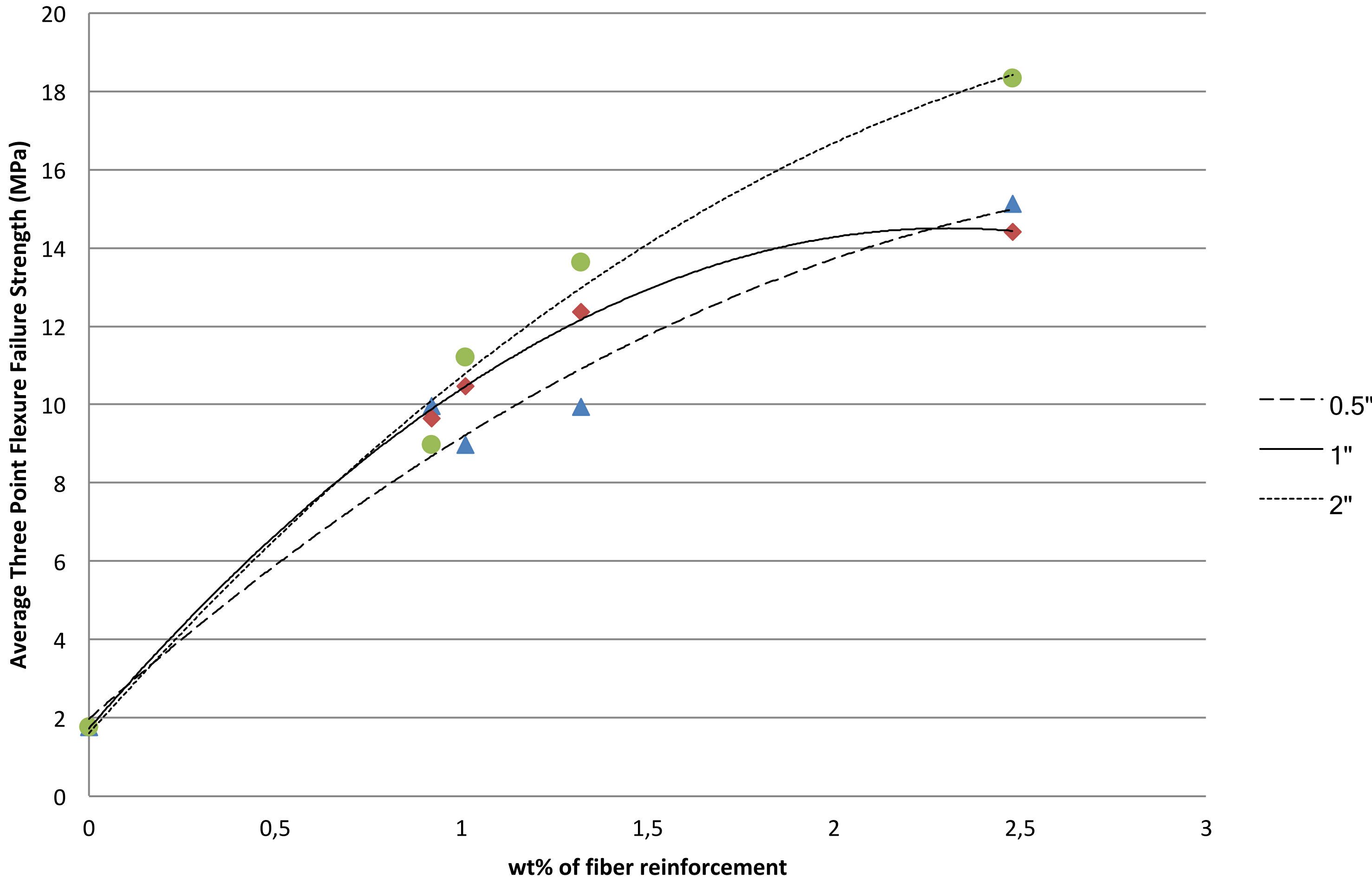
3-pt flexure of PP fiber - geopolymer composites



Average maximum failure stress of sample as a function of fiber length and composition

NaGP/CHF Composite Panels

- The uncured panel was then covered with a Delrin top piece and placed in a hydraulic press at 50 psi for 24 hours to compress the panel and remove excess geopolymer matrix material
- Following the cold press process, the panel mold was sealed and placing in the oven at 50^{o}C for 24 hours for final curing
- Cured panels were cut into mechanical testing samples using a wet tile saw with high speed diamond abrasive cutting wheel
  - Flexural test specimens: 10 mm x 60 mm
  - Straight-sided tensile test specimens: 25 mm x 150 mm

	M_f	Fiber Orientation	Wetted/Unwetted
Panel 1	*13.28%*	*Quasi-aligned*	*Wetted*
Panel 2	*12.99%*	*Quasi-aligned*	*Unwetted*
Panel 3	*14.45%*	*Random*	*Wetted*
Panel 4	*12.01%*	*Random*	*Unwetted*

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In the Amazon (Manaus, Brazil)...........

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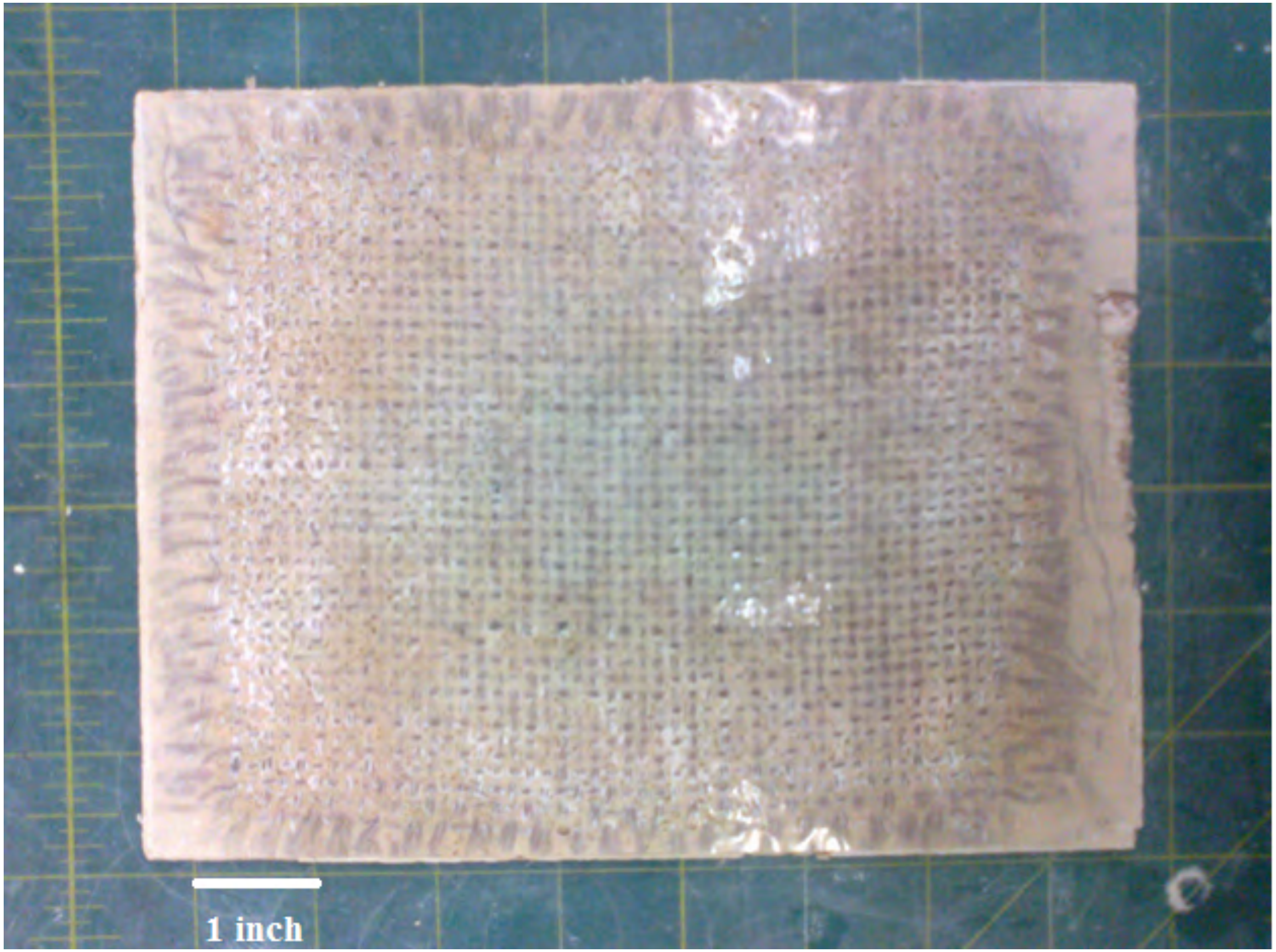

As-received jute reinforced sodium geopolymer plate

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Plate impact testing apparatus

Plate impact testing

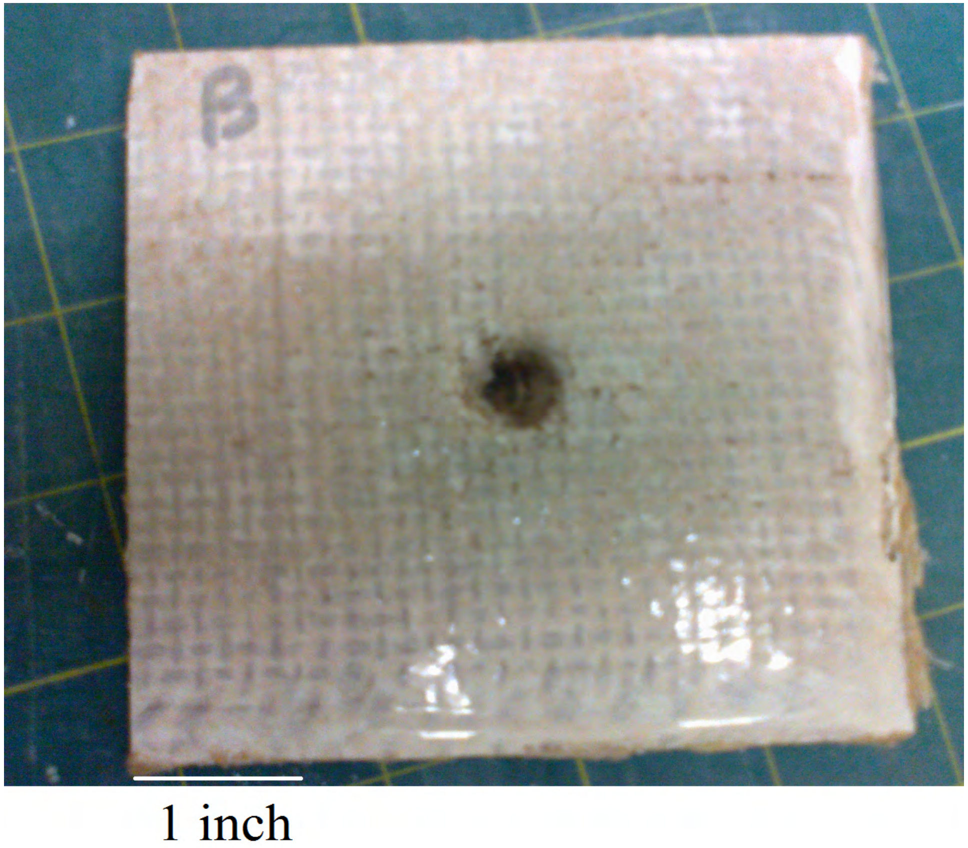

Figure shows 4" x 4" jute plates(left) after plate impact testing

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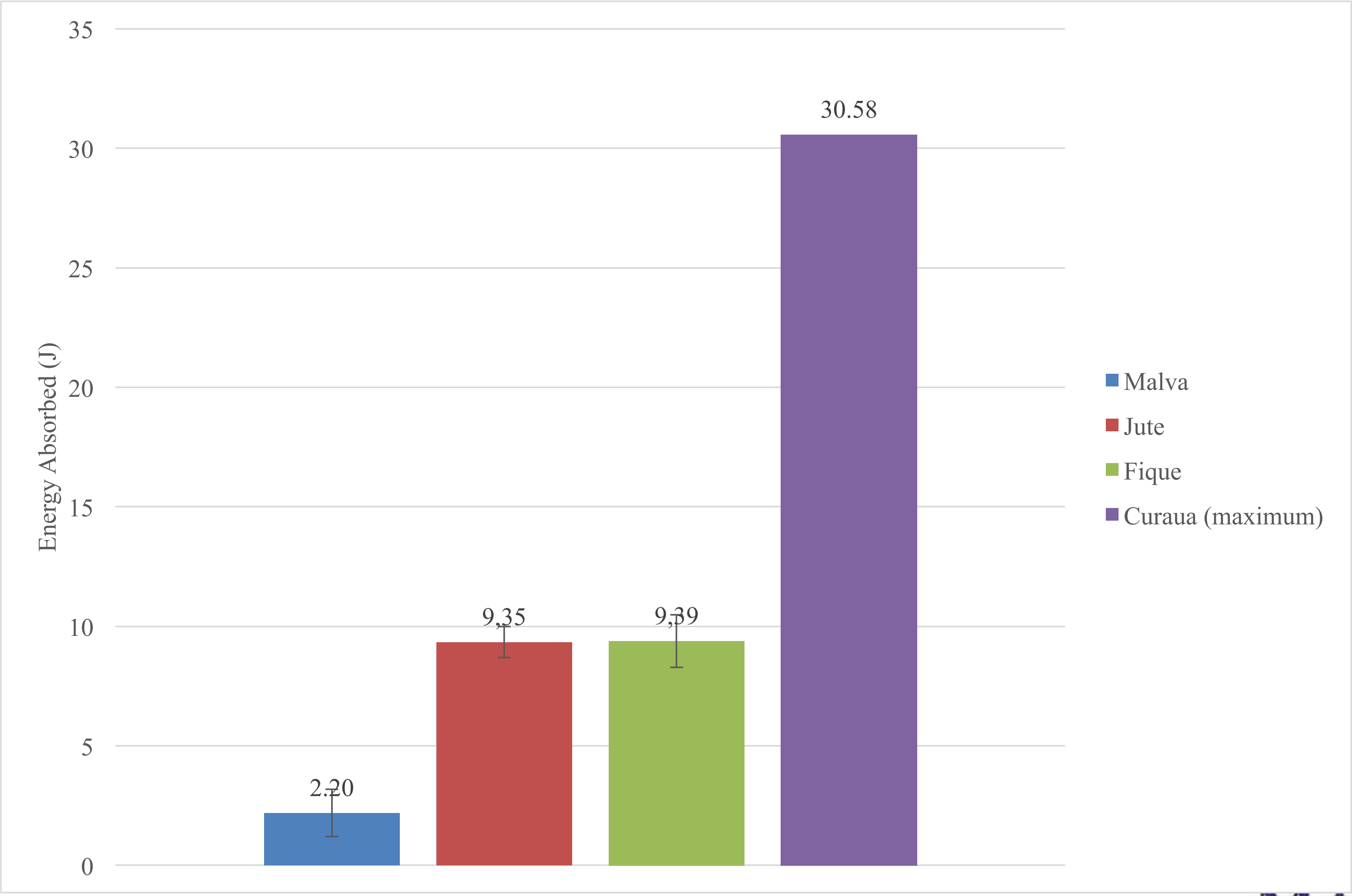
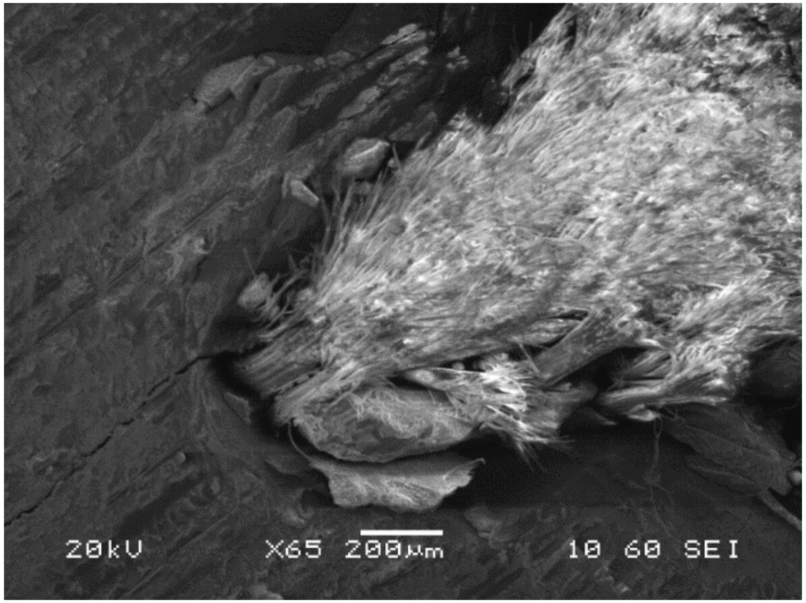
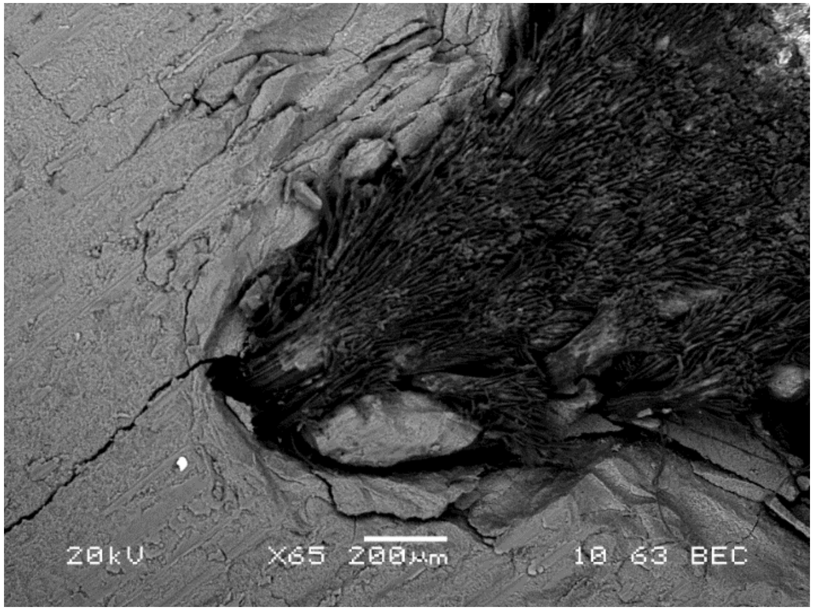
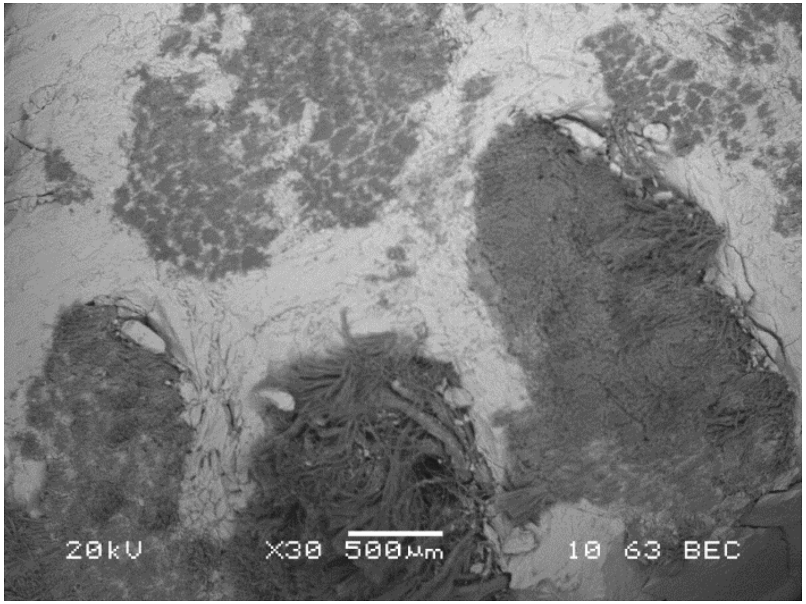
Impact tests

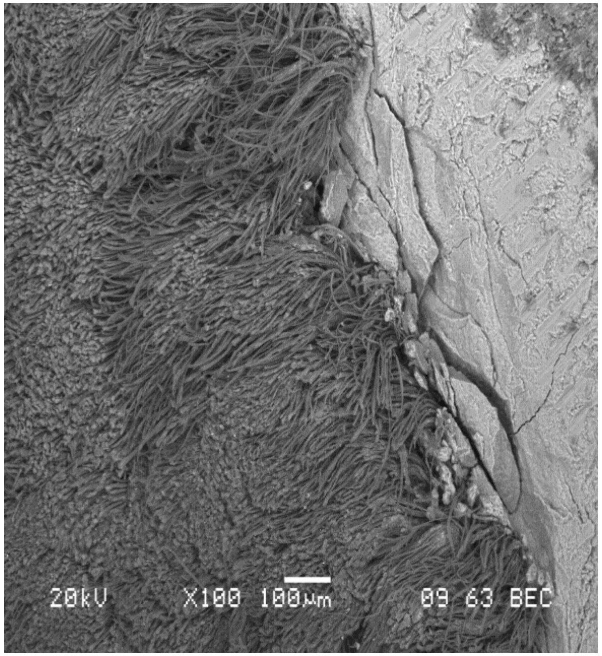


SEM of Curaua Fiber-reinforced Geopolymer









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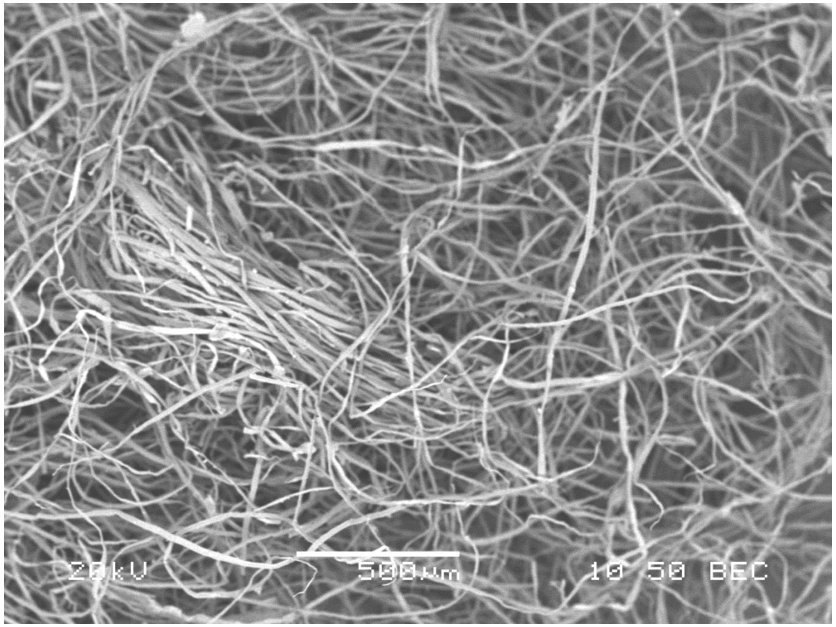
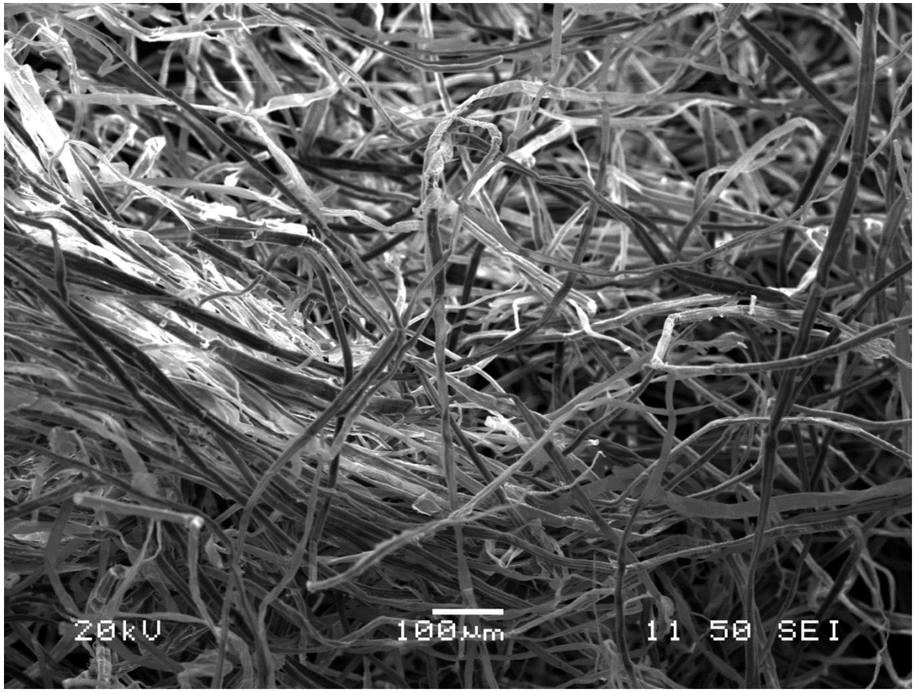
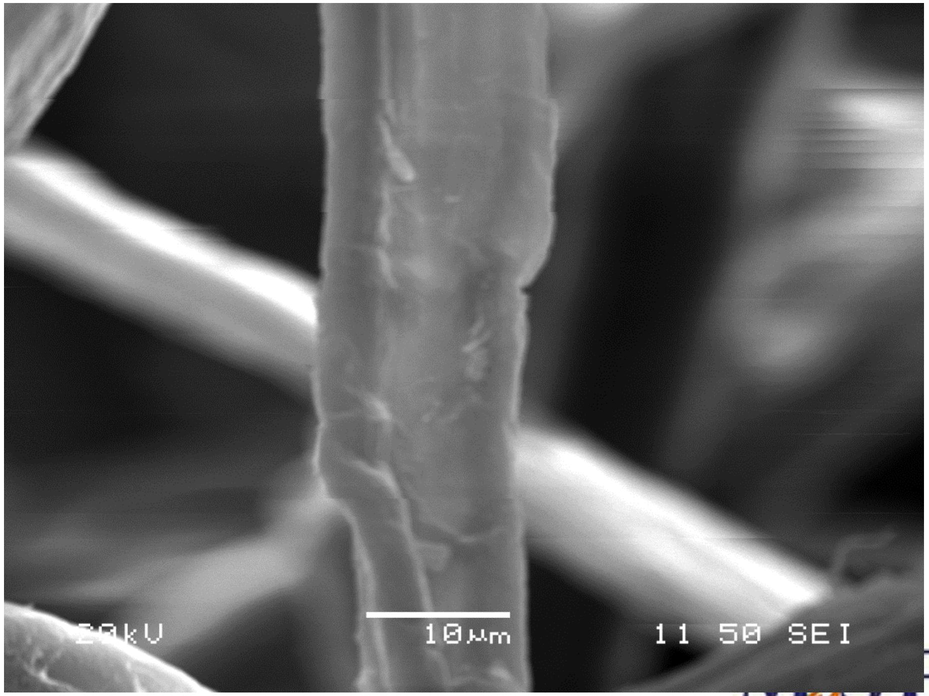
Synthetic and Biological Fiber Reinforcements

Material	Flexure Strength (MPa)		
NaGP-polypropylene	1.8	→	18.3 for 2.5 wt% (2”),
½”, 1”, 2”		→	15 MPa (1”), 14.5 (½”)
Corn husk fibers	2.5	→	7.6, pullout 15.3% strain
China jute weave	2.5	→	20.5 treated, 15 untreated
Colombian fique	2.5	→	11.4 I-D treated 7.1 2-D treated
Brazilian malva	2.5	→	18.3
Brazilian curaua	2.5	→	12.8
Abaca (Manila hemp)	7.9	→	52.3

"Abaca" fiber, also known as "Manila hemp," is grown in the Phillippines, Ecuador and Costa Rica
The extremely strong fibers are often used in twine and ropes, and can be pulped to make specialized paper







Amazonian Bamboo

Fast growing and high yield renewable resource
Readily available in Brazil
Governmental support
40-50 culms per clump
10-20 culms yearly
Maximum height in 4-6 months
Daily increment of 150-180mm
4 years to mature

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Brazil

Mineral reserves as source of **aluminosilicates**

Photo by Ruy A. Sá Ribeiro

Amazonian MK

Replacing Portland Cement

Photo by Ruy A. Sá Ribeiro

Mineral reserves and byproducts as source of aluminosilicates

Photo from brminer.com

Fibers, particles and strip of native or natural materials grown in the Amazon

Promote higher strength, rigidity, and workability, generating green high-performance materials

Photo from flickr.com/lozanoginel

Regional and local materials

in the production of geopolymeric composites

reduce environmental impacts and **raise their practicality**

Photo from flickr.com/nasfizo

Bamboo fibers

Low cost

High strength and biodegradability

Absorption of CO_2 and production of O_2 3 times more than other plants

Photo from flickr.com/harnisch

Strong Colombian bamboo Collaboration with
INPA (Instituto National de Pesquinsas de Amazonas)

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Experimental procedures

Chopped bamboo fibers

micro BF1A and short BF4W fibers

Experimental procedures

Mixed Alkali Regional **Metakaolin-Based Geopolymer**

1 = powder

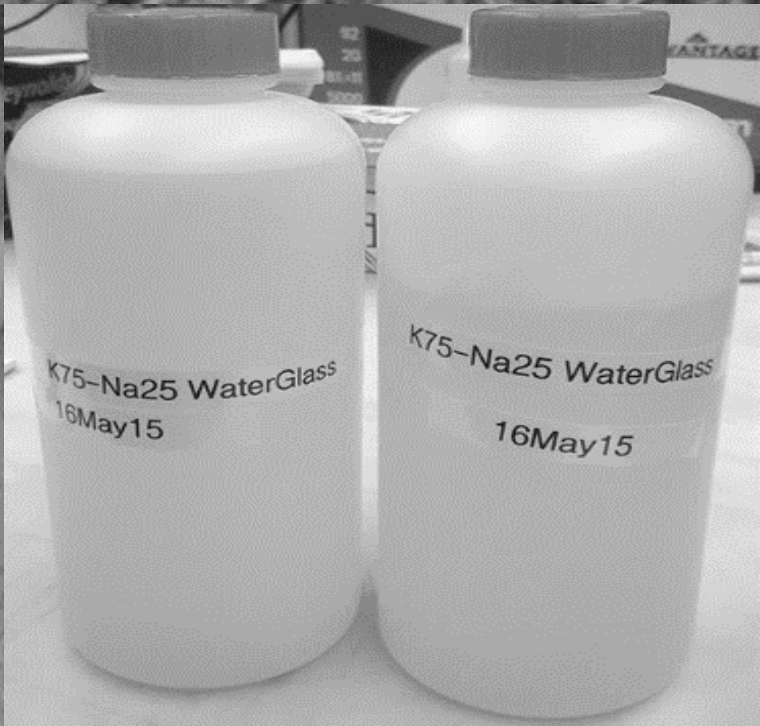
4 = fibers

GPC Compressive Strength

ASTM C1424-10

KNa-MKA / KNa-MKA-BF1A /

KNa-MKA-BF4A / KNa-MKA-BF4W



Chopped Guadua Angustifolia bamboo reinforcements

Figure 1. (A) Water-treated bamboo culms; (B) mechanically attained air-dried fiber bundles; (C) washed BFs reinforcement just before use

Panels and test samples of 6 “ x 8 “

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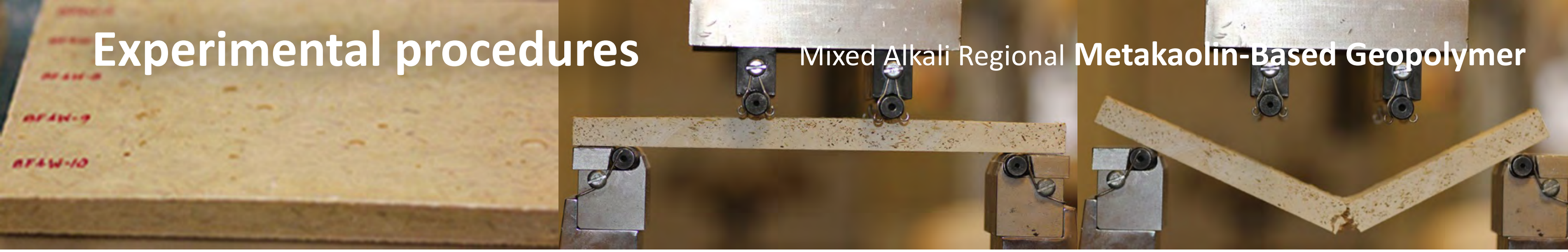
Experimental procedures

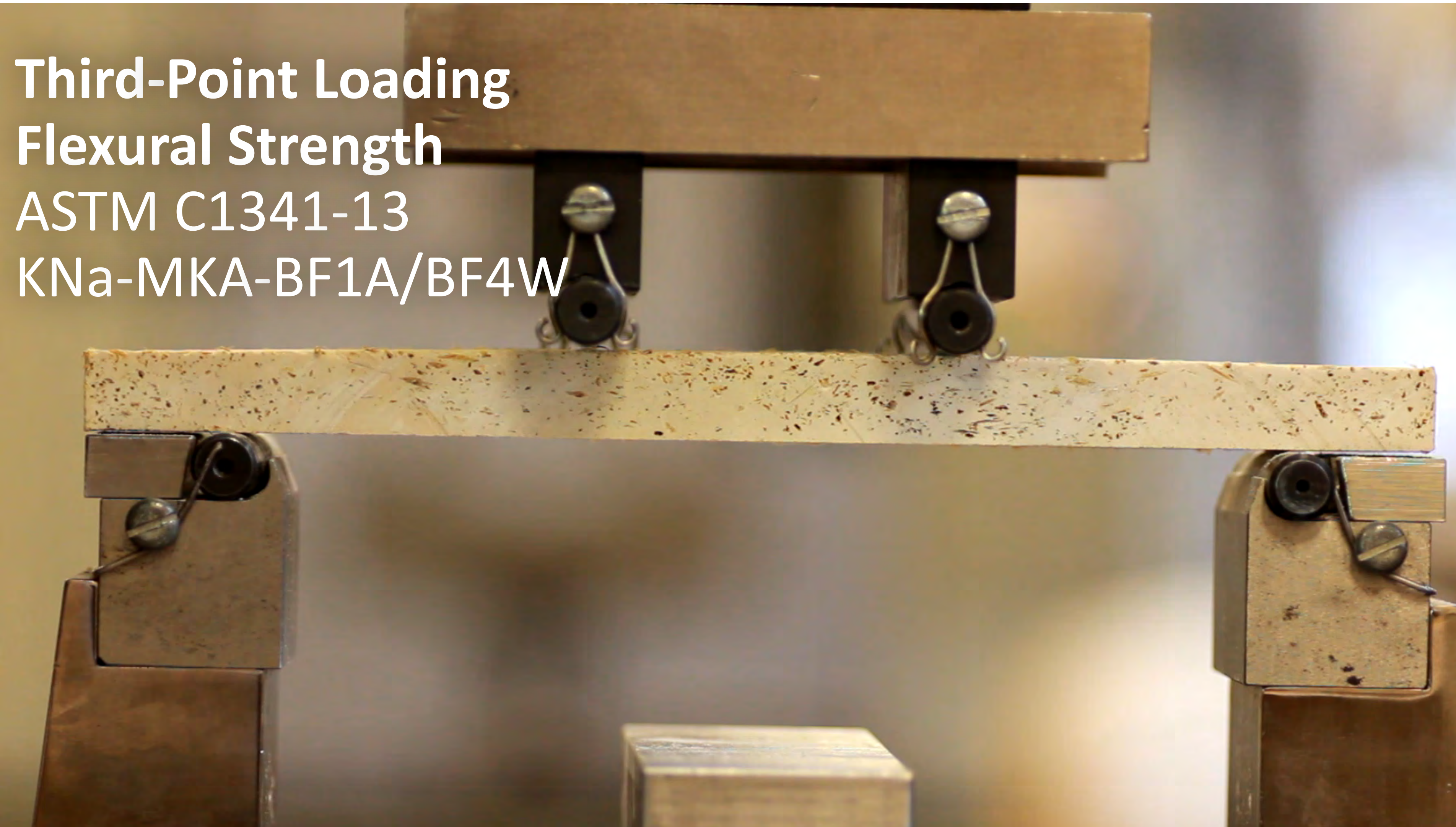
Mixed Alkali Regional **Metakaolin-Based Geopolymer**



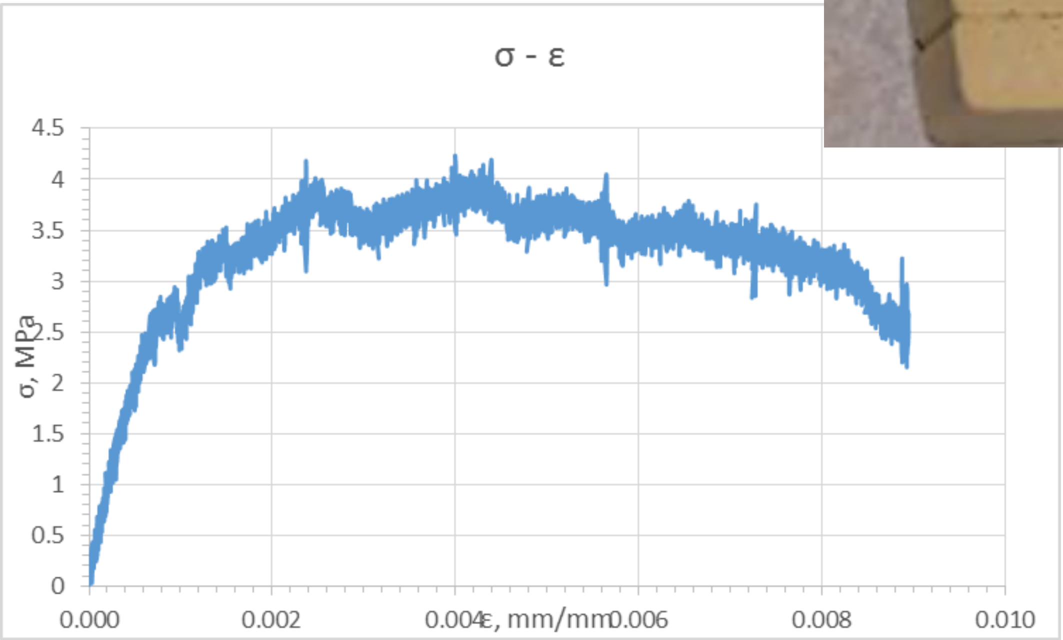
Third-Point Loading
Flexural Strength

ASTM C1341-13

KNa-MKA-BF1A/BF4W



Using natural Amazonian clay



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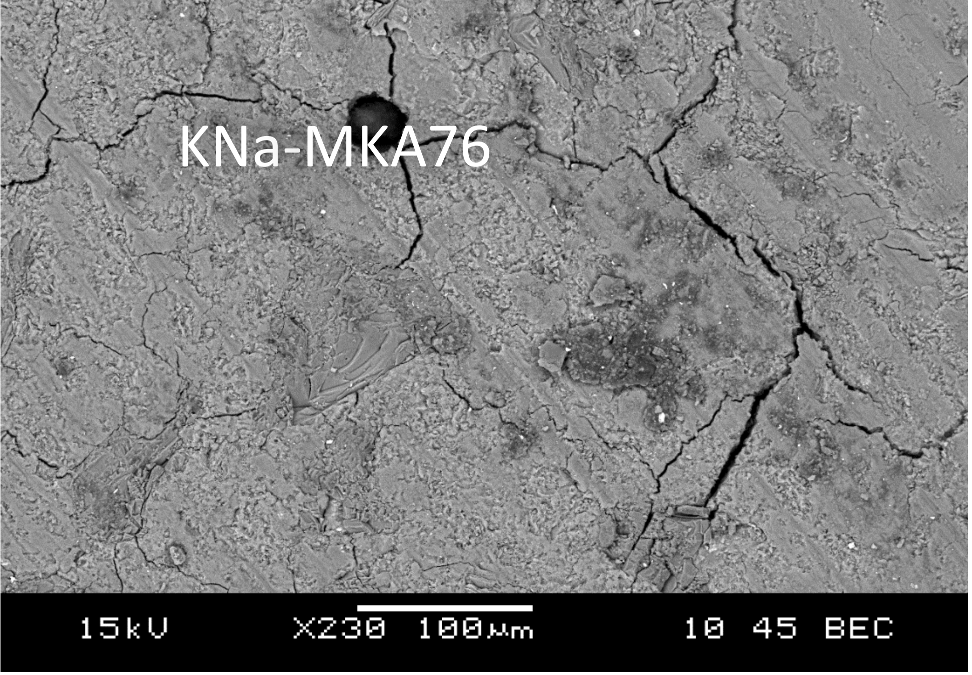
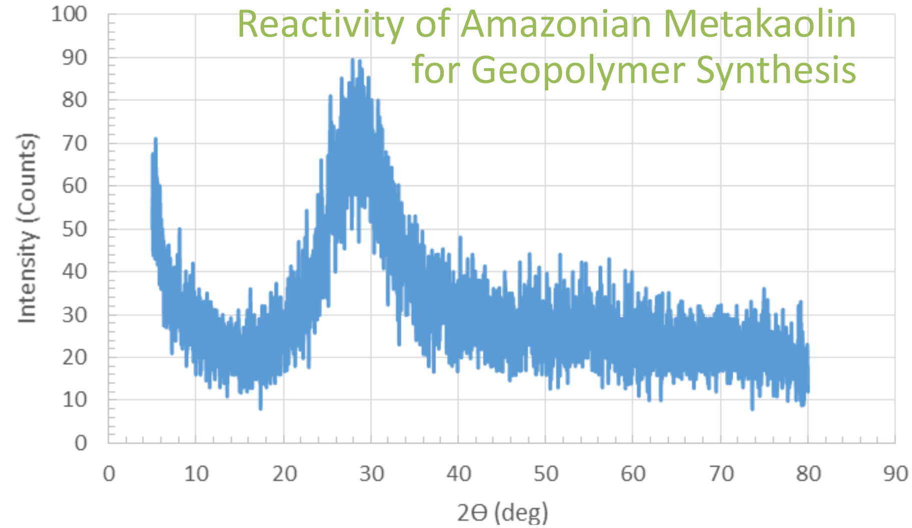
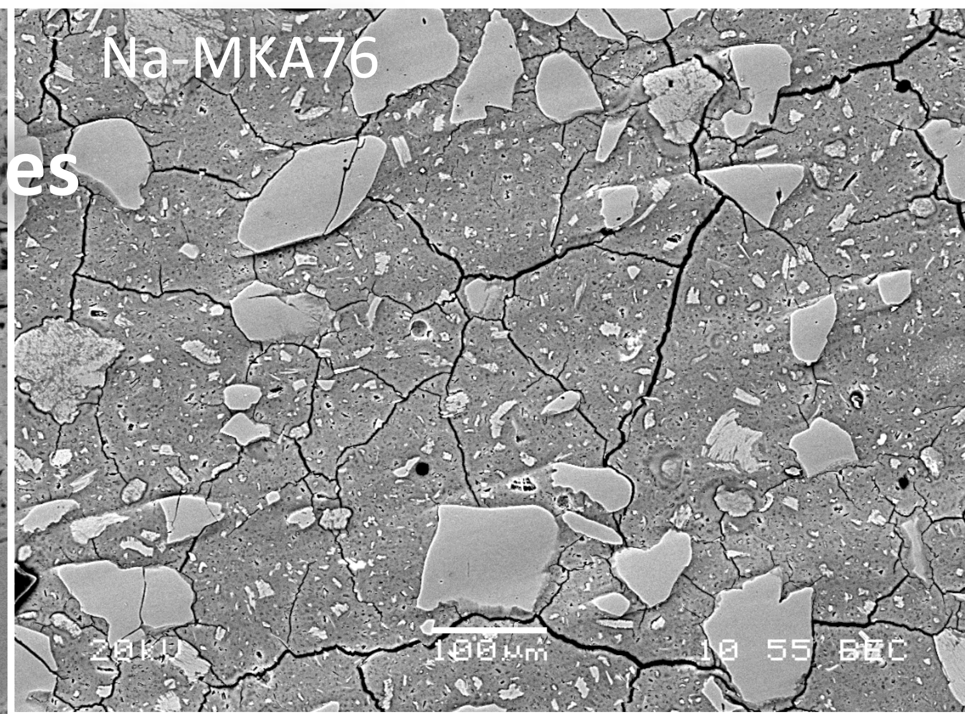
Experimental procedures

GPCs'
strengths tested in compression and flexural loading

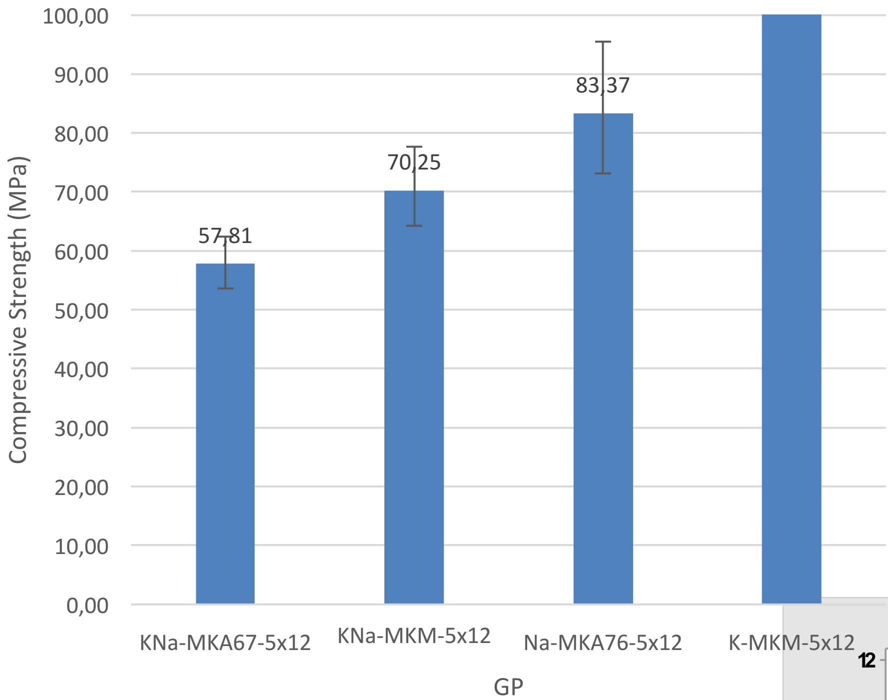
Reactivity of Amazonian Metakaolin for Geopolymer Synthesis



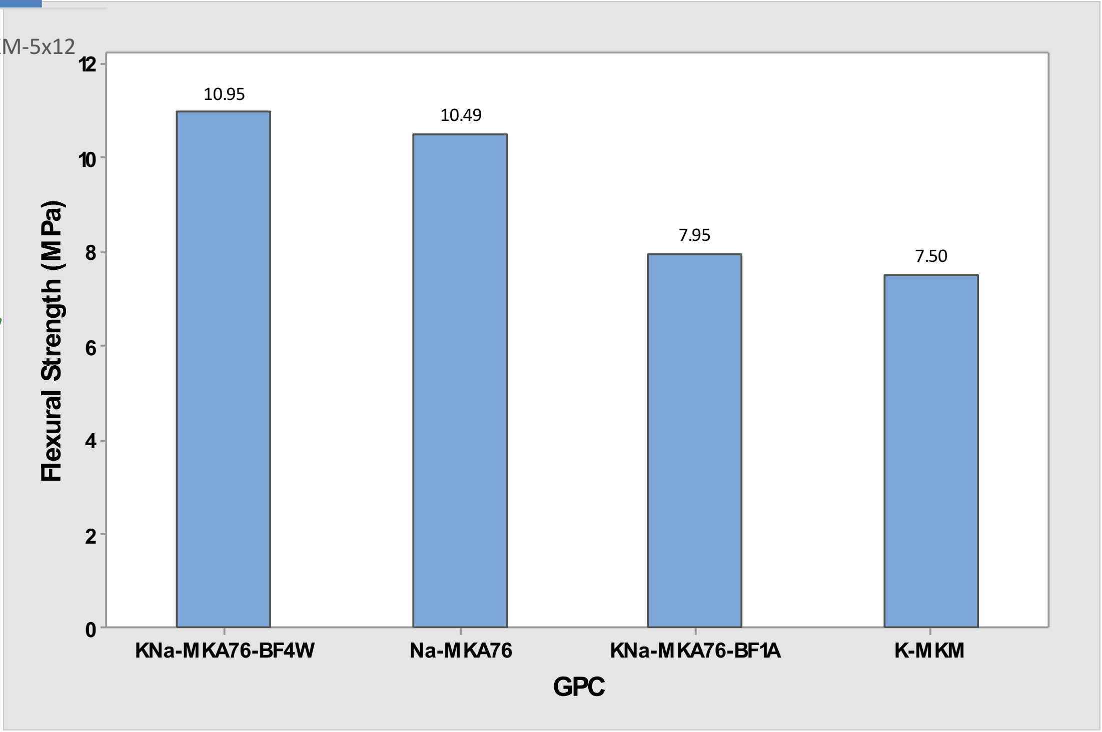


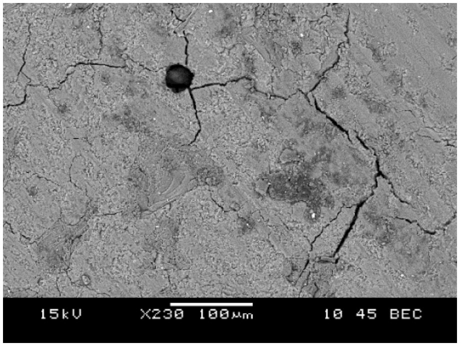


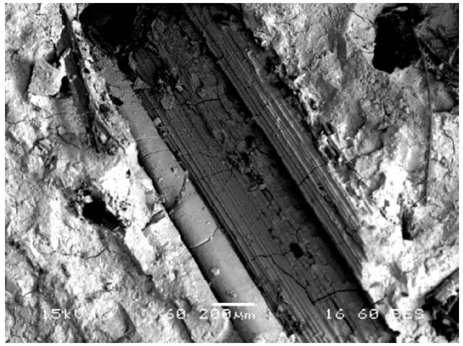
XRD and SEM techniques used to characterize the material



Test results demonstrate the great effect of **highly reactive MKA on GP strength**





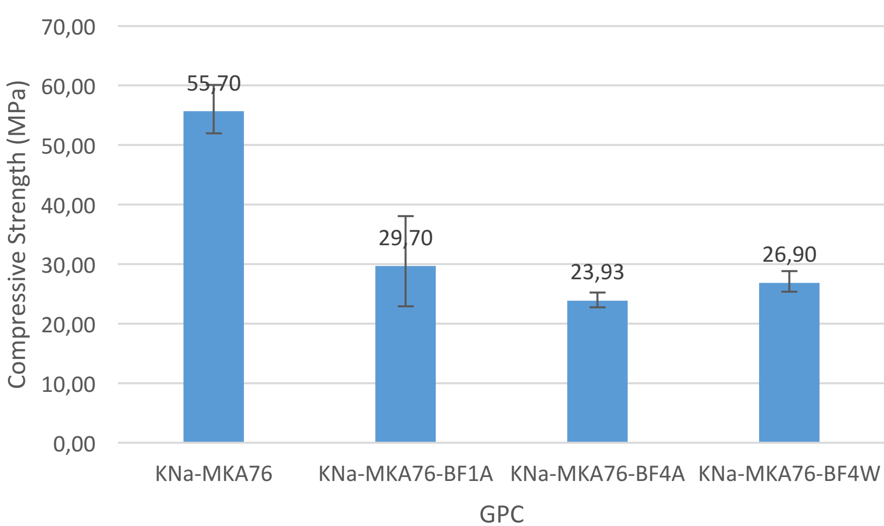


SEM

KNa-MKA GP

KNa-MKA-BF4W GPC

GPC	Modulus β Shape	Scale σ_o (MPa)	$\sigma_{c\text{-}avg}$ (MPa)	SD (MPa)	L95% (MPa)	U95% (MPa)
KNa-MKA76	11.590	58.20	55.70	5.83	51.98	60.12
KNa-MKA76-BF1A	3.303	33.11	29.70	9.90	22.91	38.08
KNa-MKA76-BF4A	15.38	24.76	23.93	1.91	22.75	25.24
KNa-MKA76-BF4W	12.010	28.07	26.90	2.72	25.37	28.84



Compressive Strength

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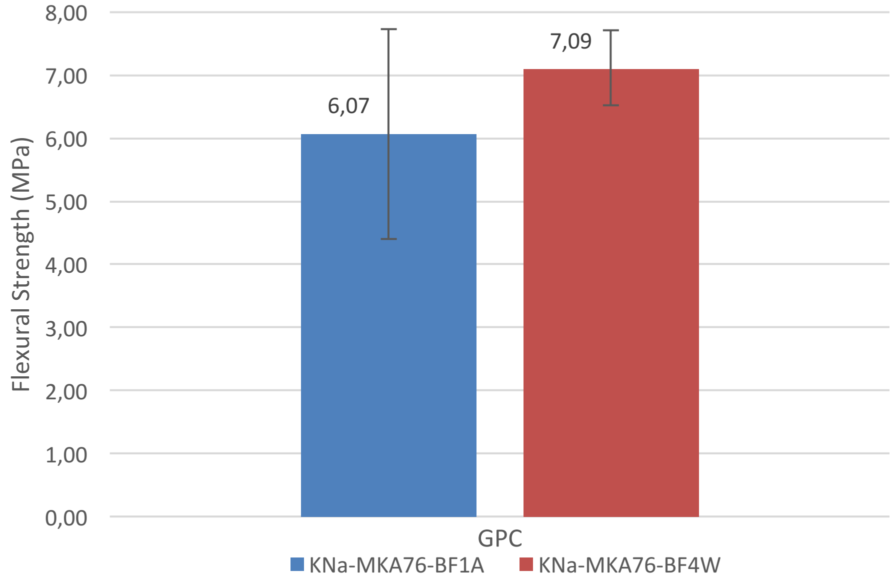
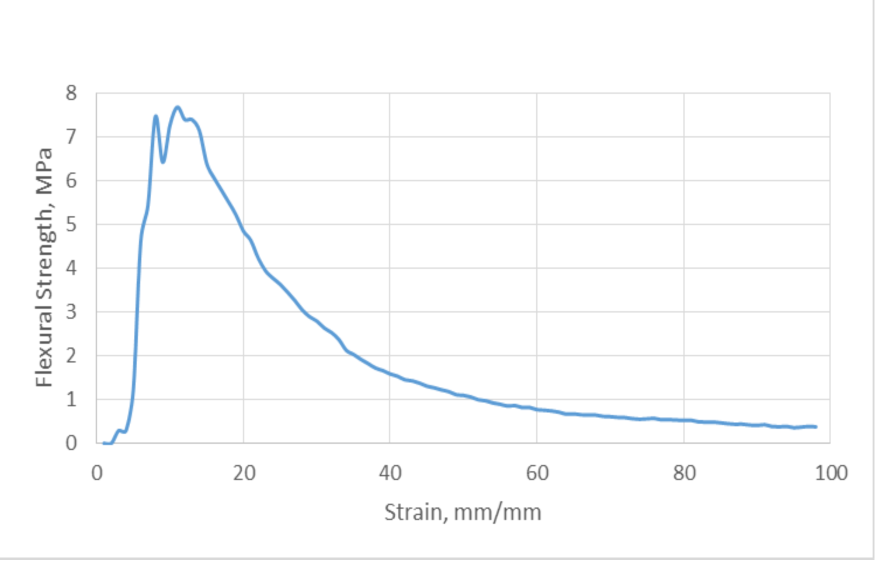
Flexure Strength

GPC	Modulus β Shape	Scale σ_o (MPa)	$\sigma_{f\text{-}avg}$ (MPa)	SD (MPa)	L95% (MPa)	U95% (MPa)
KNa-MKA76-BF1A	1.323	6.59	6.07	4.63	3.96	7.73
KNa-MKA76-BF4W	8.619	7.50	7.09	0.98	6.52	7.71





Summary of Amazonian Resources

1) SEM reviewed near to full reactive Amazonian MK Na-GP matrix; resulting in high 1-day compressive strength of **83.4 MPa** and flexural strength of **10.5 MPa**

2) XRD confirmed amorphous GP formation with a hump at **28° 2ϴ**.

3) Amazonian MK is a **viable precursor** for geopolymer synthesis

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Table 1. Comparison of Mechanical Properties of Portland Cements (OPC) with (GPC)

Property	OPC	GPC
Compressive strength (MPa)	60	100 - 120
Flexure Strength (MPa)	5-6	10-15
Density (g/cc)	2.7	1.4
Setting time (days)	28	1

Table 2: Flexure Strength of GPCs with Organic or Biological Reinforcements

Reinforcement	**% addition**	**Flexure strength (MPa)**
Polypropylene chopped fibers (1/2 ")	2.5 wt %	14.5
Polypropylene chopped fibers (1 ")	2.5 wt %	15
Polypropylene chopped fibers (2 ")	2.5 wt %	18.3
Cork particles	60 wt%	2.5 (0.75 % strain to failure)
Abaca (banana leaf random fibers)	8.0 wt%	52.3
Corn husk fibers	13 wt %	7.6 (7 % strain to failure)
Jute weave	30 wt %	20.5
Colombian fique / sisal (unidirectional)	50 wt %	11.4
Amazon**ian** malva (unidirectional)	5.5 wt %	31.55
Amazon**ian** curaua (unidirectional)	8.3 wt %	18.86
Chopped bamboo in Amazonian kaolinite	15 wt %	6-7

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Table 6. Flexure strength of GPCs with organic or biological reinforcements

Reinforcement	Wt % Additions	Flexure strength (MPa)
Polypropylene chopped fibers (1/2")[70]	2.5	14.5
Polypropylene chopped fibers (1")[70]	2.5	15
Polypropylene chopped fibers (2")[70]	2.5	18.3
Cork particles[82]	60	2.5 (0.75 % strain)
Abaca (banana leaf fibers or "Manila hemp")[93]	8	52
Corn husk fibers[75]	13	7.6 (7 % strain)
Rice husk stems in rice husk silica-based GP[80]	7	12.4
Jute weave[78]	30	20.5
Colombian *fique*/*sisal* (unidirectional)[79]	50	11.4
Amazonian *malva* (unidirectional)[95]	5.5	31.55
Amazonian *curaua* (unidirectional)[96]	8.3	18.86
Amazonian *Guadua Angustifolia chopped bamboo* dispersed in Amazonian clay-based geopolymer[84-87]	20	4

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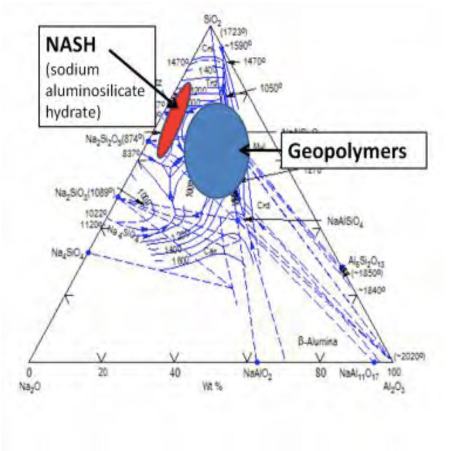
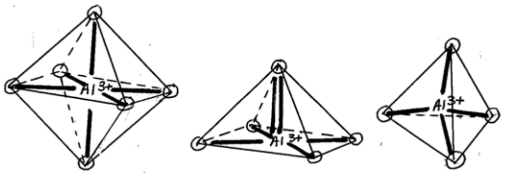
SUMMARY of GP COMPOSITES

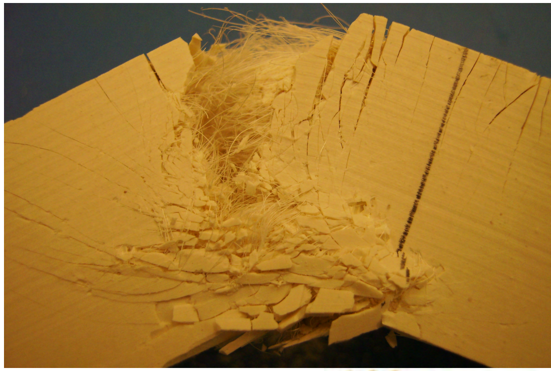


- Alkali activated cements are not the same as geopolymers

- A key feature of geopolymers is their formation by dissolution of the aluminosilicate source to form AlO_4^- tetrahedra which undergo polycondensation with SiO_4 tetrahedra to precipitate out as an inorganic aluminosilicates



- Strong geopolymer composites can be made using ceramic, synthetic or biological reinforcements



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