International Conference on Nanotechnology for Renewable Materials

Synergistic Reinforcement of Polyamide 6 Using Cellulose Nanocrystals and Wood Fibers

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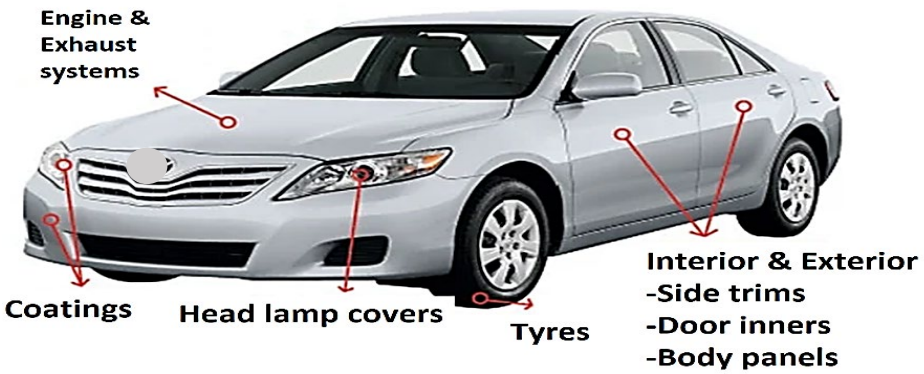
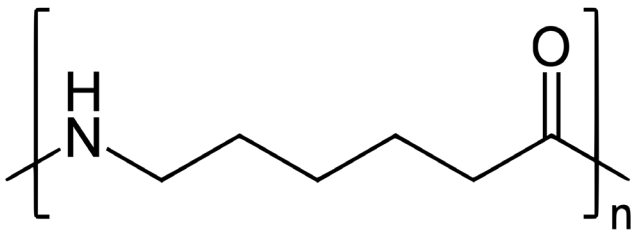
Key Drivers for Polymer Composite Usage

- High specific strength and modulus (2-4GPa)
- Fuel-saving (30%)
- Recyclability
- Design flexibility
- Corrosion resistance

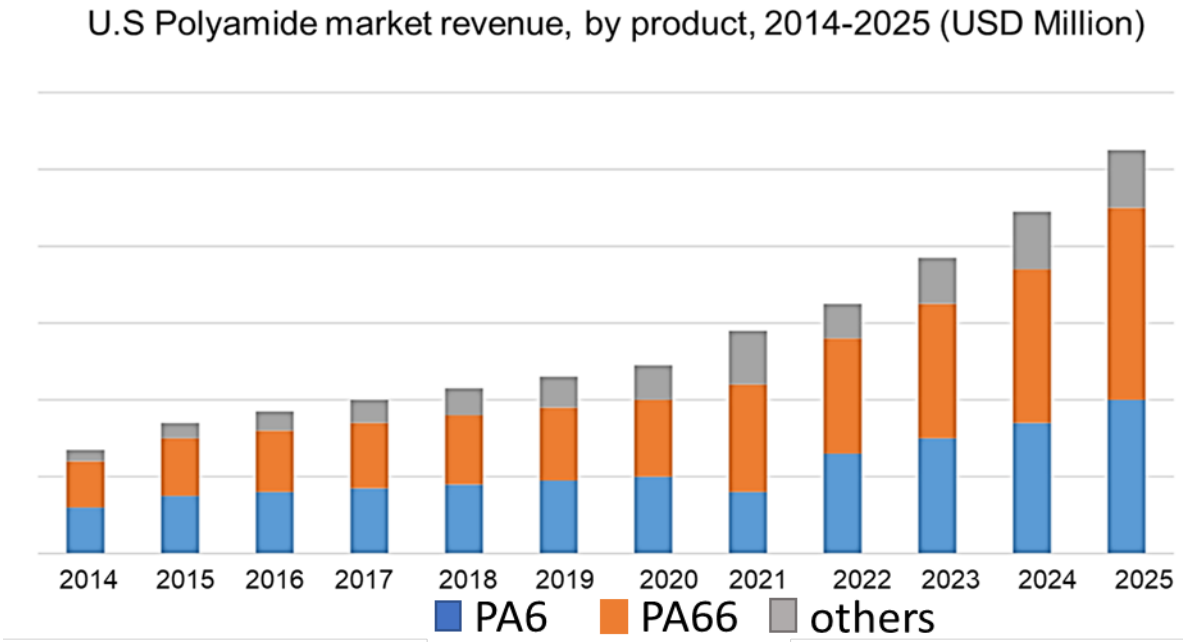


Matrix



Polyamide 6 (PA6)

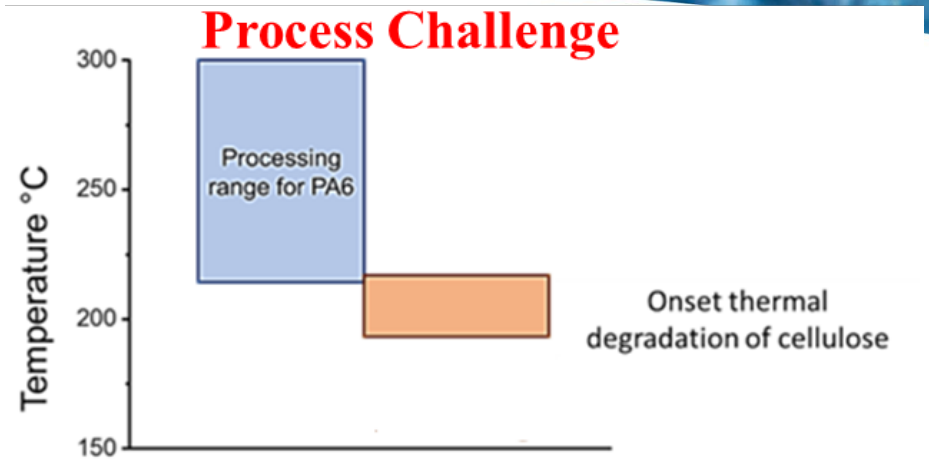
T_m=220°C



Jarvis V, et al. ACS Applied Polymer Materials. 2021.

Bio-based Fillers

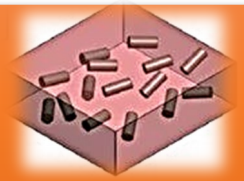
- Biodegradable
- Lightweight
- Cost-effective and accessible
- Sustainable



Cellulose nanomaterials (CNMs)

nm

Saw dust



µm

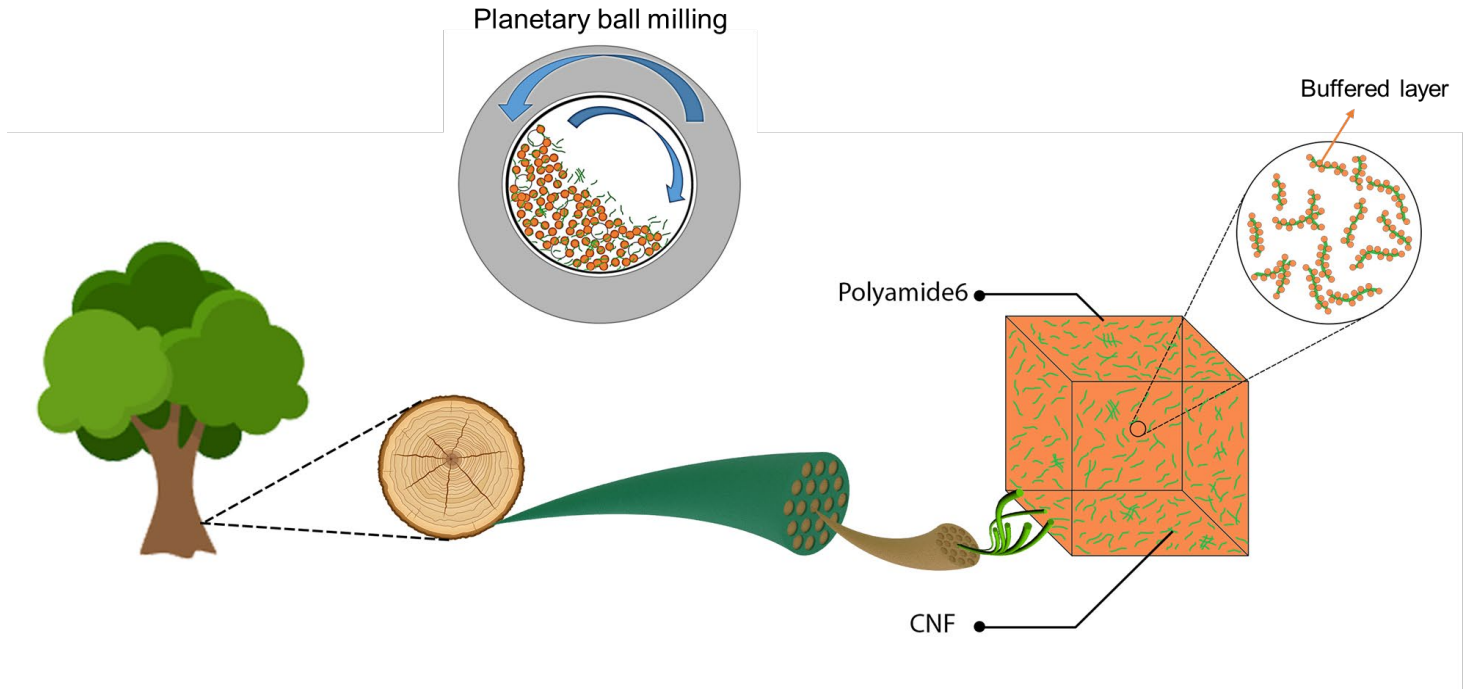
Pulp fibers (PFs)

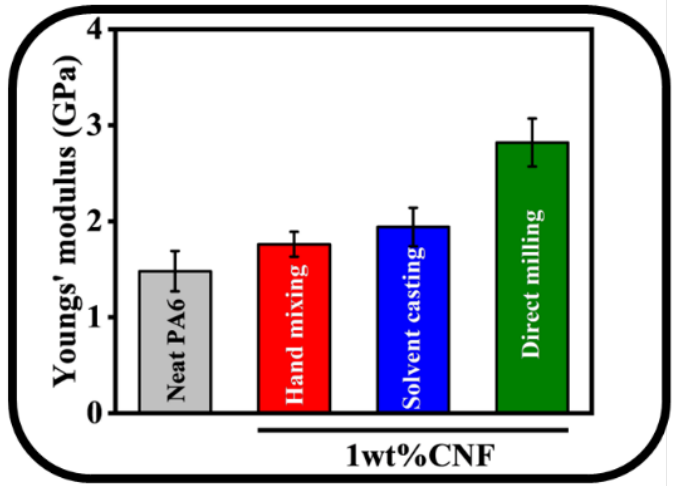
mm

Hypothesis

1- Employing premixing planetary ball milling to reduce the degradation of cellulose fibers prior to polymer processing.

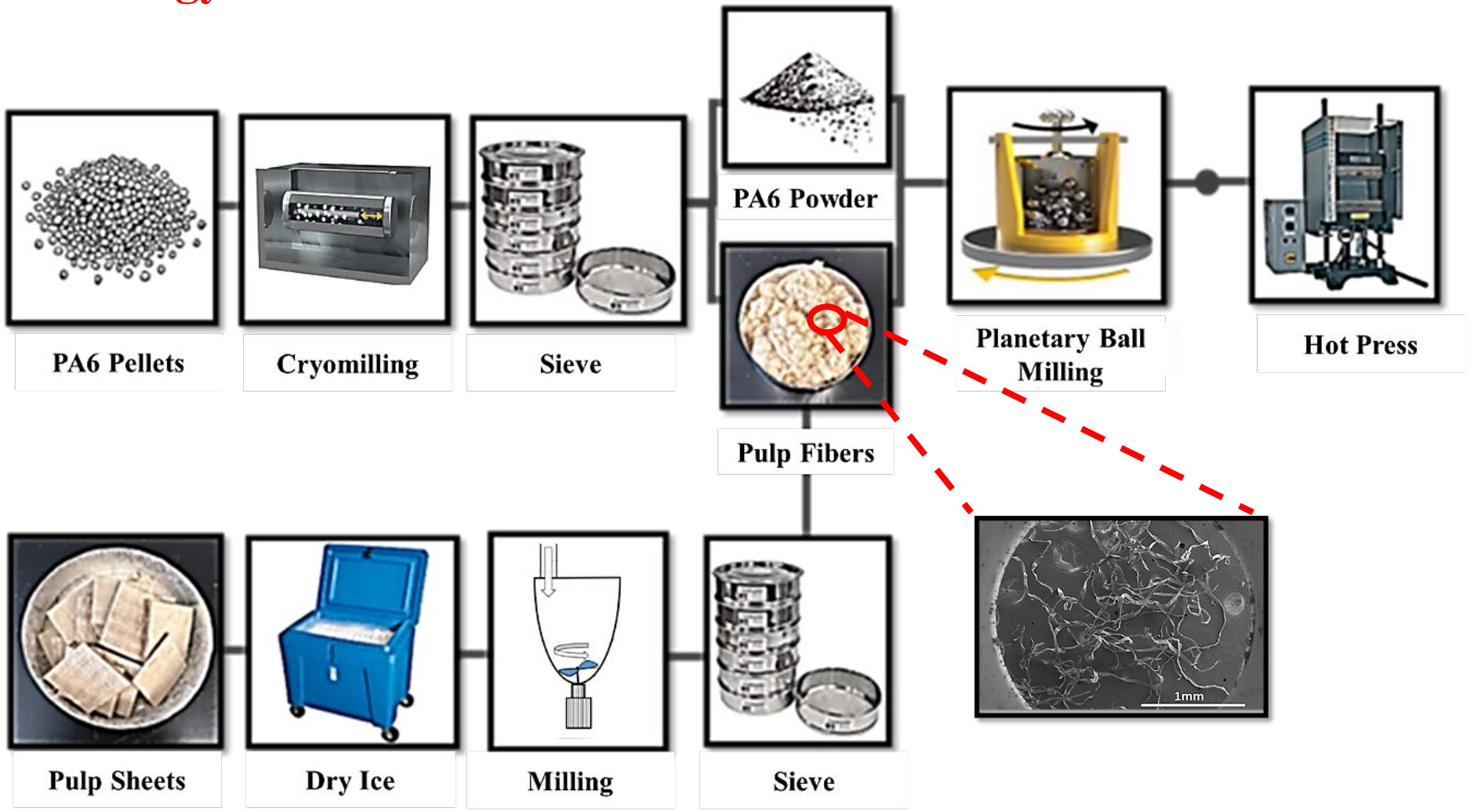
2- Micronizing the cellulose fibers improves thermal stability and dispersion quality.





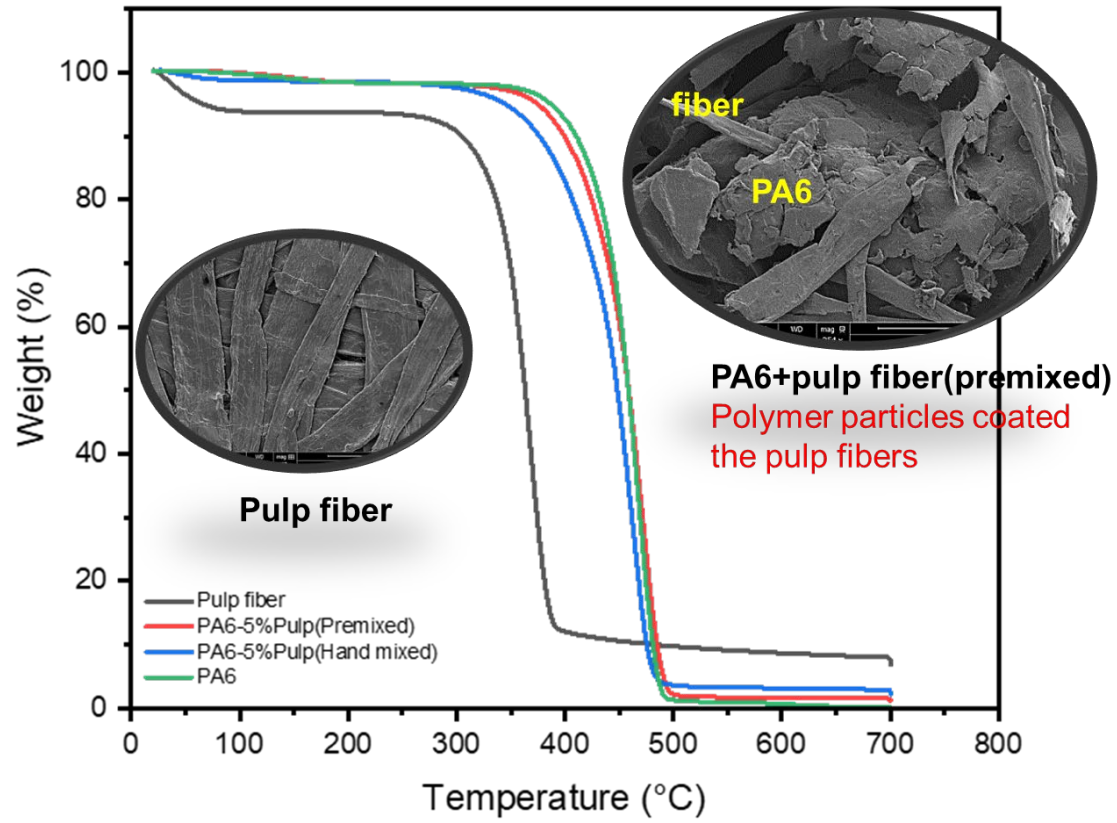
A, Jalaee,et al, Carbohydrate Polymers, Volume 314, 15 August 2023, 120932

Methodology



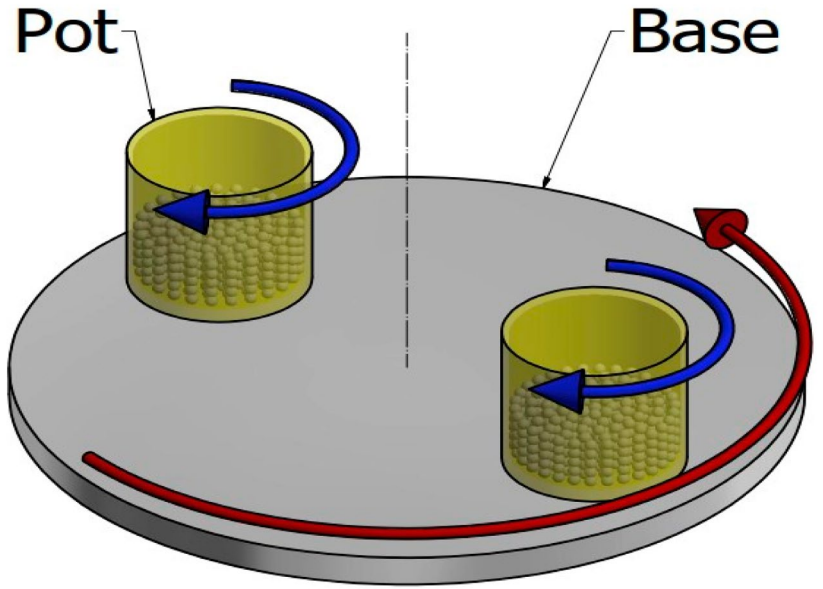
Effect of Premixing on Thermal Stability of Composite

Sample	Onset thermal degradation(°C)
PA6	387
PA6-5%pulp fiber (premixed)	370
PA6-5%pulp fiber (HM)	290
Pulp fiber	160



Effective Parameters in Planetary Ball Milling Process

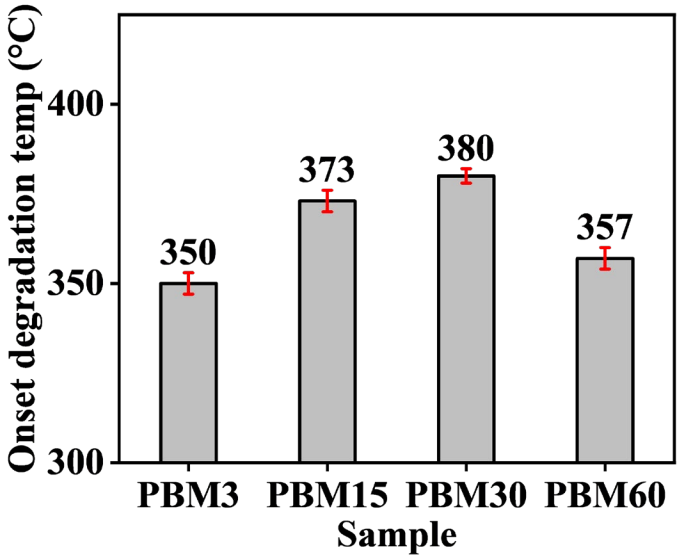
- Milling temperature
- Ball to powder weight ratio
- Diameter of ball milling
- Milling time

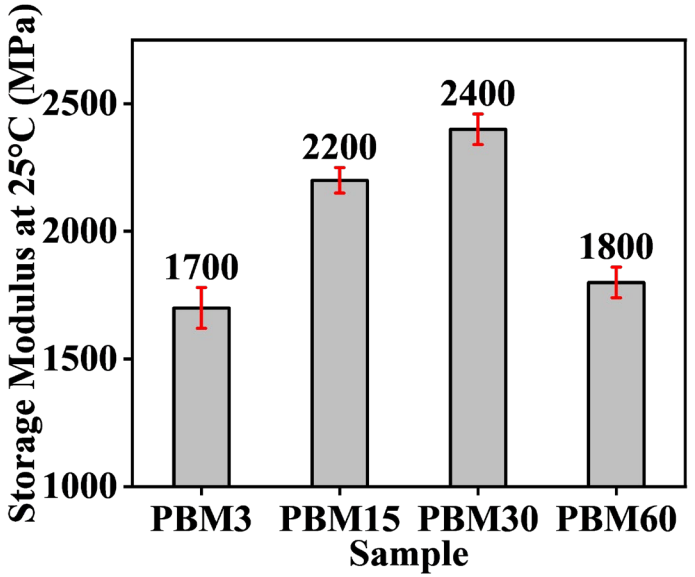


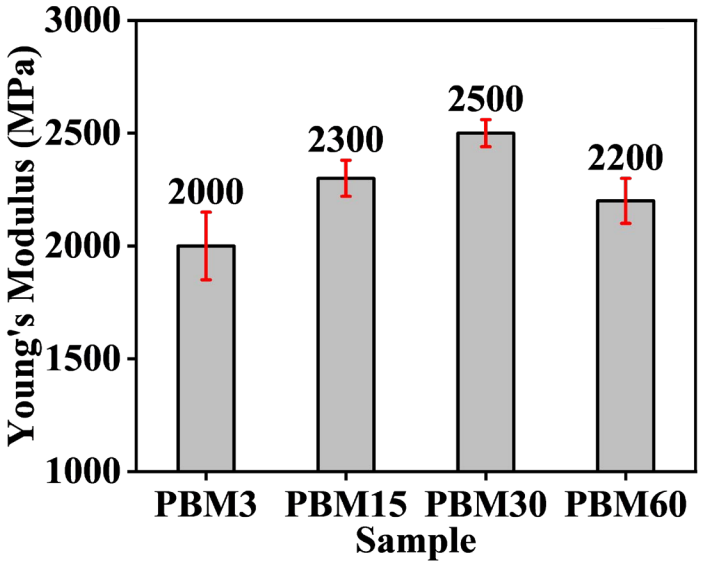
PA6- 5wt%Pulp fiber

Sample name	Milling time (min)
PBM3	3
PBM15	15
PBM30	30
PBM60	60

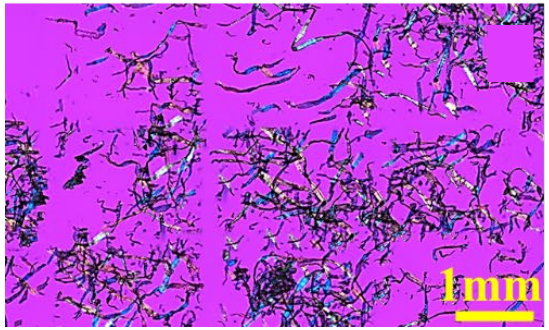
Thermomechanical Analysis



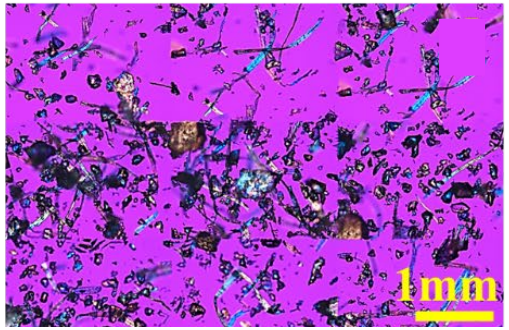




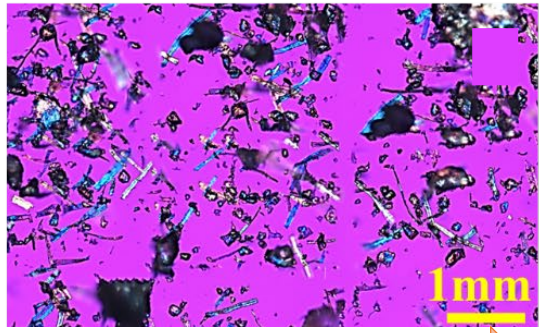
Neat pulp fibers before ball mill



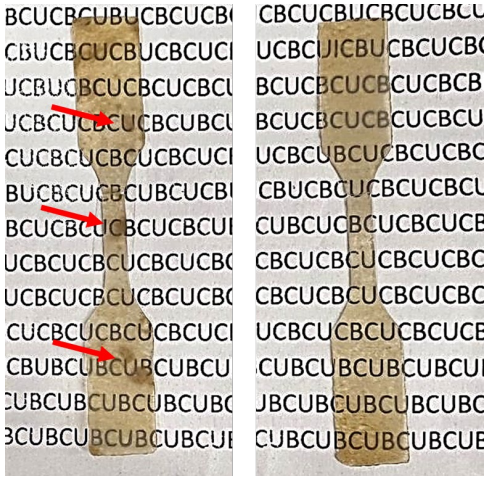
PBM15



PBM30



Aspect ratio decrease from 15 to 12



PBM15 PBM30

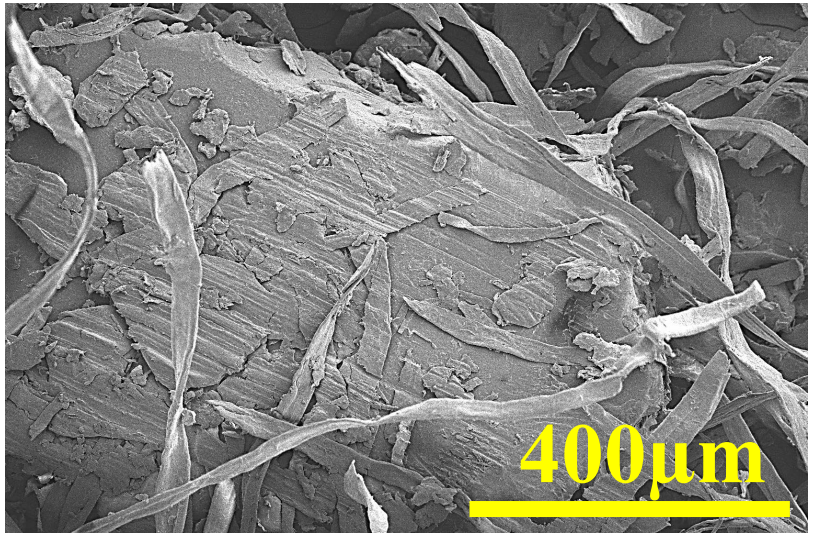
Composite of PA6 with Different Concentrations of Pulp Fibers

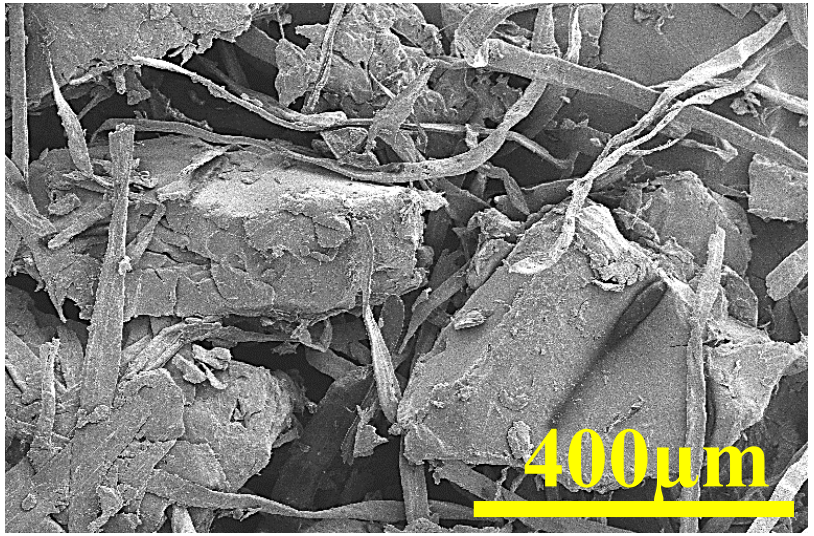
According to the optimization study on the milling time, 30 min is chosen as a premixing time.

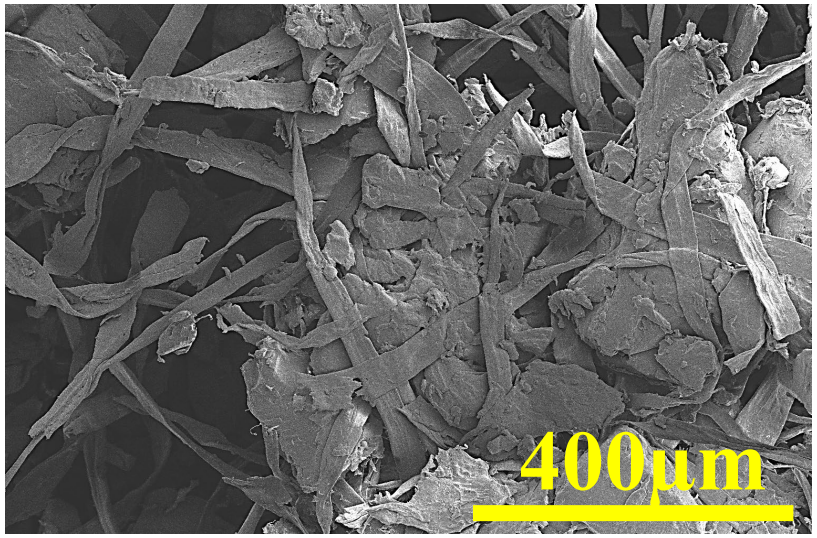
Sample name	PA6 (wt%)	Pulp fibers (wt%)
Neat PA6	100	0
PA6-5PF	95	5
PA6-10PF	90	10
PA6-20PF	80	20
PA6-30PF	70	30
PA6-40PF	It was not possible to make a film due to phase separation	

Morphology

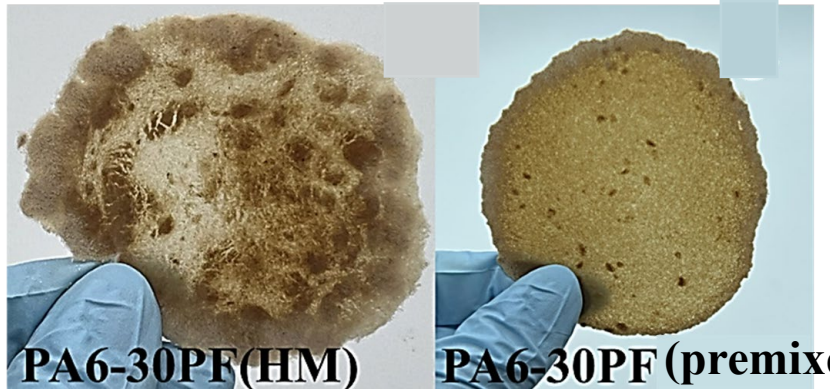
Sample of PA6 reinforced with 30wt% PF after 30 min premixing





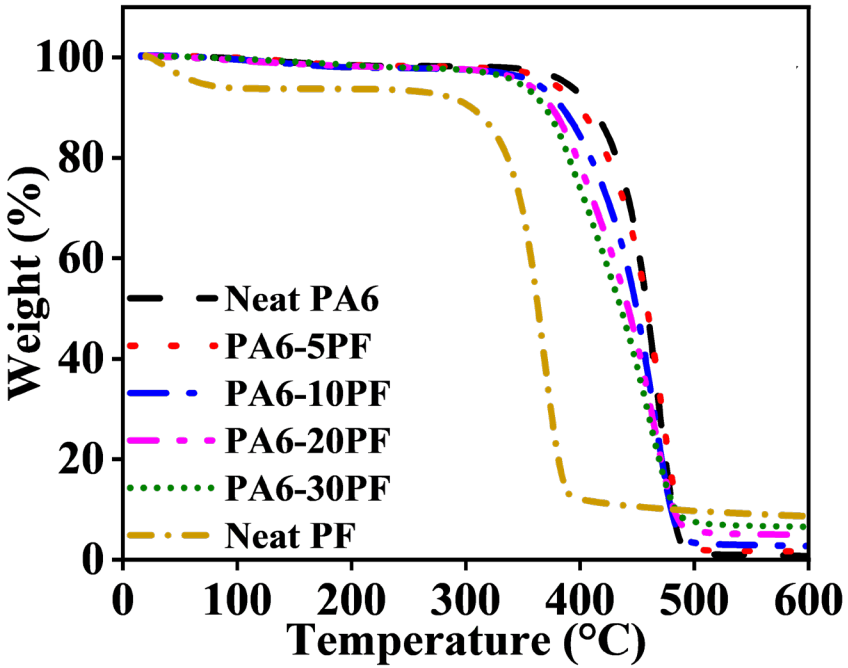


- Consistent distribution of pulp fibers
- PF embedded in polymer particles
- Chunky formed of fibers changed to spread-like form



Thermal Analysis

TGA



DSC

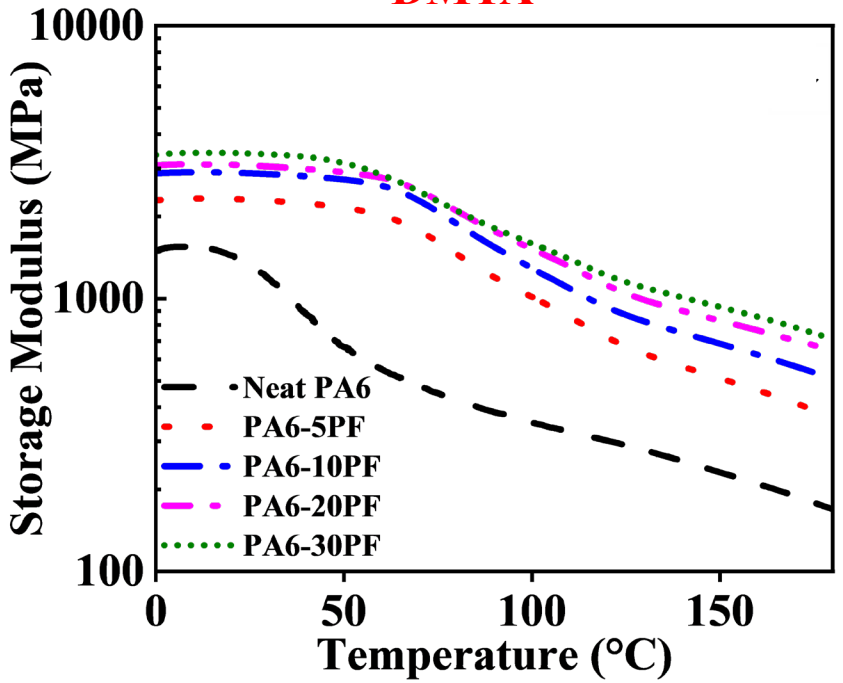
Sample name	Tm(°C)	Tc(°C)	%Xc
Neat PA6	221	190	26
PA6-5PF	221	189	27
PA6-10PF	222	188	28
PA6-20PF	223	187	31
PA6-30PF	222	187	33

- By incorporation of PFs, Onset degradation temperature decrease.
- Composite decomposition is the function of the amount of PFs.
- PFs can act as effective nucleating agents, promoting the formation of additional crystalline regions within the polymer matrix.

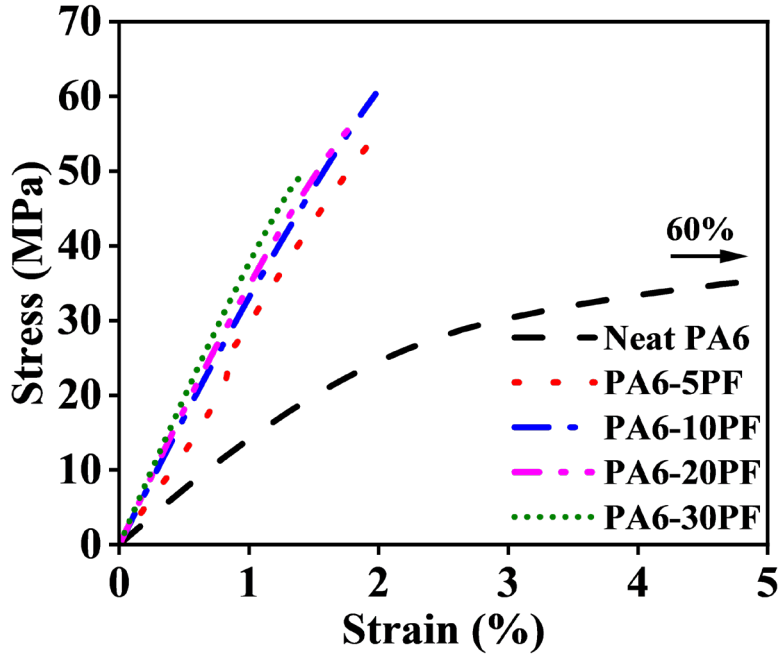
Polarized optical microscope

Mechanical Analysis

DMTA



Tensile Test

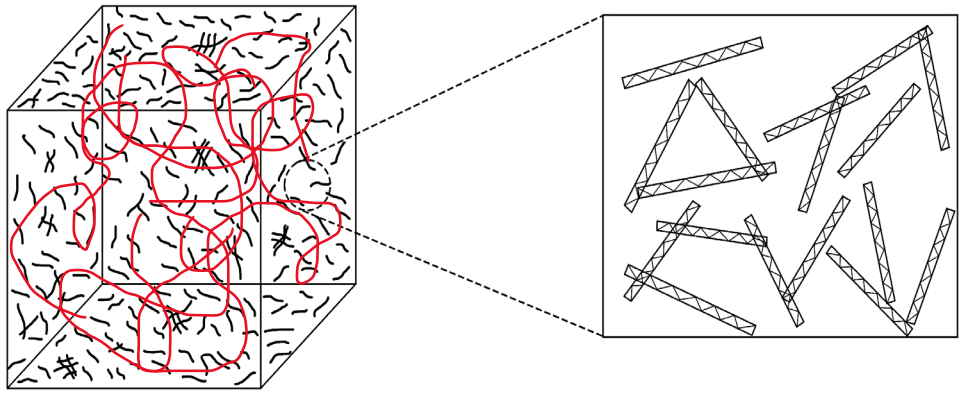


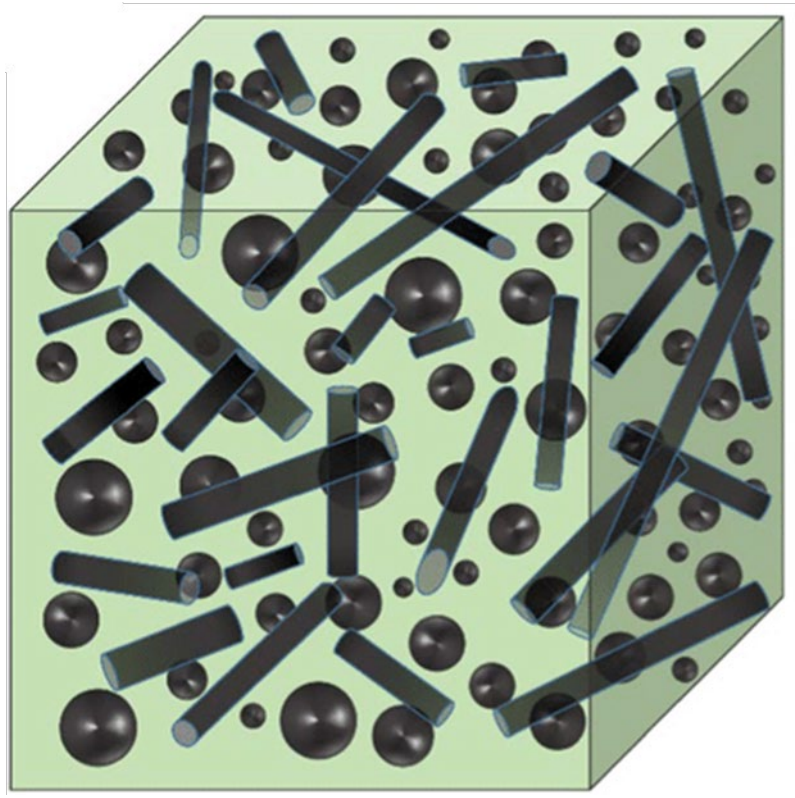
At 25°C

Sample name	Storage modulus(MPa)	Young's modulus(MPa)	Ultimate tensile strength(MPa)
Neat PA6	1300±110	1500±200	49±5
PA6-5PF	2300±50	2500±300	53±4
PA6-10PF	2900±90	3200±50	60±4
PA6-20PF	3150±60	3300±50	56±2
PA6-30PF	3400±100	3900±100	49±1

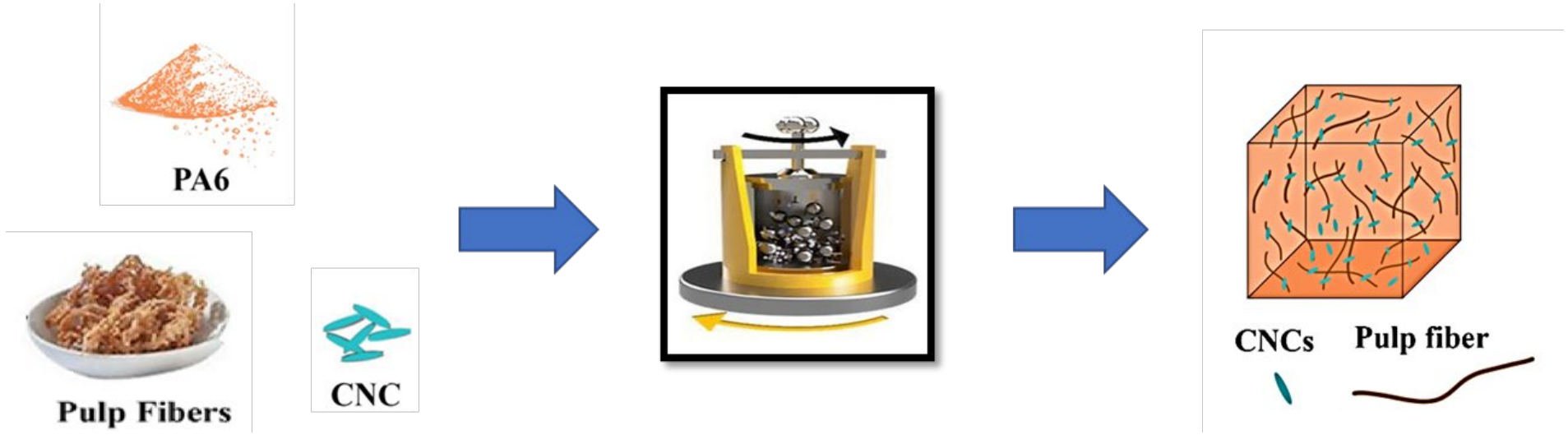
General Approach of Adding Nanoparticles to Polymer Composite:

- Enhanced mechanical properties without a decrease in processability.
- Improved flow behavior and viscosity control
- Improved compatibility with the polymer matrix
- Increased the sustainability of polymer composite.





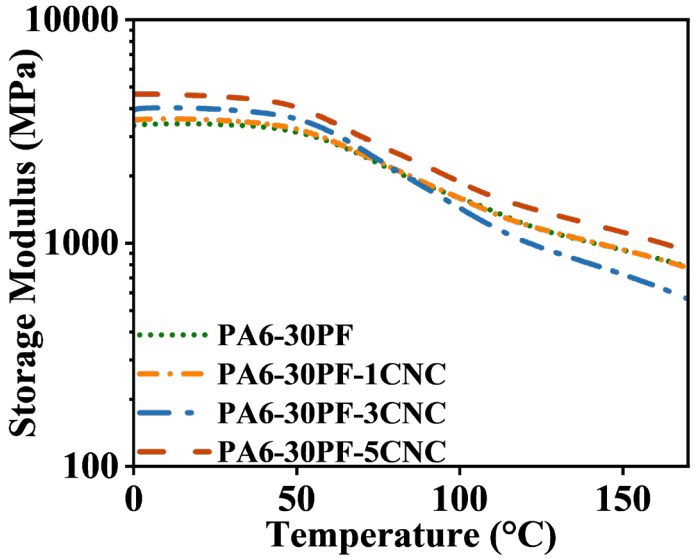
Addition of CNC to Improve Mechanical Properties



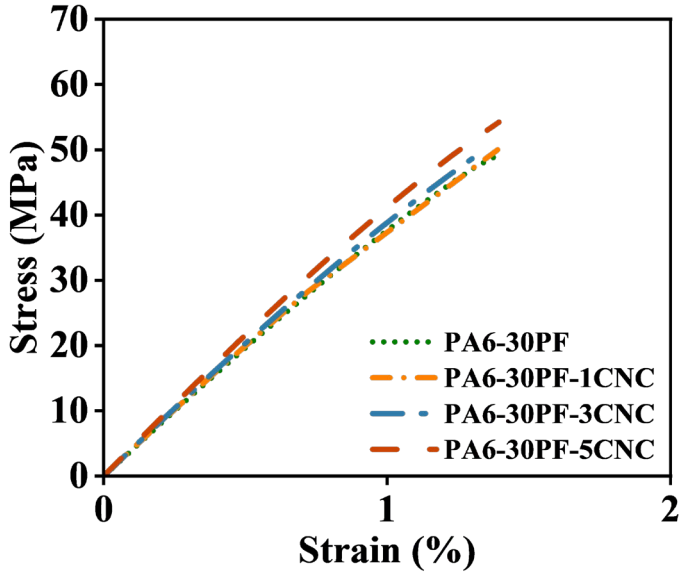
Sample name	PA6(wt%)	PF(wt%)	CNC(wt%)
PA6-30PF-1CNC	69	30	1
PA6-30PF-3CNC	67	30	3
PA6-30PF-5CNC	65	30	5

Mechanical Analysis

DMTA



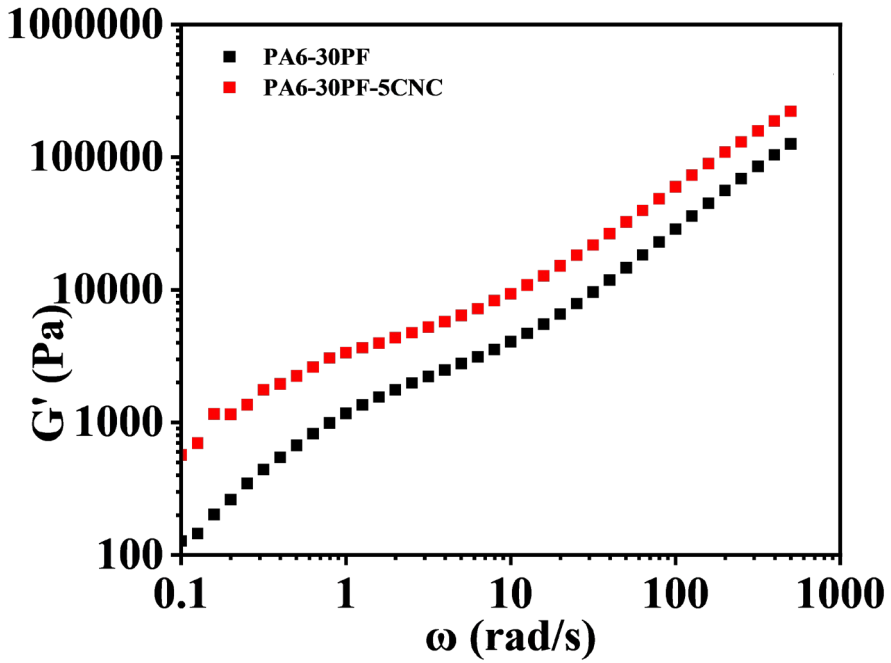
Tensile Test

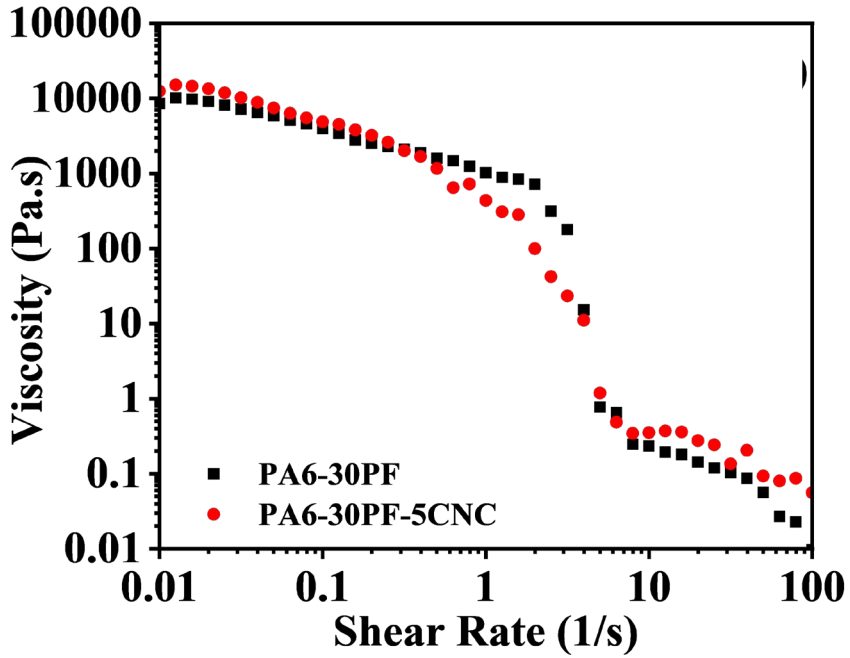


At 25°C

Sample name	Storage modulus (MPa)	Young's Modulus (MPa)	Ultimate tensile strength (MPa)
PA6-30PF	3400±100	3900±100	49±1
PA6-30PF-1CNC	3550±80	3900±120	48±1
PA6-30PF-3CNC	4000±50	4100±50	49±1
PA6-30PF-5CNC	4500±100	4300±90	54±2

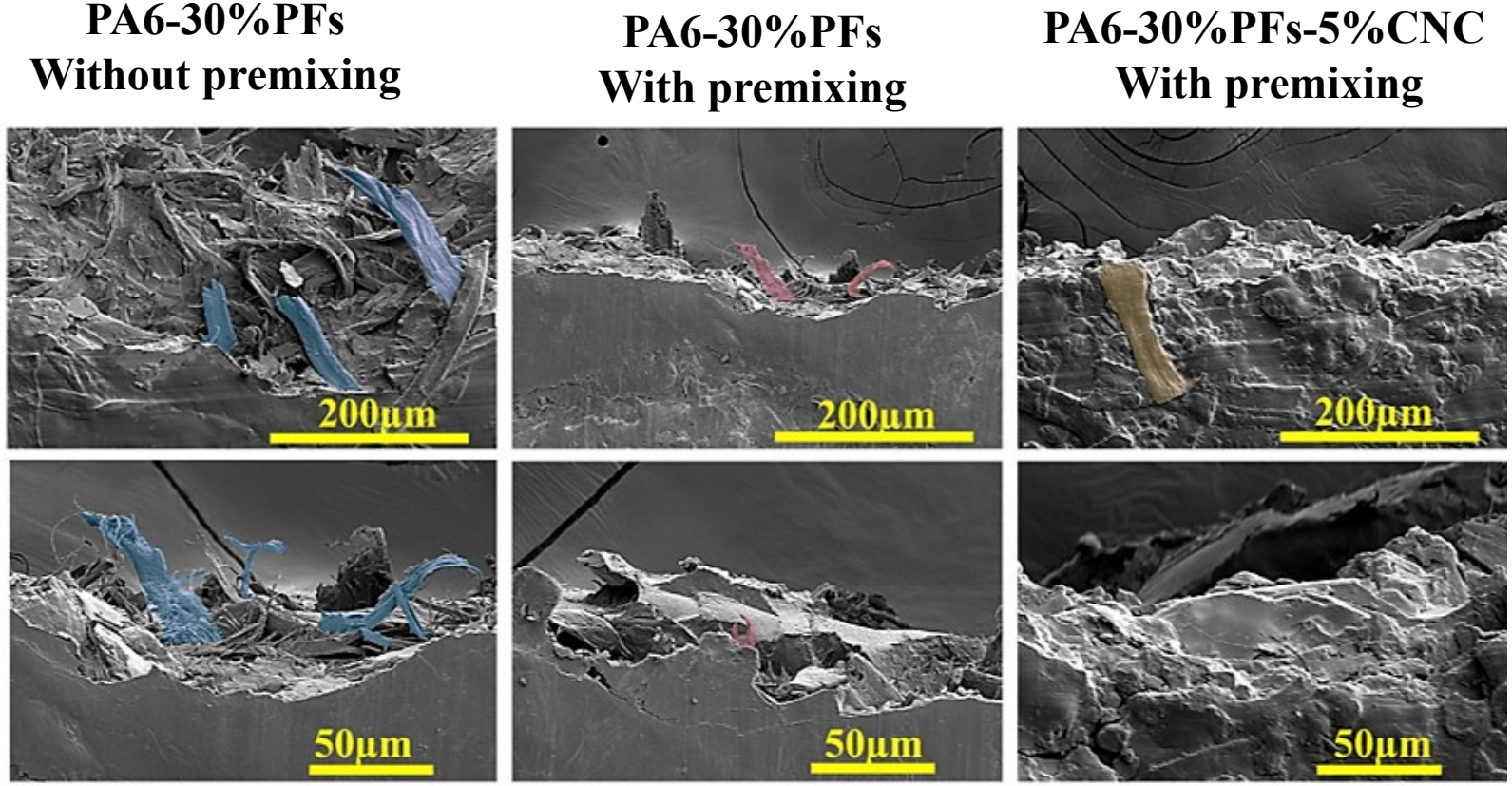
Rheological Analysis





- The storage modulus of the polymer composite increases with the addition of CNC.
- The increase in viscosity was not significant, the processability of the composite is maintained

Morphology of Fracture Surface



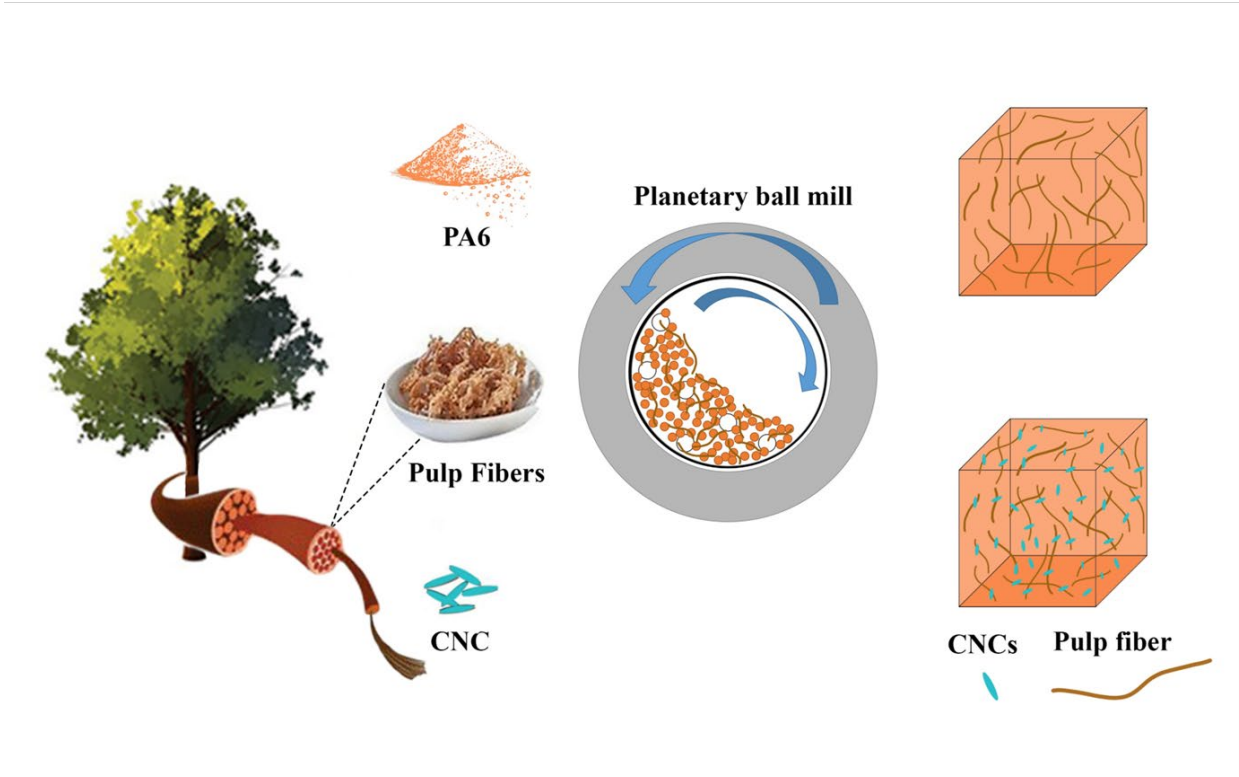
- Pulled-out fibers are longer in the sample without premixing.
- Premixed samples show better dispersion of micronized fibers and more embedment in polymer matrices.
- Sample with 5wt% CNC, the interface between fibers and polymer becomes stronger, so fibers are not pulled out.

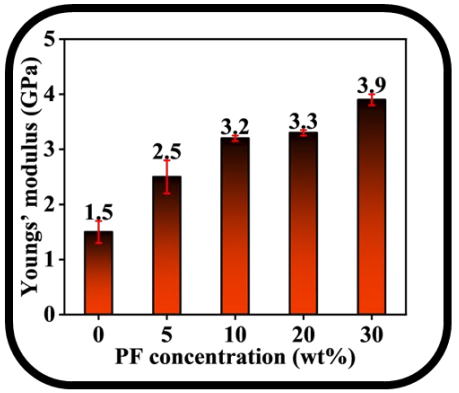
Conclusion

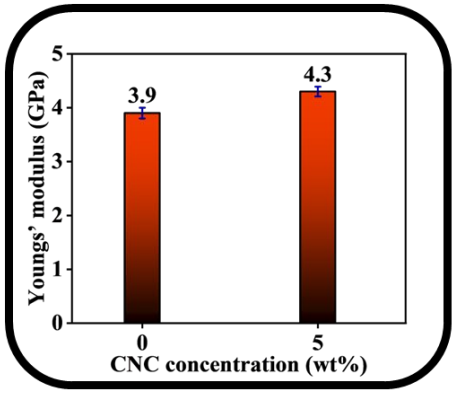
1-We showed premixing ball milling method is a promising technique to make polymer reinforced with CNF.

2-By introducing PFs up to 30wt% via ball milling, we made a strong polymer composite.

3- CNCs had a superior effect on mechanical properties of composite reinforced with PFs.



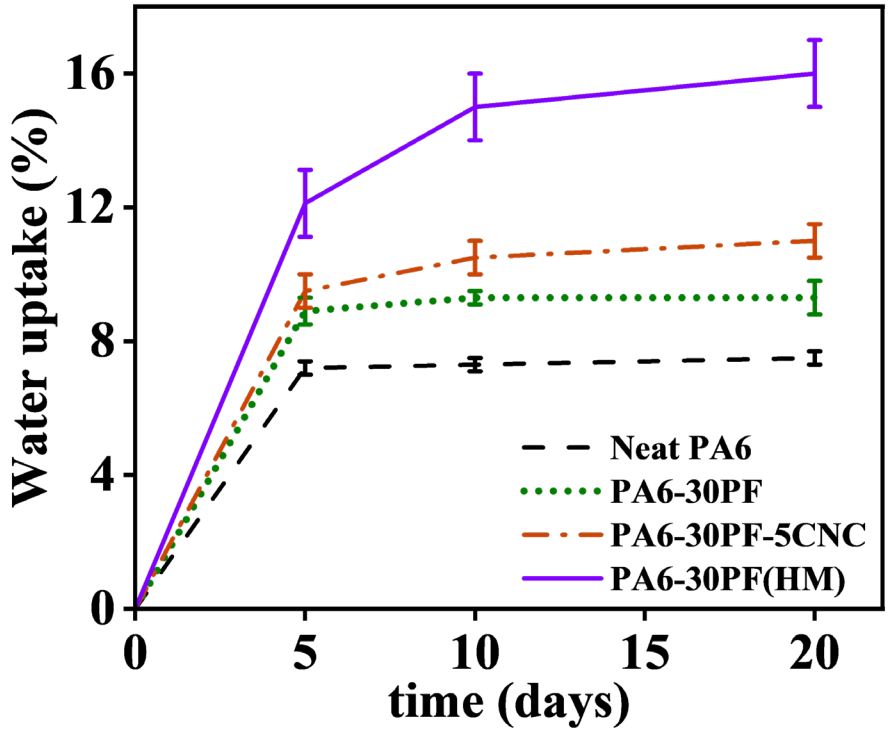




Special Thanks

Thank you

Questions?

Water Uptake Capacity

- Premixed samples have lower water absorption.
  - Dimensional stability
  - Better mechanical properties
- Higher water uptake for the HM sample is related to bad dispersion of PFs in the sample.