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Investigating the Aging of Nitrile Rubber NBR Reinforced with Date Palm Fiber

Presented by

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Investigating the Aging of Nitrile Rubber NBR Reinforced with Date Palm Fiber

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Content

1. Introduction
2. Experimental work
3. Results and Discussion
4. Conclusions & Recommendations

Introduction

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- Part of the Kingdom vision 2030.
- The interest in bio-based/sustainable material is increasing.
- The Kingdom is rich in petroleum products.
- Date palm trees are very popular in the Middle East
- Came the idea of combining both recourses
  - optimize the oil consumption.
  - Increase sustainability.
  - Reduce product prices.
  - Contribute towards realization of Vision 2030.

Introduction

- Why DPF and NBR
  - NBR has wide uses
  - Compatibility
- Date palm tree is rich in cellulose.
  - Can be used as reinforcing/ filler
    - Very good chemical and mechanical properties (natural fibers)
    - Availability
    - price
- In the past, by product of the pruning like
  - Leaf’s
  - Spadix stem
  - Trunk and sheath
- Were burnt !

Introduction

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- Fibers (high aspect ratio)
  - Natural (cotton, wool, silk , Hemp …etc.)
  - Man-made (glass fibers, carbon fiber, polyester…etc.)
- A fibers rich plant is (date palm tree)
- More that 82M tree in MENA region

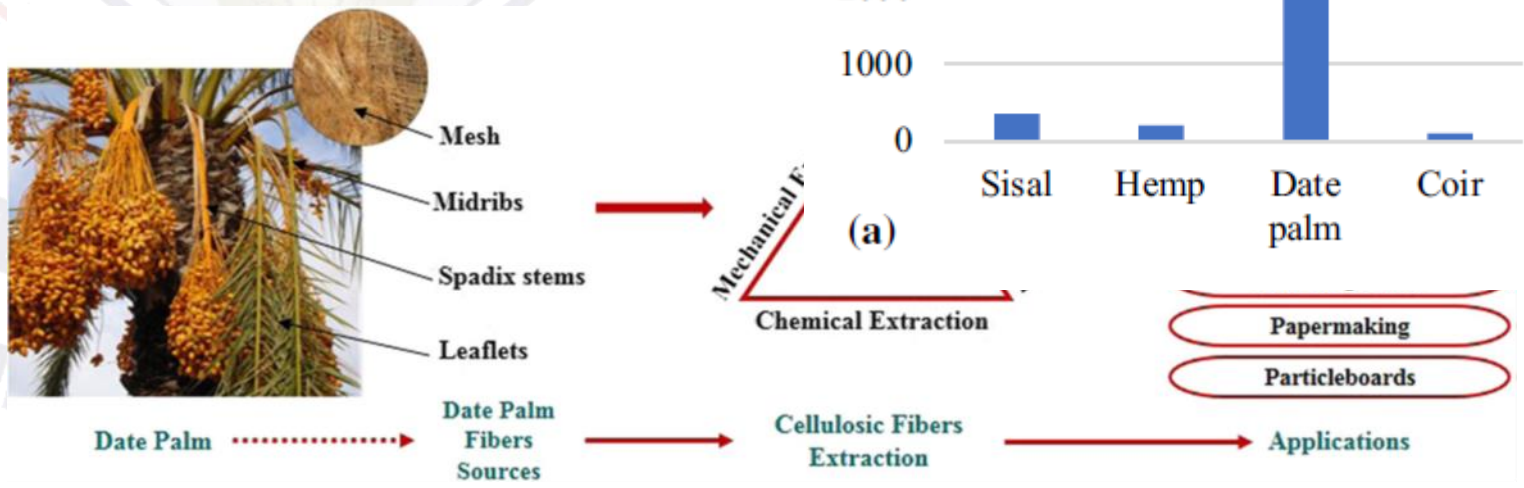
Introduction

Great potentials (production/cost)

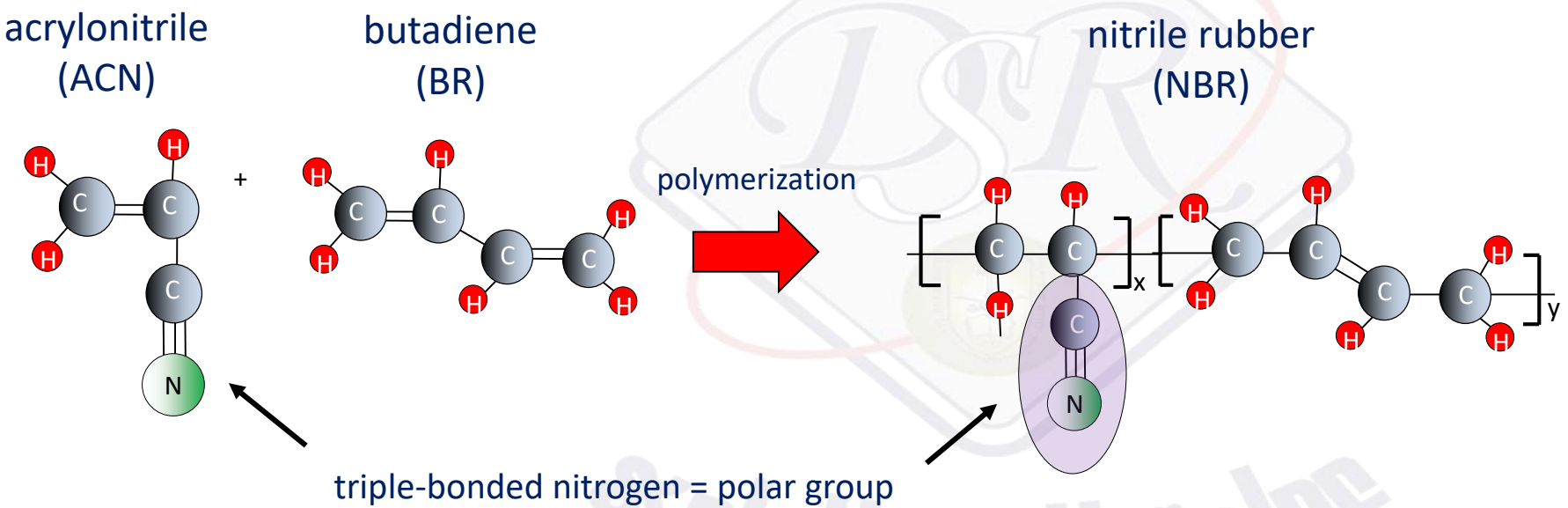
- The highest annual production plant
- The cheapest

Great potentials

Use it as fibers



Nitrile Rubber (NBR) –Introduction- The Structure



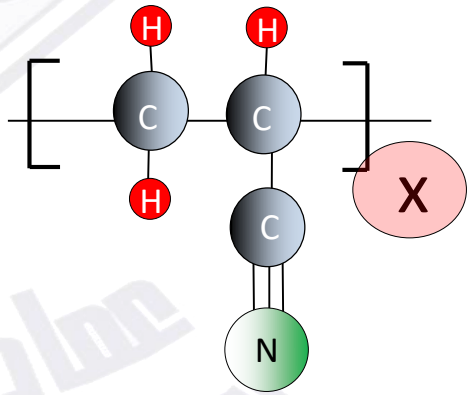
- The elastomer, NBR, is made by copolymerization of butadiene (BR) with acrylonitrile (ACN).
- The nitrile pendants make NBR a polar elastomer.

Nitrile Rubber (NBR) – Oil Resistance

- Nitrile rubbers are <u>polar</u>➔ resistant to <u>non-polar</u> liquids hydrocarbon oils and solvents

More nitrile groups → more polar

Designation	ACN Content
low nitrile	<30%
medium nitrile	30% to 45%
high nitrile	45% to 50% (or more)



acrylonitrile

Nitrile rubbers are designated (named) by concentration of C≡ N groups, the fraction “x”.

Nitrile Rubber (NBR) – Resilience

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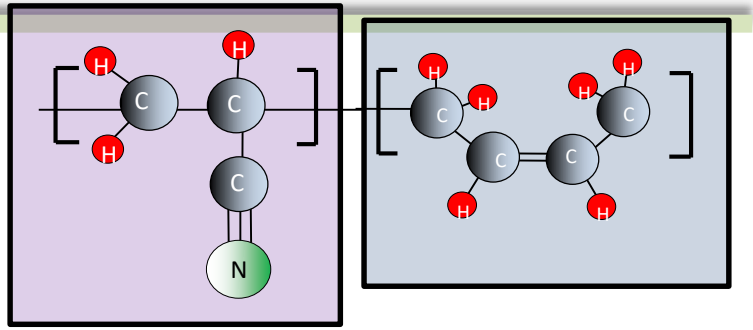
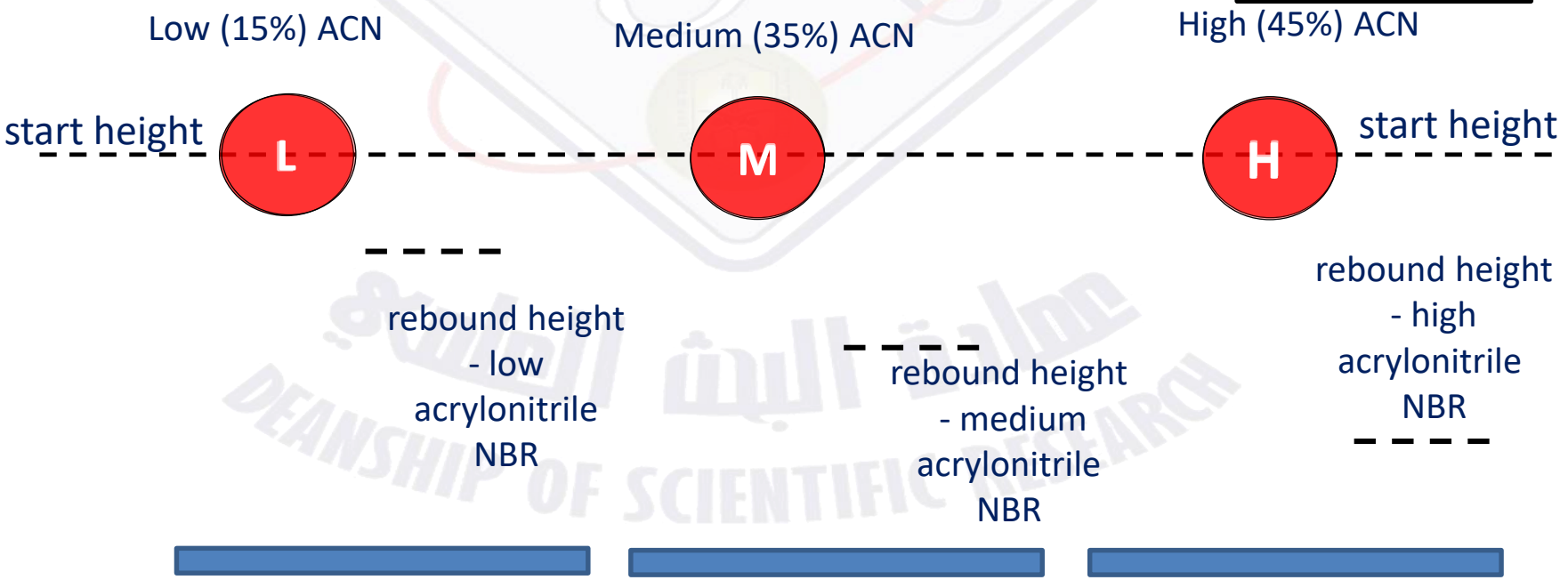
- Rebound height depends on acrylonitrile (ACN) concentrations:





As the concentration of plastic (ACN) repeat unit increases, resilience decreases.

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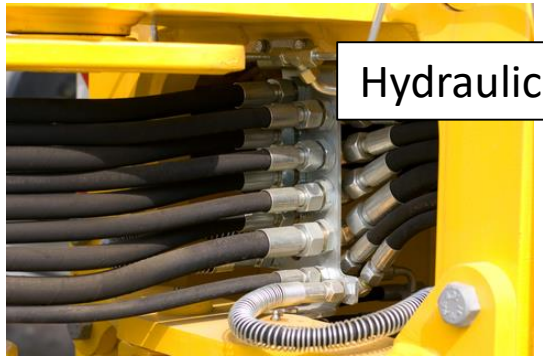
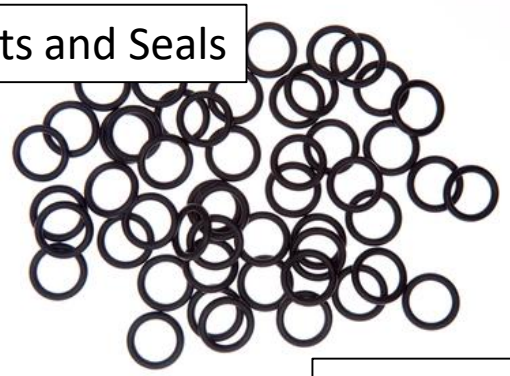
Nitrile Rubber (NBR) – Common Uses



Hydraulic Hoses

O-Rings, Gaskets and Seals

Rubber Conveyor Belts



Printing Roll Covers

Work Plan

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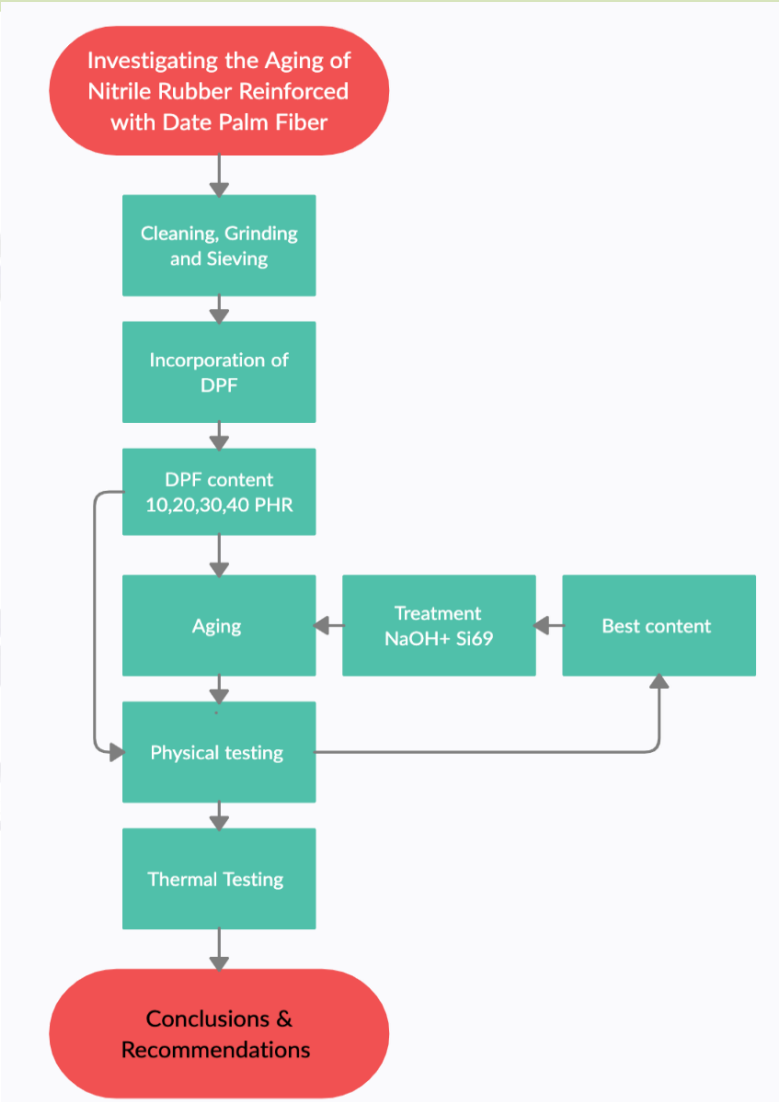

Experimental work

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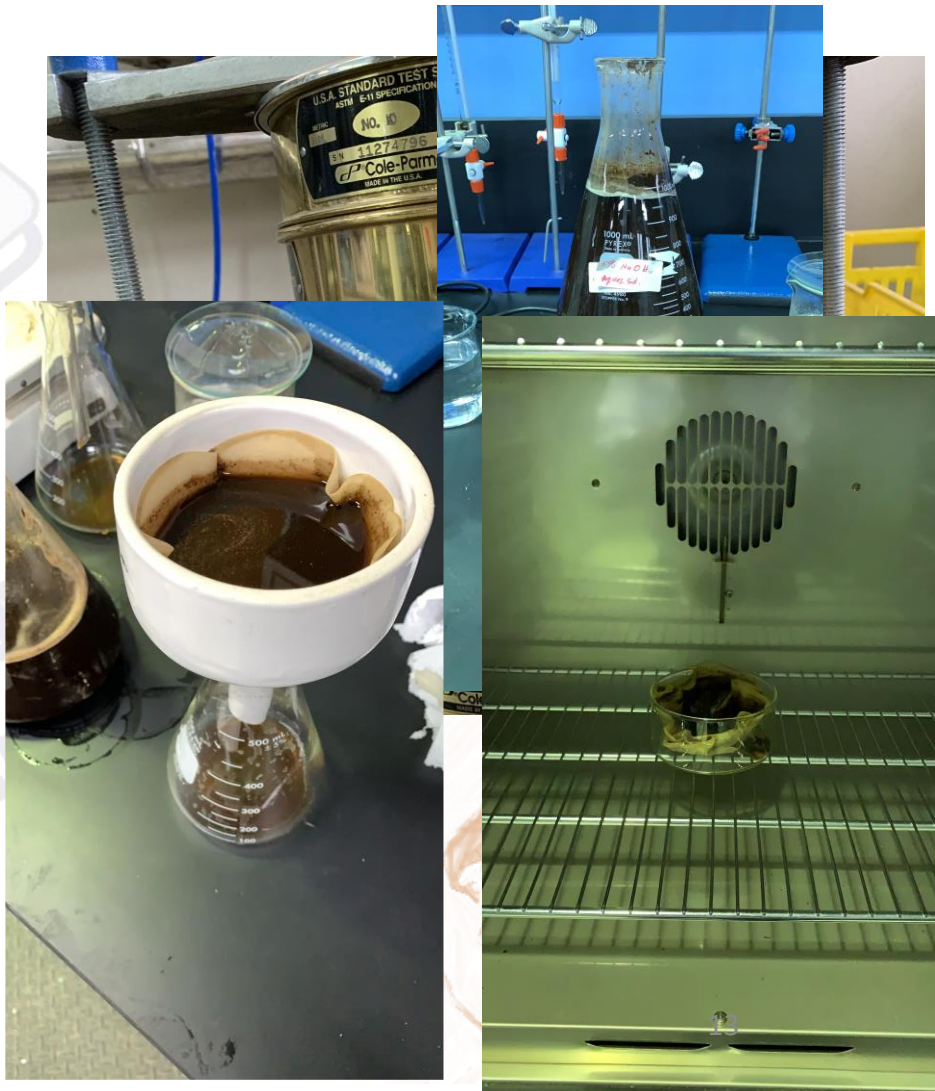
- DPF
  - Njoom- Al-Kharj, Saudi Arabia
  - Grinded
    - Stardust (Japan) coffee beans grinders
  - Sieved
    - Cole-Parmer sieves and a shaker
  - Particle size analyzer
    - Malvern mastersizer 2000
  - Treatment aqueous solution of 5 % NaOH for 24h at 70^0C
  - Washing and Drying



Design of Experiment

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Design of Experiment

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Ingredients	NBR Unfilled	NBR + CB	NBR+DPF 10 PHR	NBR+DPF 10 PHR treated	NBR+DPF 20 PHR	NBR+DPF 30 PHR	NBR+DPF 40 PHR	NBR+DPF 40 PHR treated
NBR (Nitrile rubber) 3250	100	100	100	100	100	100	100	100
ZnO	5	5	5	5	5	5	5	5
Stearic Acid	2	2	2	2	2	2	2	2
DPF	0	0	10	10	20	30	40	40
CB	0	10	0	0	0	0	0	0
Aromatic oil	5	5	5	5	5	5	5	5
Sulfur	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
6PPD	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
DPG	2	2	2	2	2	2	2	2
CBS	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Si 69	0	0	0	7	0	0	0	7

Experimental work

- Mixing
  - 2 stage mixing
    - Internal mixer (Plasti-corder Lab station- **Brabender**) fitted with two counter-rotating tangential blades.

Time (min)	Ingredient	RPM
0	Rubber (NBR 3250)	65
1	Additives (ZnO-Steraic Acid)	
2	Filler (CB/DPF) and Si69	90
3	Oil	
7	End	

- **Comerio lab** Open mill to mix the curatives te
  of 50 RPM

Experimental work

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- Cure characteristics
  - GÖTTFERT Moving Die Rheometer (MDR) (D 6601)
  - Temperature 160^{0}C
  - 15 min (treated compounds – 60min)
- Curing
  - Wabash heated press
  - Temperature 160 ^{0}C
  - $T_{90} + 5$ minutes.
- Samples
  - Dumb-bell shape
  - Standard ASTM D412 Die

Mechanical testing

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- Tensile properties
  - Tensile tester equipped with a long range extensiometer- the crosshead speed was 500 mm/minute using ASTM D412
- Dynamic Mechanical Analyzer
  - Strain sweep test from 0.01 to 4% strain. The frequency was 1 Hz at room temperature. The fixture used was thin film fixture.

Aging

- Air oven
  - Binder Heating ovens using ASTM D 573 temperature of 150^0 C for 24h + 0.5h warming
- Ozone chamber
  - Ektron Ozone test chamber - using ASTM D1149 concentration of 150 PPHM 24h at 50^0C
- Oil submergence
  - CCSi Elastek heating block using ASTM D471 temperature of 120 ^{0}C for 24h + 0.5h warming

Thermal Testing

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- TGA
  - Around 12 mg
  - Room temperature to 550 ^{0}C in Nitrogen environment then from 550 ^{0}C to 900 ^{0}C in oxygen environment.
  - 20^{0}C/min
- DSC
  - TA Q2000
  - 5-10 mg
  - Temperature range -80^{0}C to 70^{0}C.
  - 10^{0}C/min.

Results and Discussion

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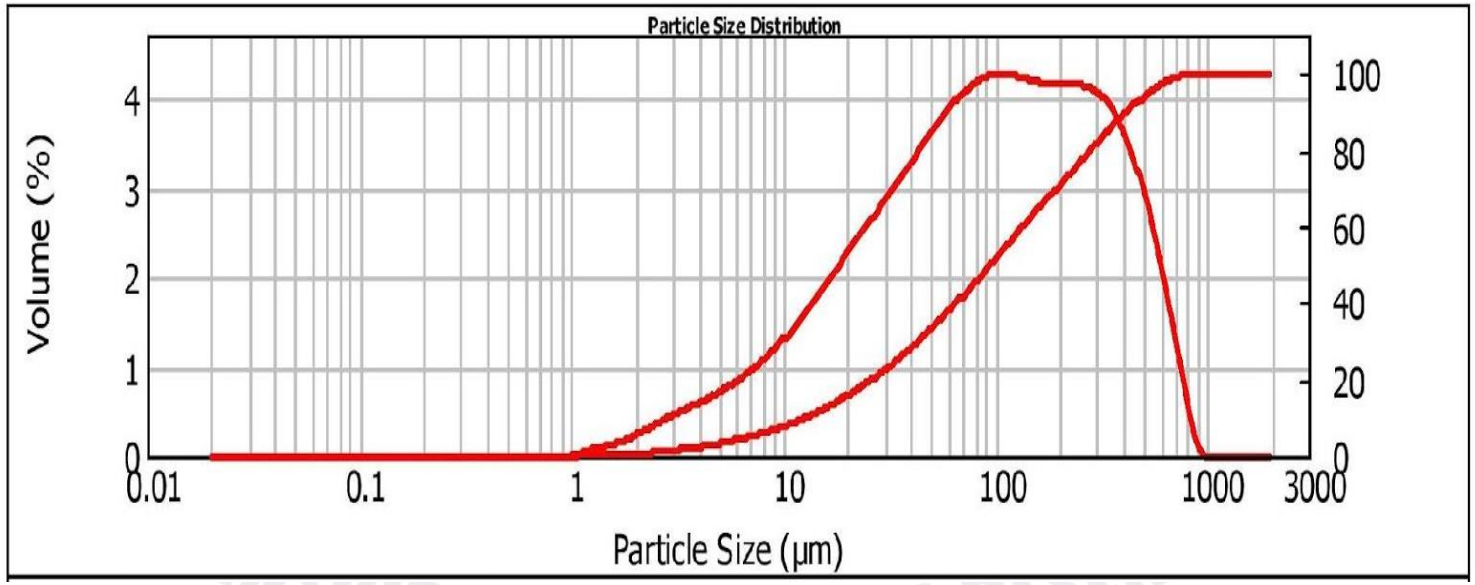
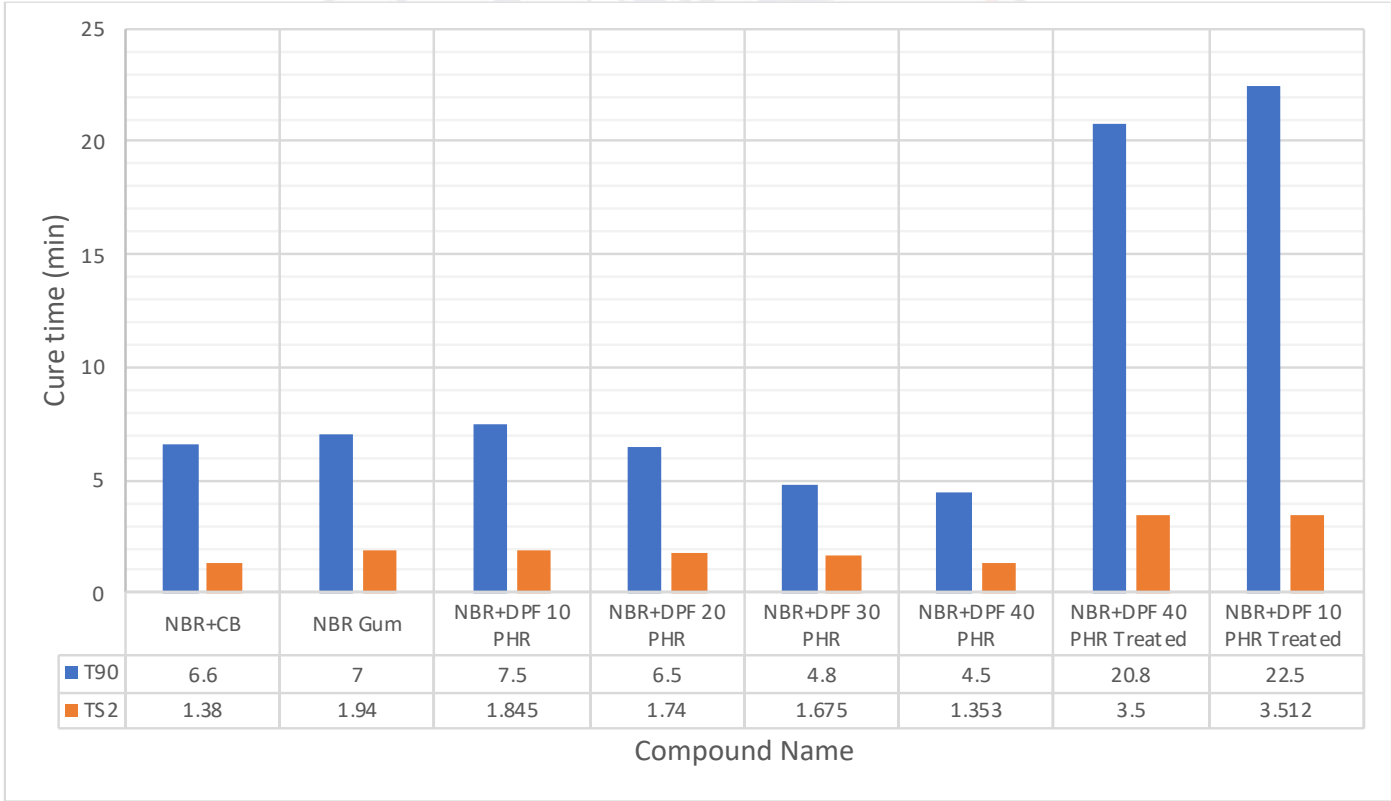
- Size Analysis



Results and Discussion

Deanship of Scientific Research

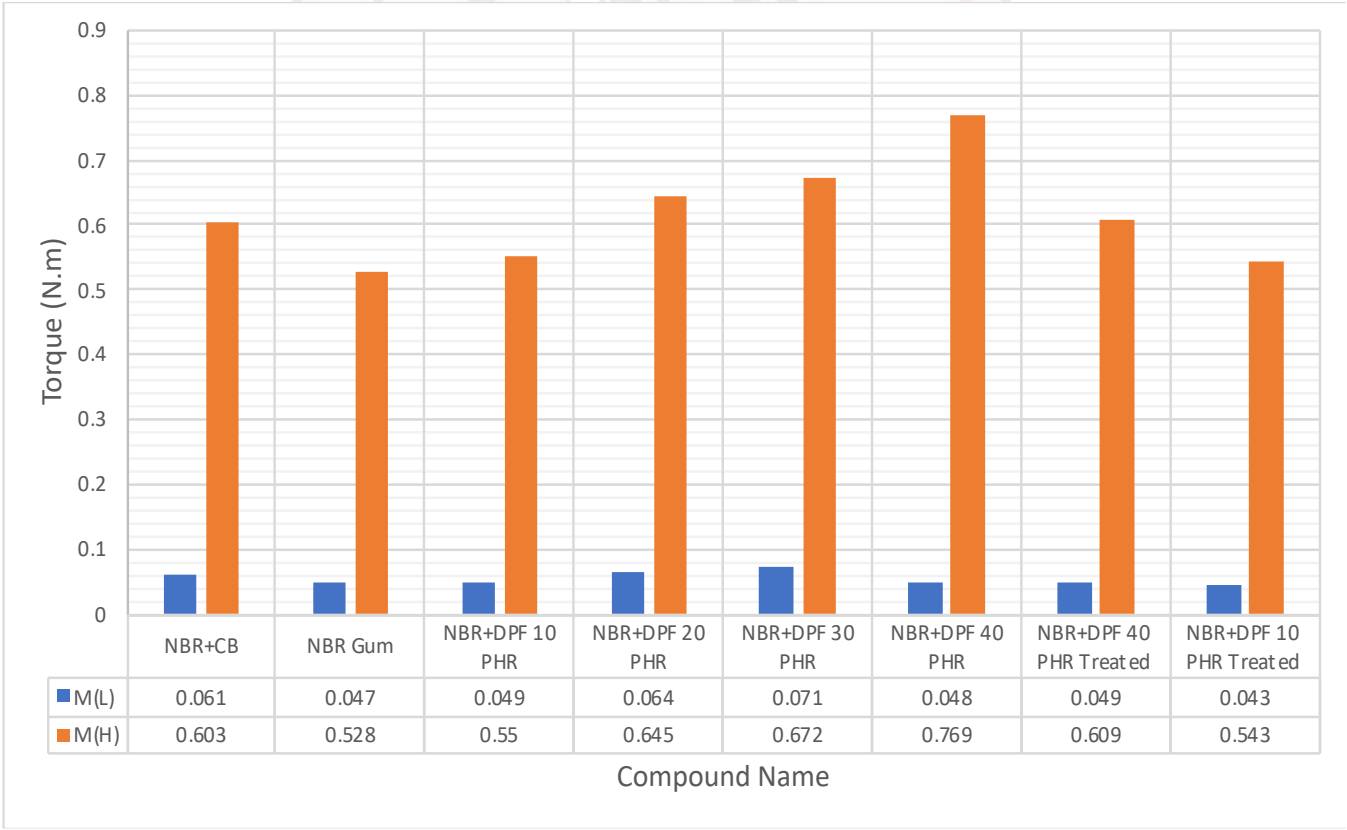
- Cure Characteristics



Results and Discussion

Deanship of Scientific Research

- Cure Characteristics



	NBR+CB	NBR Gum	NBR+DPF 10 PHR	NBR+DPF 20 PHR	NBR+DPF 30 PHR	NBR+DPF 40 PHR	NBR+DPF 40 PHR Treated	NBR+DPF 10 PHR Treated
M(L)	0.061	0.047	0.049	0.064	0.071	0.048	0.049	0.043
M(H)	0.603	0.528	0.55	0.645	0.672	0.769	0.609	0.543

Results and Discussion

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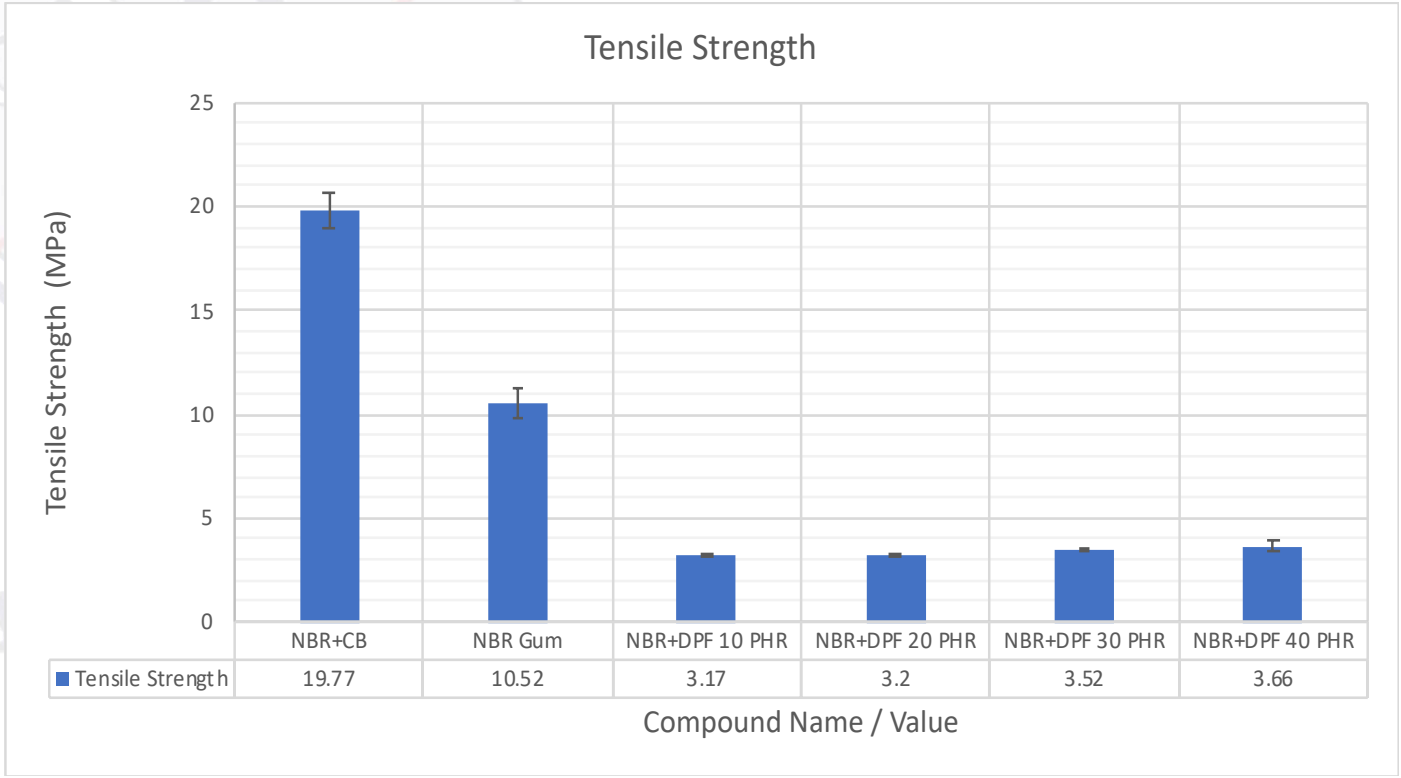
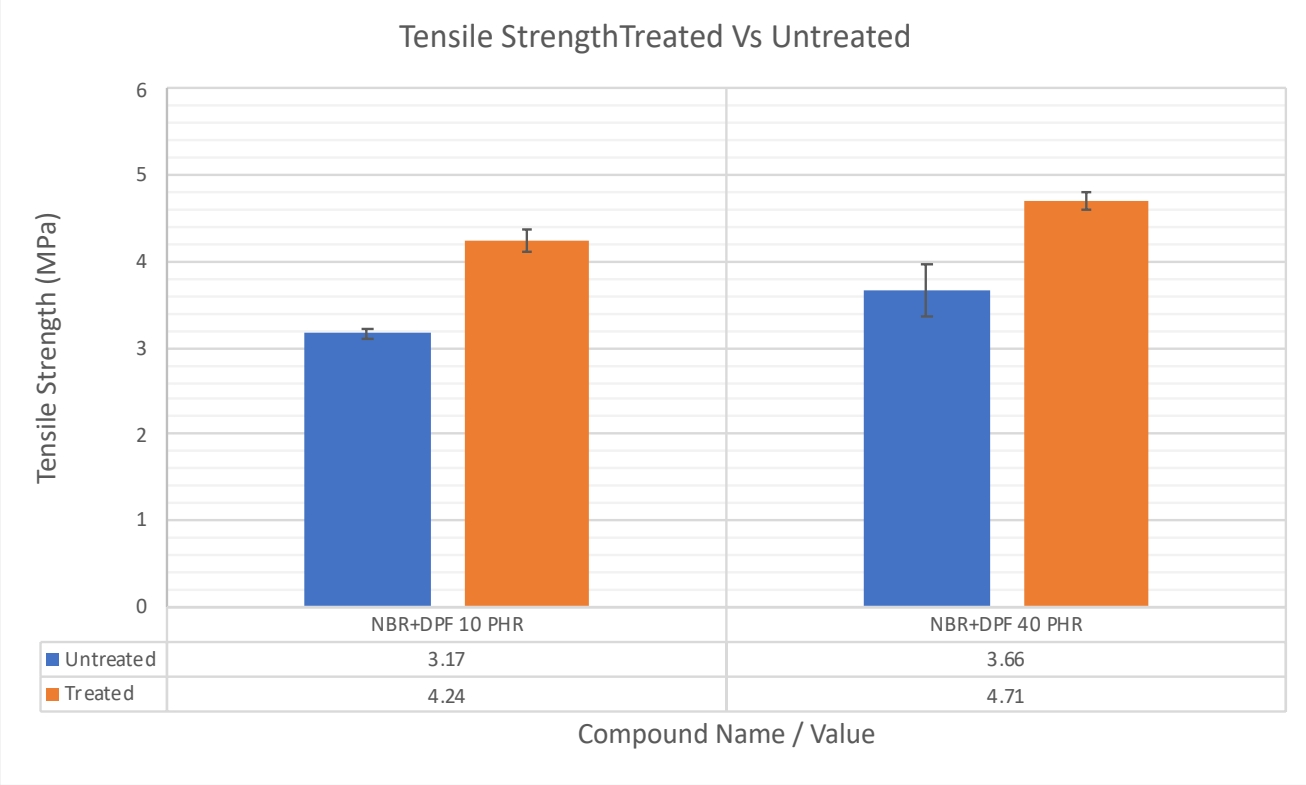
- Mechanical Results
  - Tensile Strength:
    - Unaged Tensile Strength



Results and Discussion

Deanship of Scientific Research

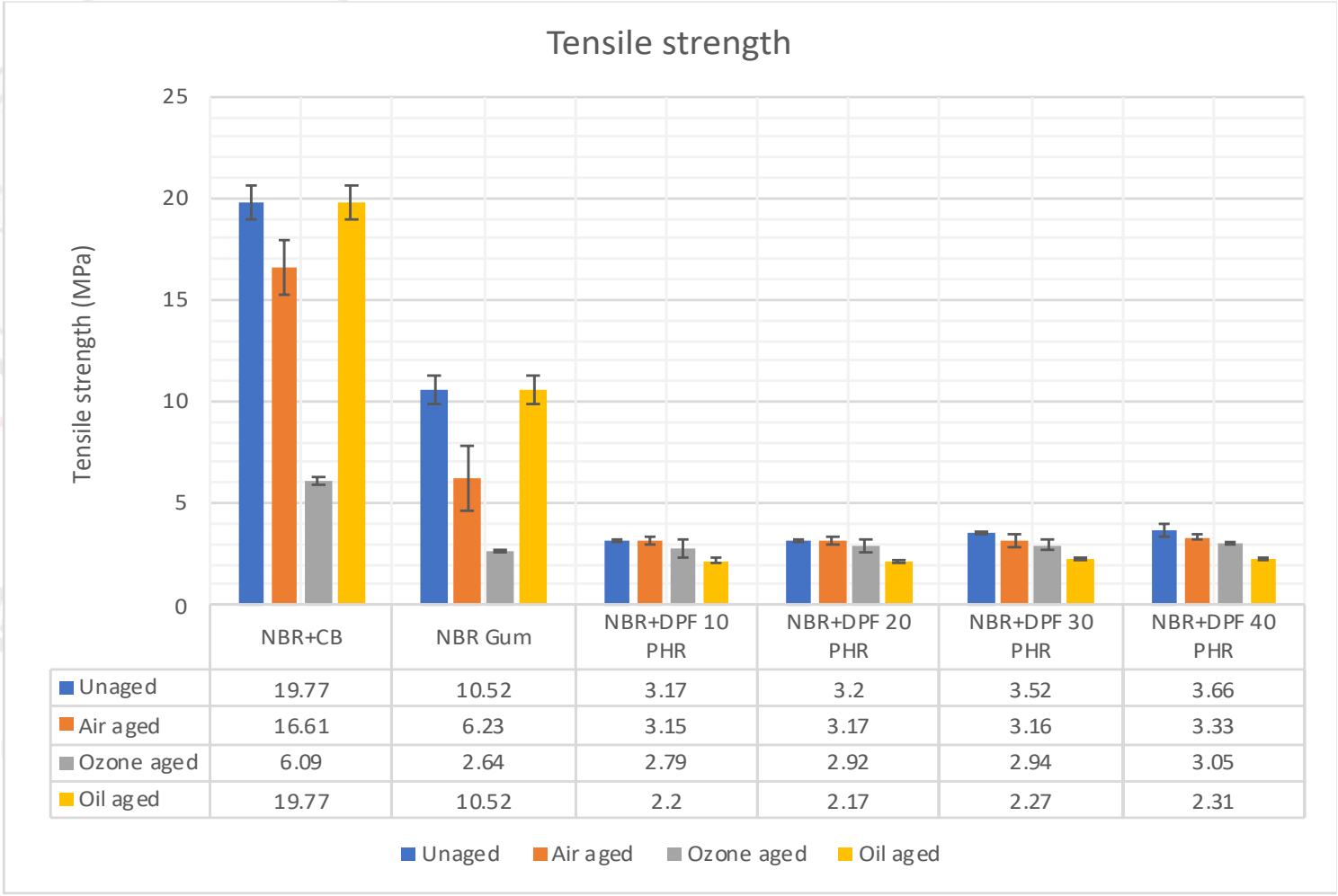
- Mechanical Results
  - Tensile Strength:
    - Unaged Tensile Strength
      - Treated Vs Untreated



Results and Discussion

Deanship of Scientific Research

- Mechanical Results
  - Tensile Strength:
    - Aged Tensile Strength



Results and Discussion

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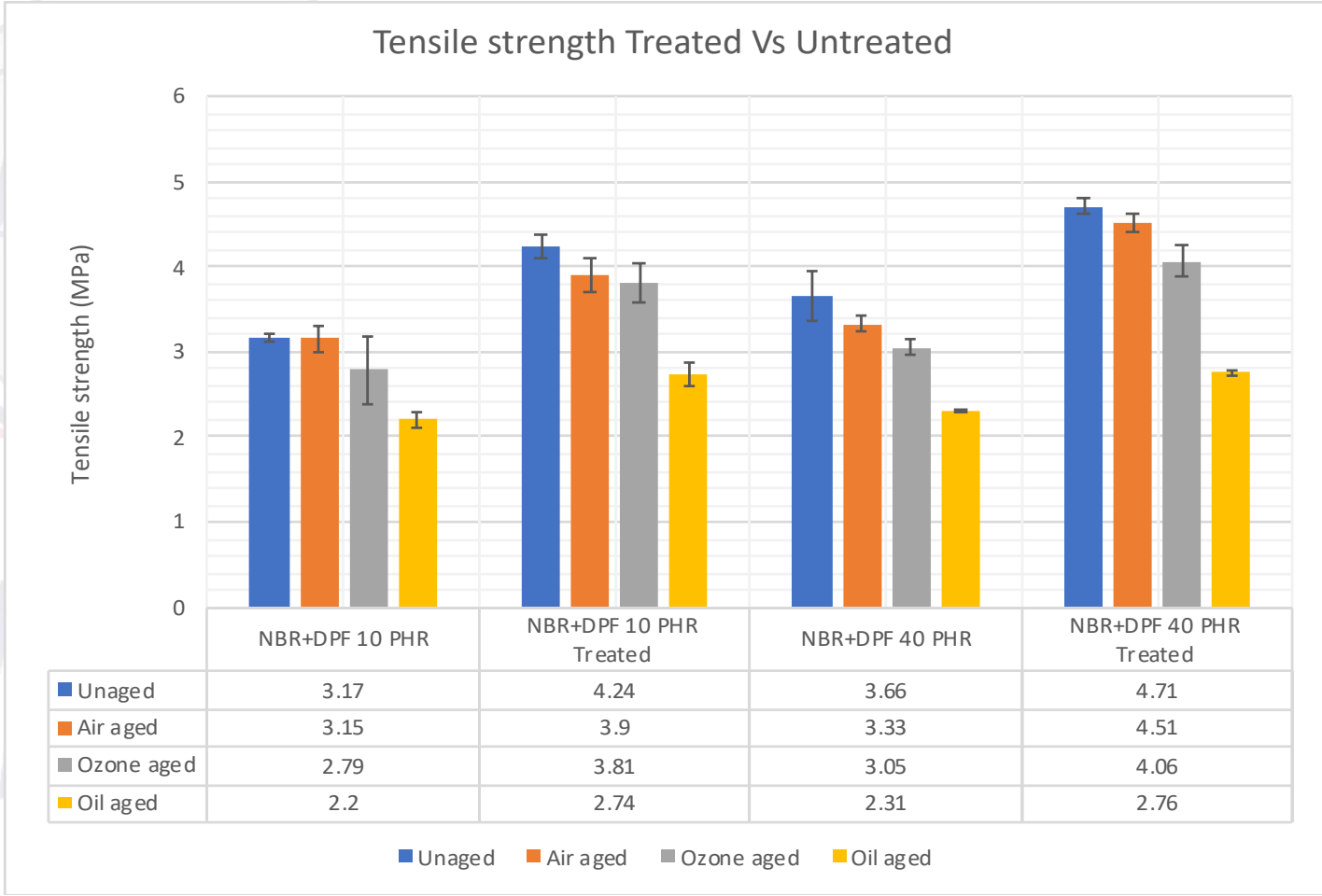
- Mechanical Results
  - Tensile Strength:
    - Aged Tensile Strength
    - Treated Vs Untreated



	NBR+DPF 10 PHR	NBR+DPF 10 PHR Treated	NBR+DPF 40 PHR	NBR+DPF 40 PHR Treated
Unaged	3.17	4.24	3.66	4.71
Air aged	3.15	3.9	3.33	4.51
Ozone aged	2.79	3.81	3.05	4.06
Oil aged	2.2	2.74	2.31	2.76

Results and Discussion

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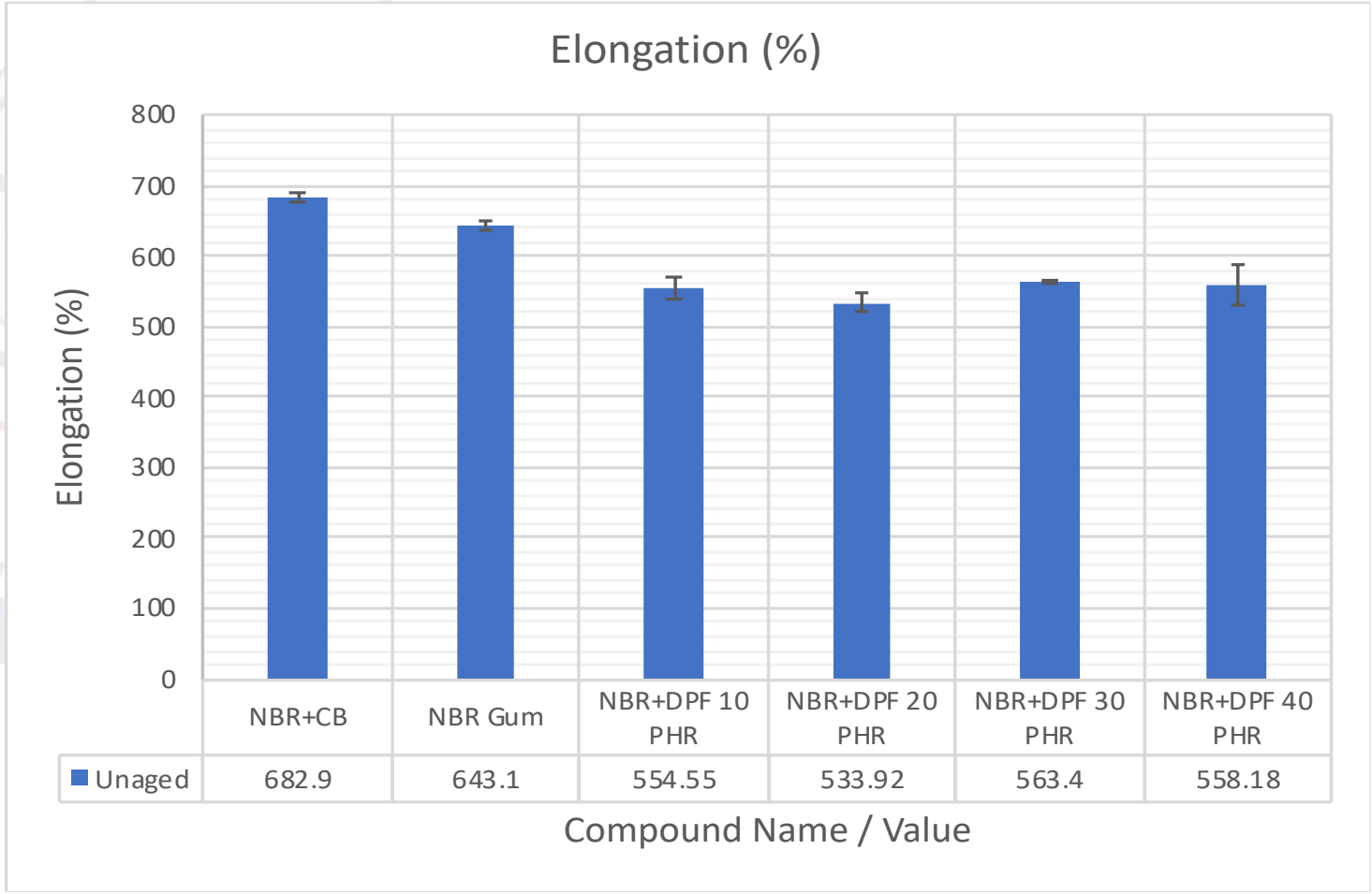
- Mechanical Results
  - Elongation :
    - Unaged Elongation



Results and Discussion

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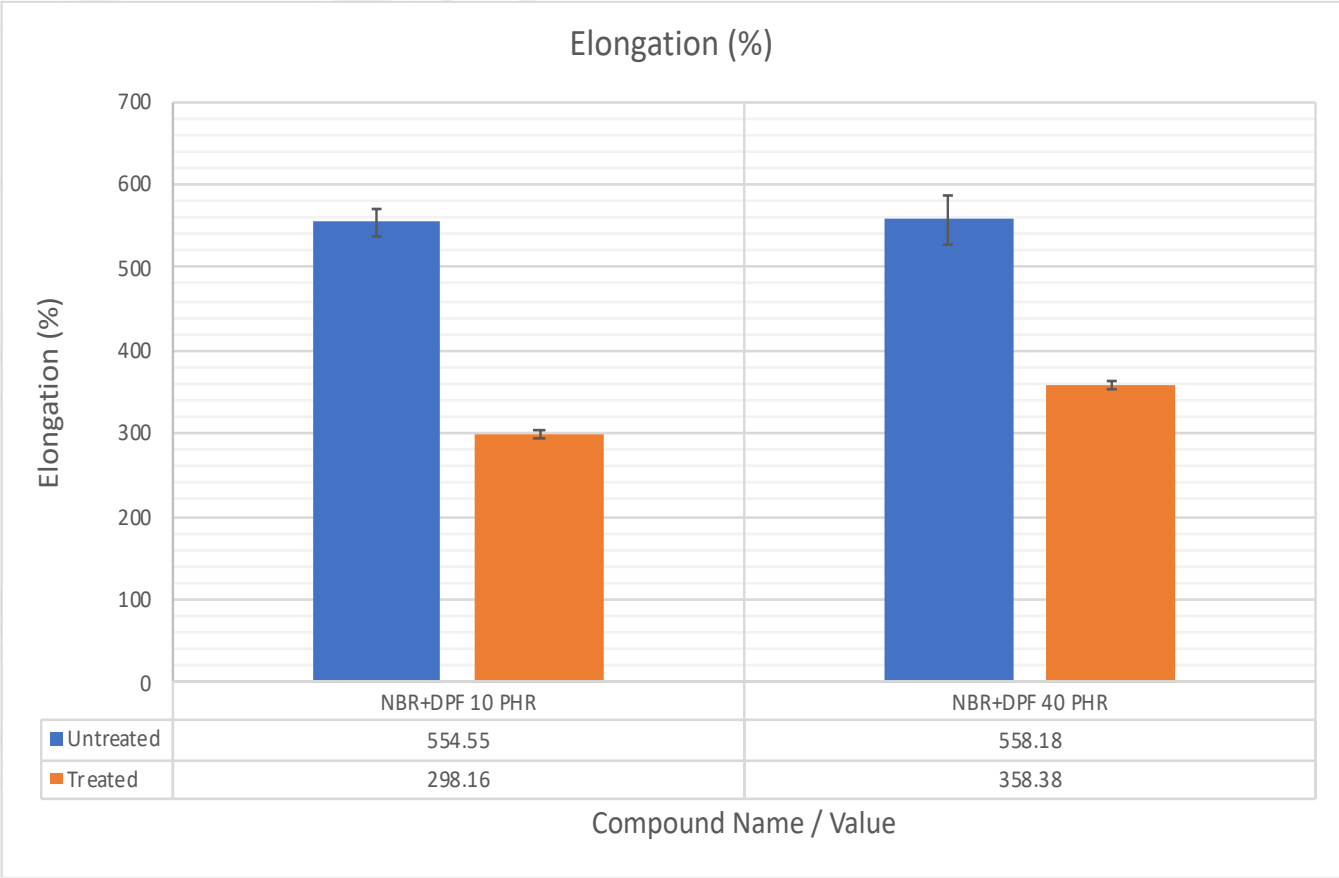
- Mechanical Results
  - Elongation :
    - Unaged Elongation
    - Treated Vs Untreated



Results and Discussion

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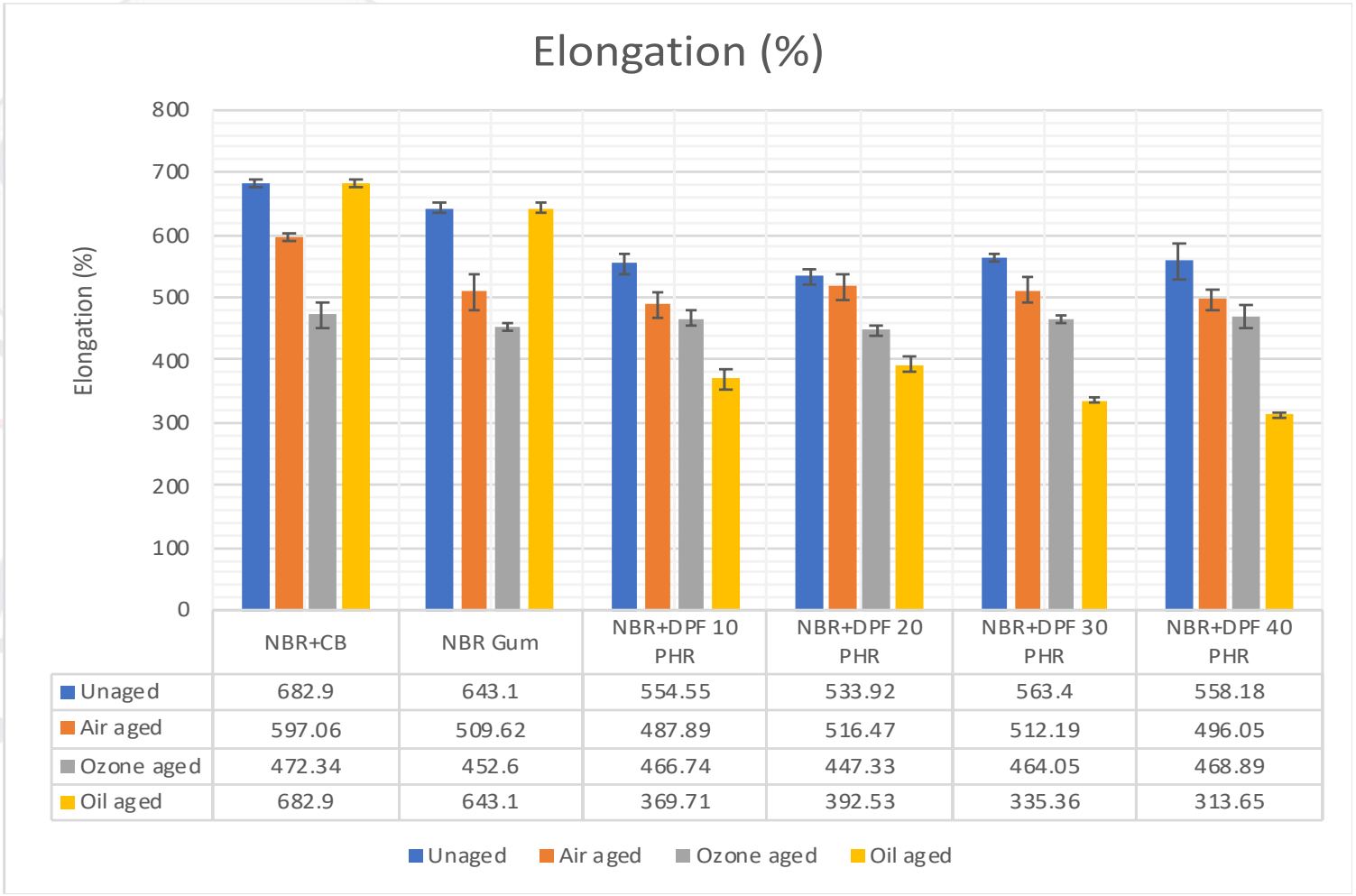
- Mechanical Results
  - Elongation:
    - Aged elongation



	NBR+CB	NBR Gum	NBR+DPF 10 PHR	NBR+DPF 20 PHR	NBR+DPF 30 PHR	NBR+DPF 40 PHR
Unaged	682.9	643.1	554.55	533.92	563.4	558.18
Air aged	597.06	509.62	487.89	516.47	512.19	496.05
Ozone aged	472.34	452.6	466.74	447.33	464.05	468.89
Oil aged	682.9	643.1	369.71	392.53	335.36	313.65

Results and Discussion

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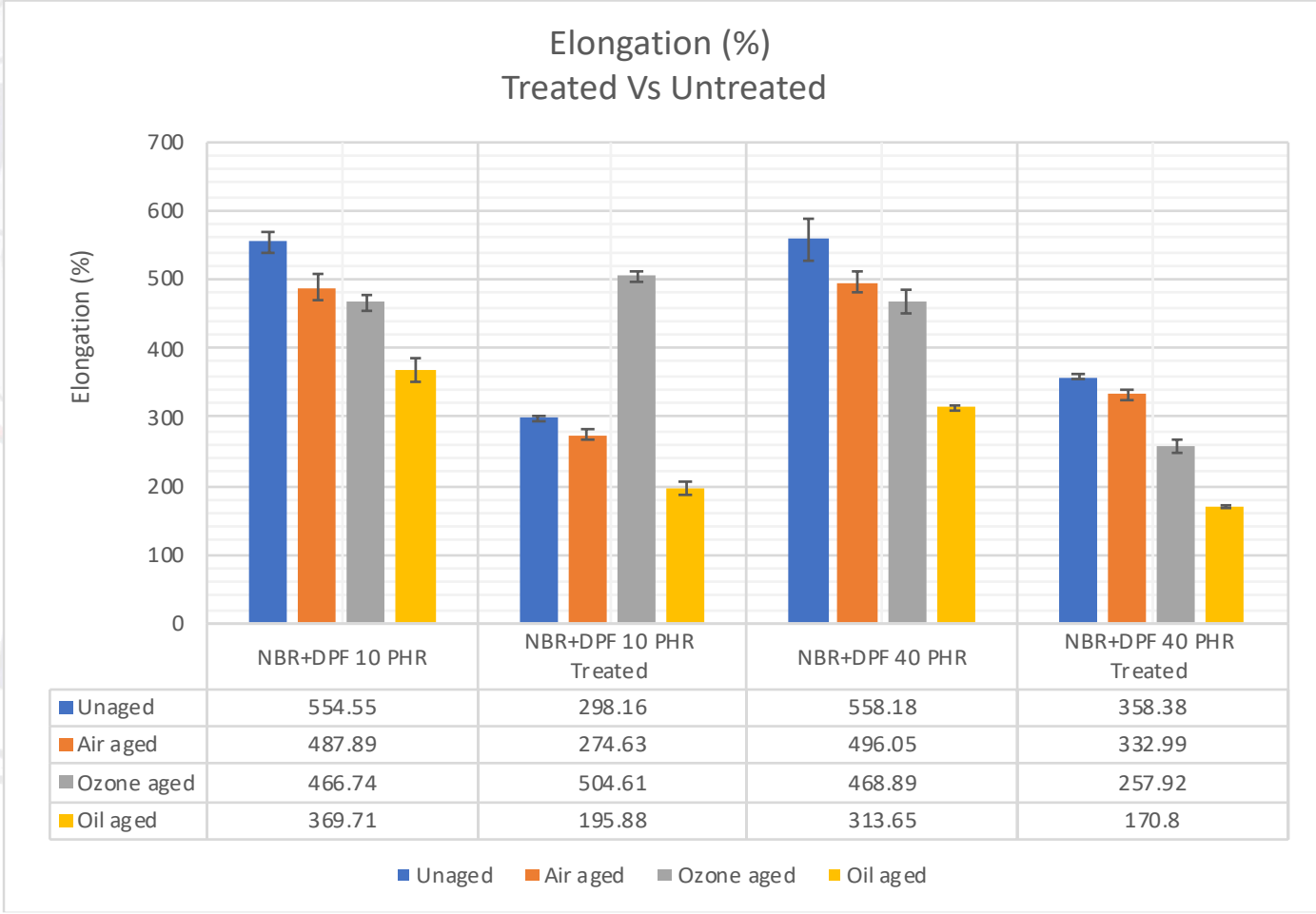
- Mechanical Results
  - Elongation:
    - Aged Elongation
    - Treated Vs Untreated



	NBR+DPF 10 PHR	NBR+DPF 10 PHR Treated	NBR+DPF 40 PHR	NBR+DPF 40 PHR Treated
Unaged	554.55	298.16	558.18	358.38
Air aged	487.89	274.63	496.05	332.99
Ozone aged	466.74	504.61	468.89	257.92
Oil aged	369.71	195.88	313.65	170.8

Results and Discussion

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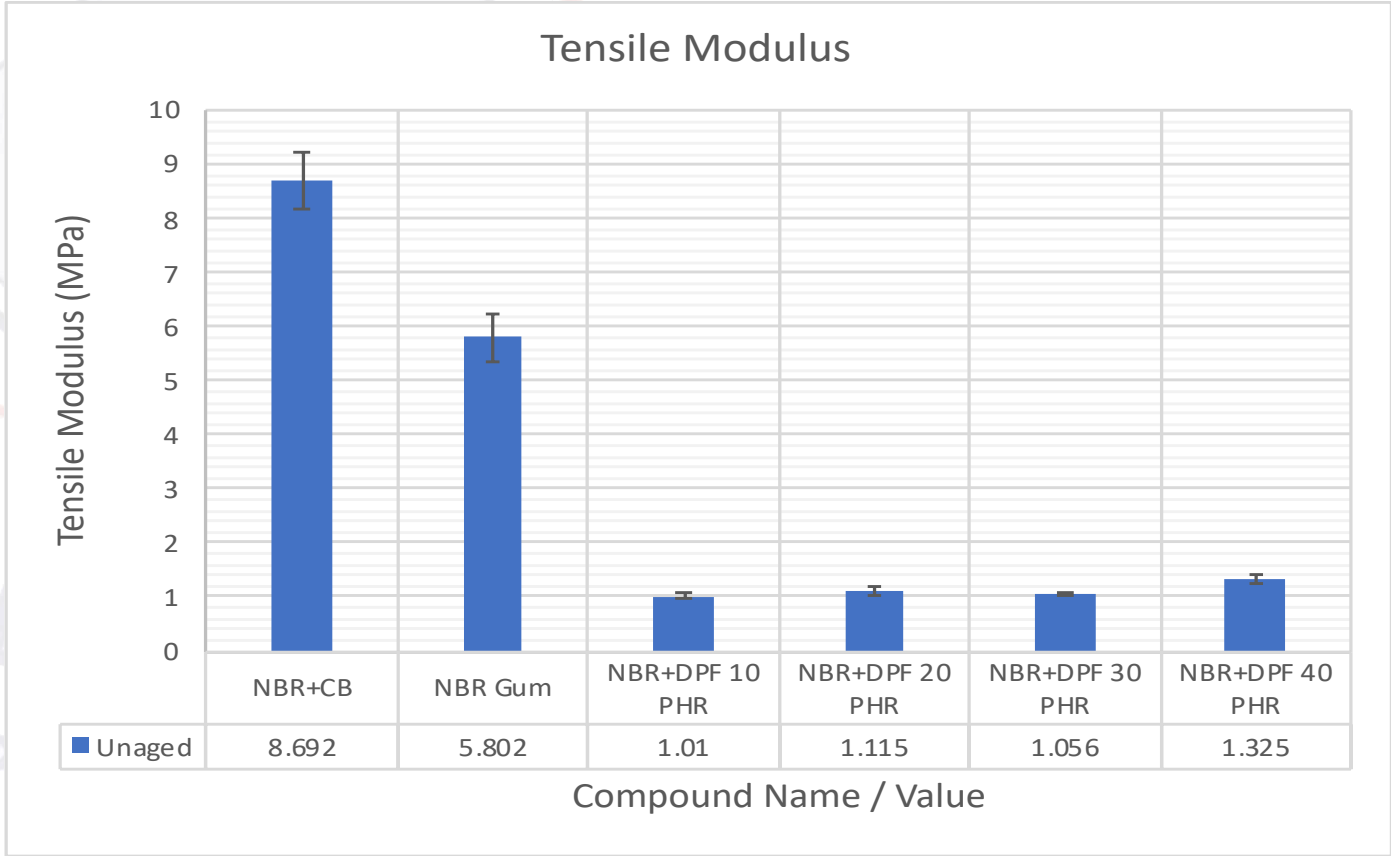
- Mechanical Results
  - Tensile Modulus :
    - Unaged Tensile Modulus



Results and Discussion

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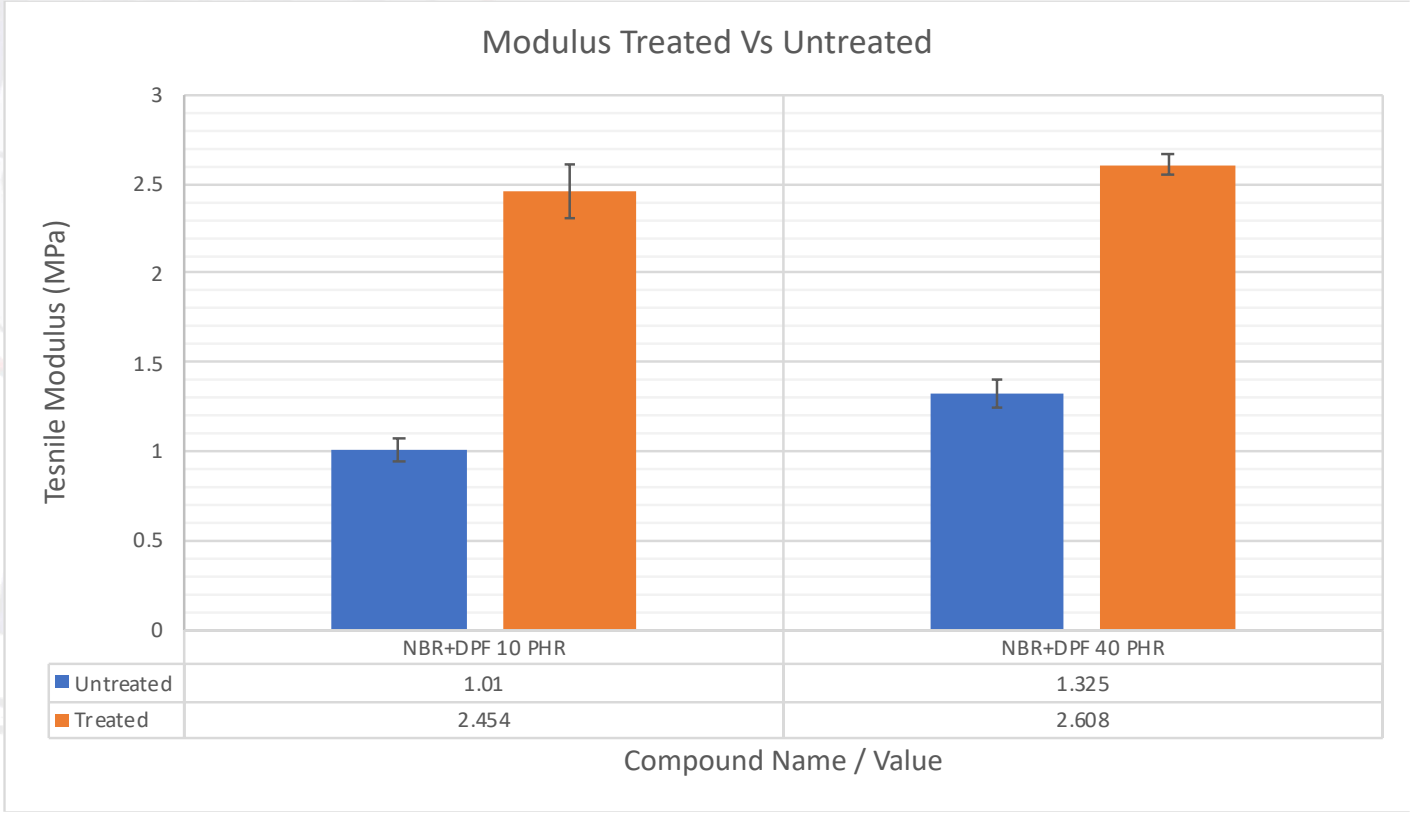
- Mechanical Results
  - Tensile Modulus :
    - Unaged Tensile Modulus
    - Treated Vs Untreated



Results and Discussion

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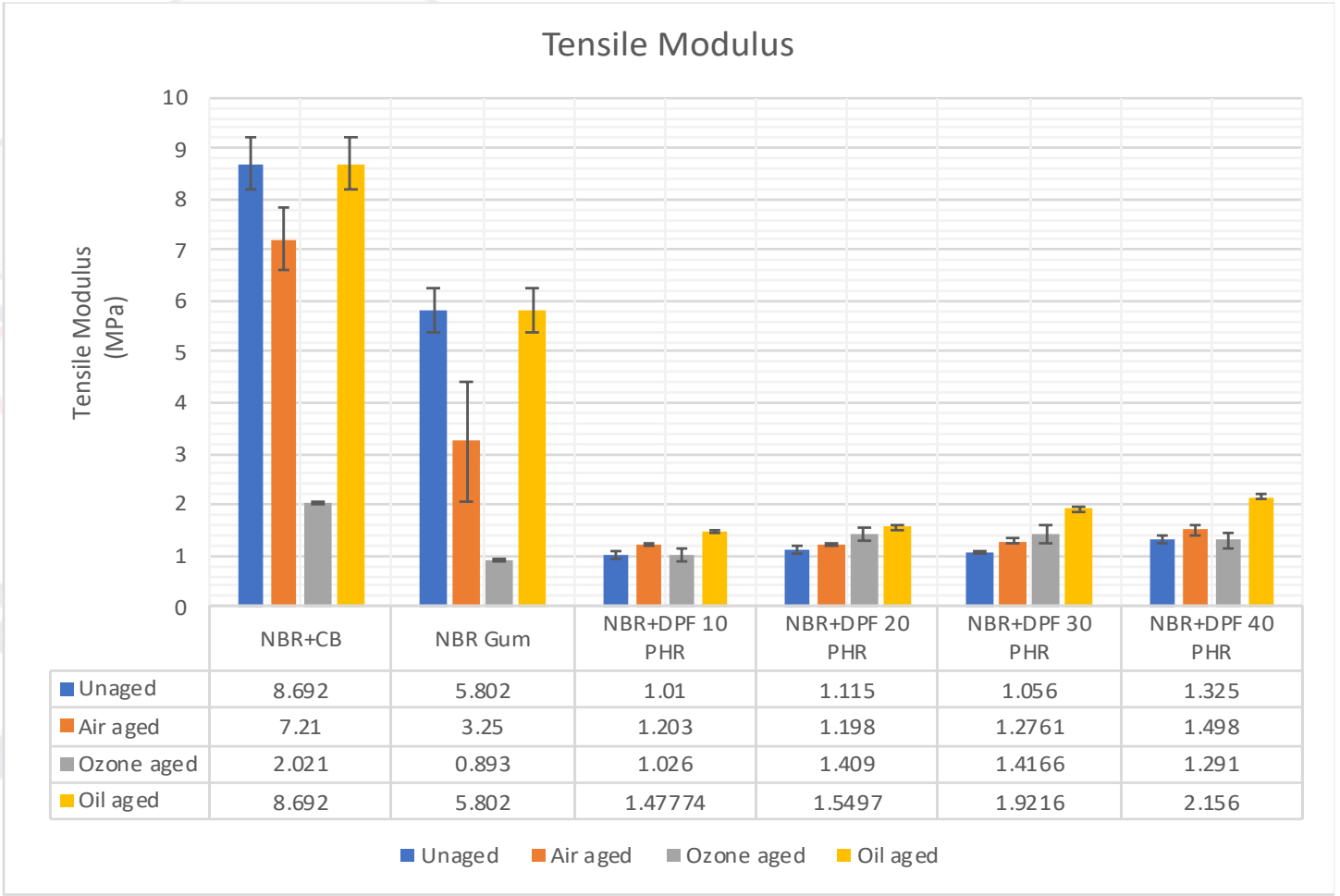
- Mechanical Results
  - Tensile Modulus :
    - Aged Tensile Modulus



	NBR+CB	NBR Gum	NBR+DPF 10 PHR	NBR+DPF 20 PHR	NBR+DPF 30 PHR	NBR+DPF 40 PHR
Unaged	8.692	5.802	1.01	1.115	1.056	1.325
Air aged	7.21	3.25	1.203	1.198	1.2761	1.498
Ozone aged	2.021	0.893	1.026	1.409	1.4166	1.291
Oil aged	8.692	5.802	1.47774	1.5497	1.9216	2.156

Results and Discussion

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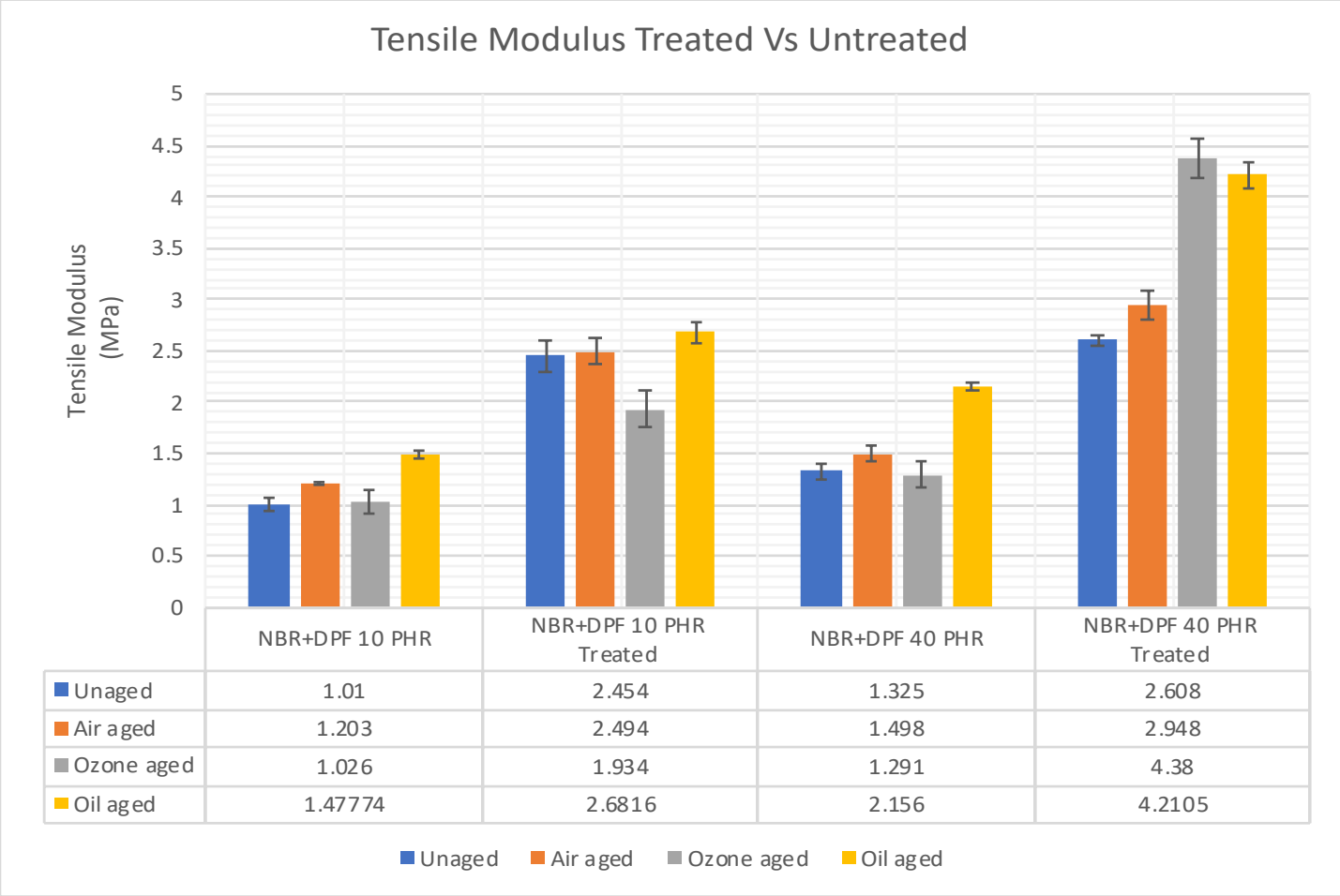
- Mechanical Results
  - Tensile Modulus :
    - Aged Tensile Modulus
    - Treated Vs Untreated



	NBR+DPF 10 PHR	NBR+DPF 10 PHR Treated	NBR+DPF 40 PHR	NBR+DPF 40 PHR Treated
Unaged	1.01	2.454	1.325	2.608
Air aged	1.203	2.494	1.498	2.948
Ozone aged	1.026	1.934	1.291	4.38
Oil aged	1.47774	2.6816	2.156	4.2105

Results and Discussion

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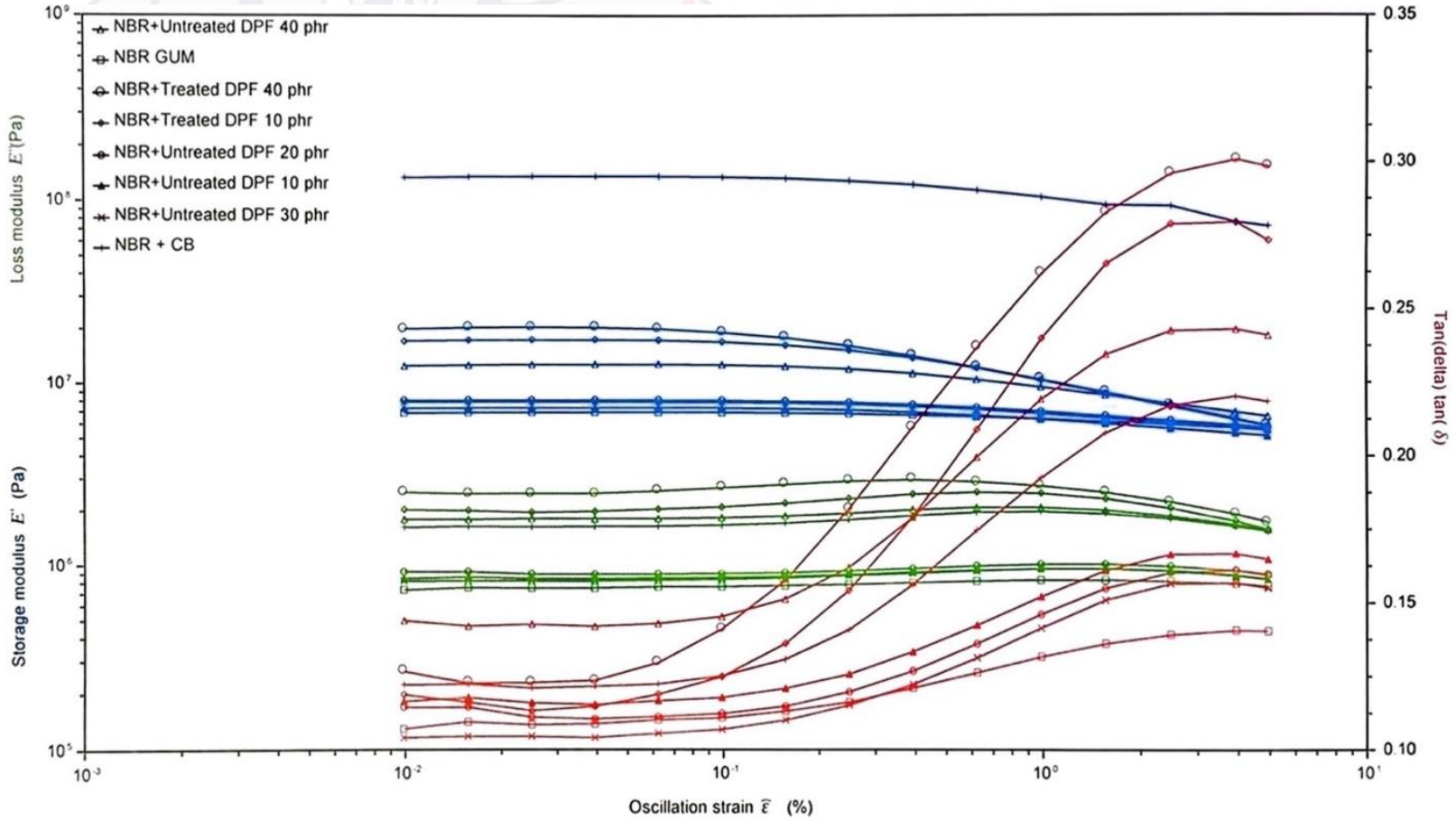
- Mechanical Results

  DMA



Results and Discussion

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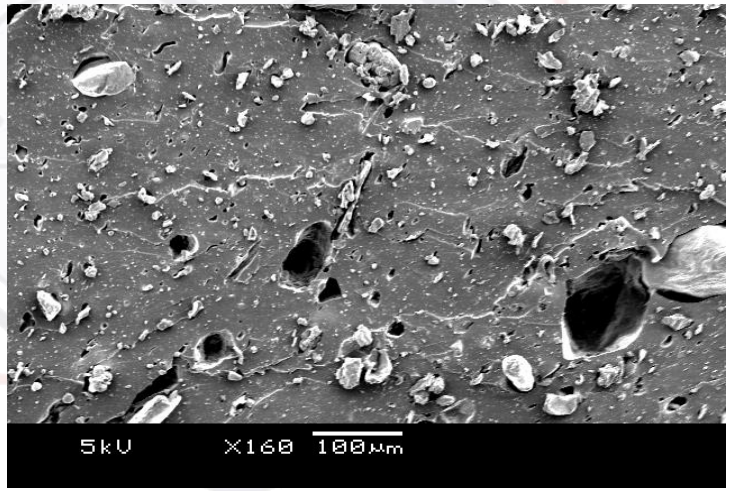
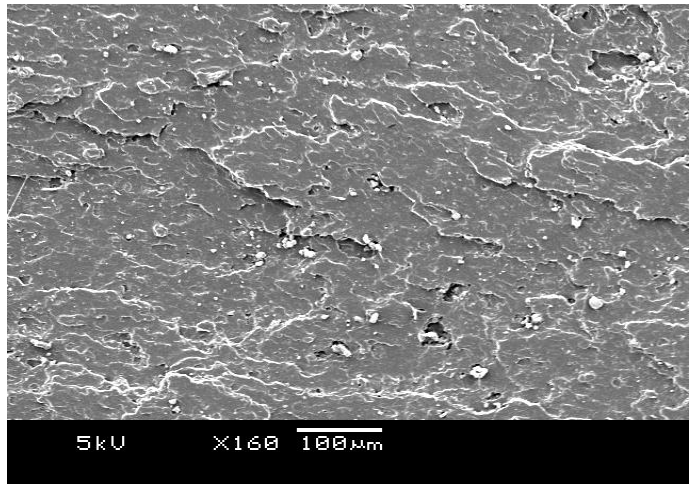
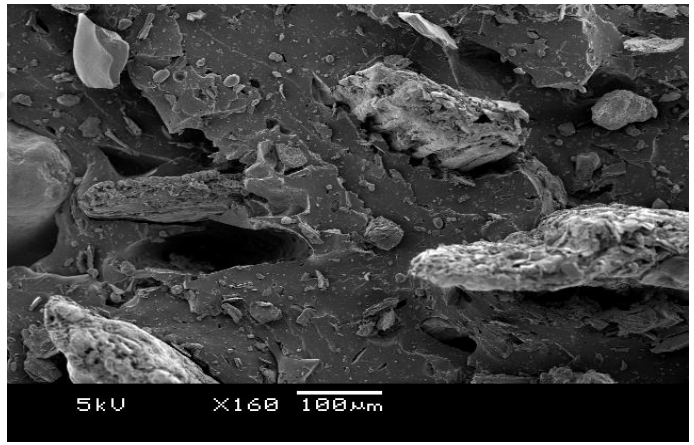
- Morphological Results:
  - SEM
    - *(A) NBR + Untreated DPF 10 PHR.*
    - *(B) Unfilled NBR.*
    - *(C) Untreated DPF 40 PHR.*
    - *(D) Treated 40 PHR*







Results and Discussion

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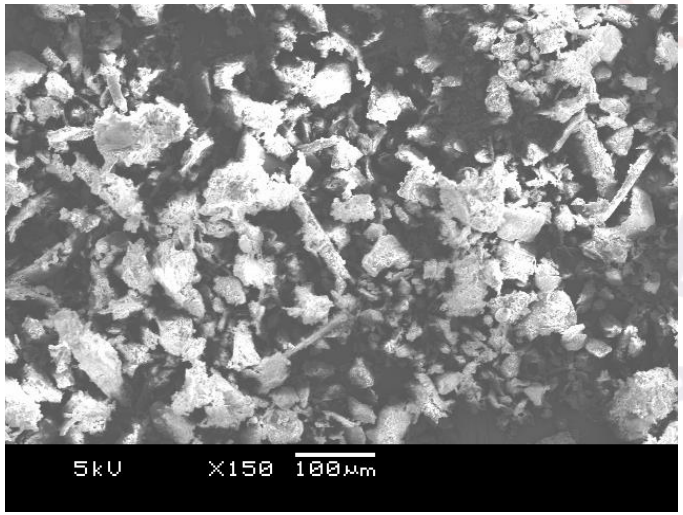
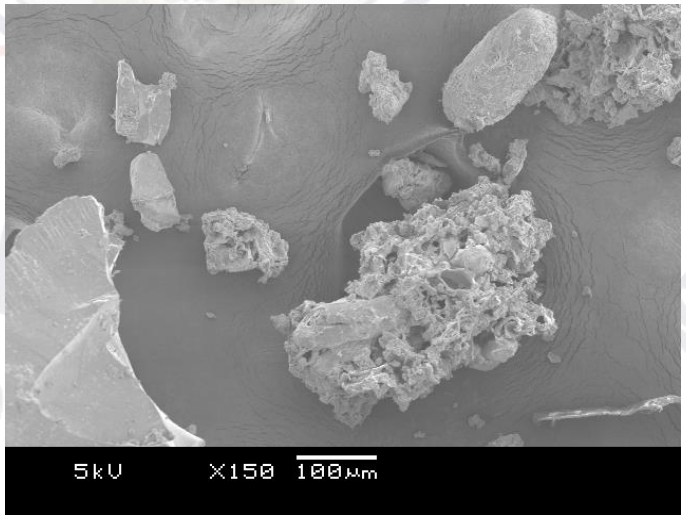
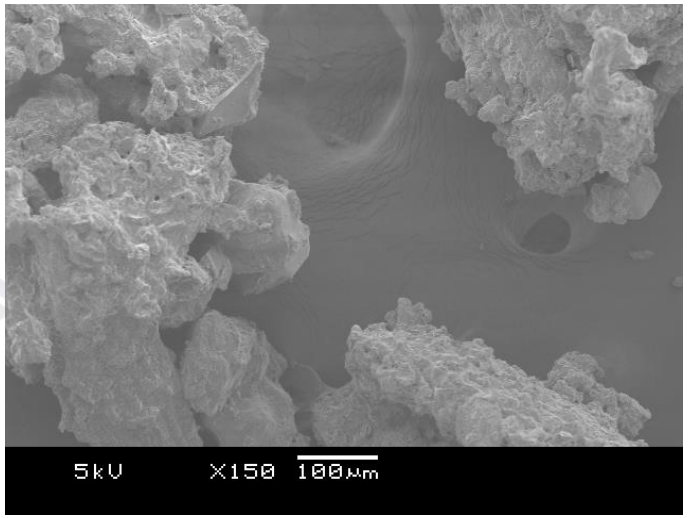
- Morphological Results:
  - SEM (fibers only)
    - A) Untreated DPF. B) NaOH Treated DPF. C) NaOH + Si69 Treated DPF.







Results and Discussion

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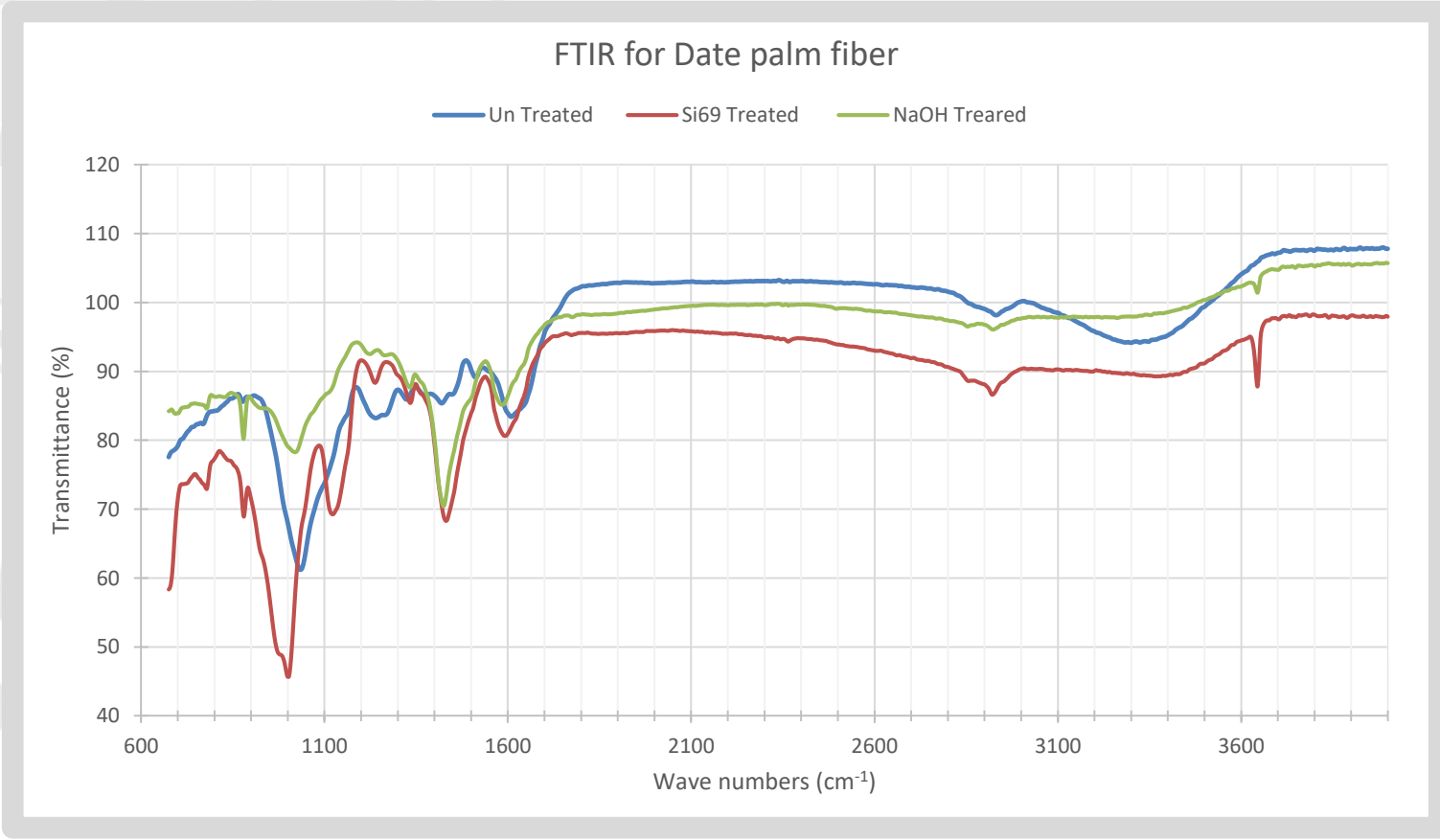
- Morphological Results:
  - FTIR (Fibers only)



Results and Discussion

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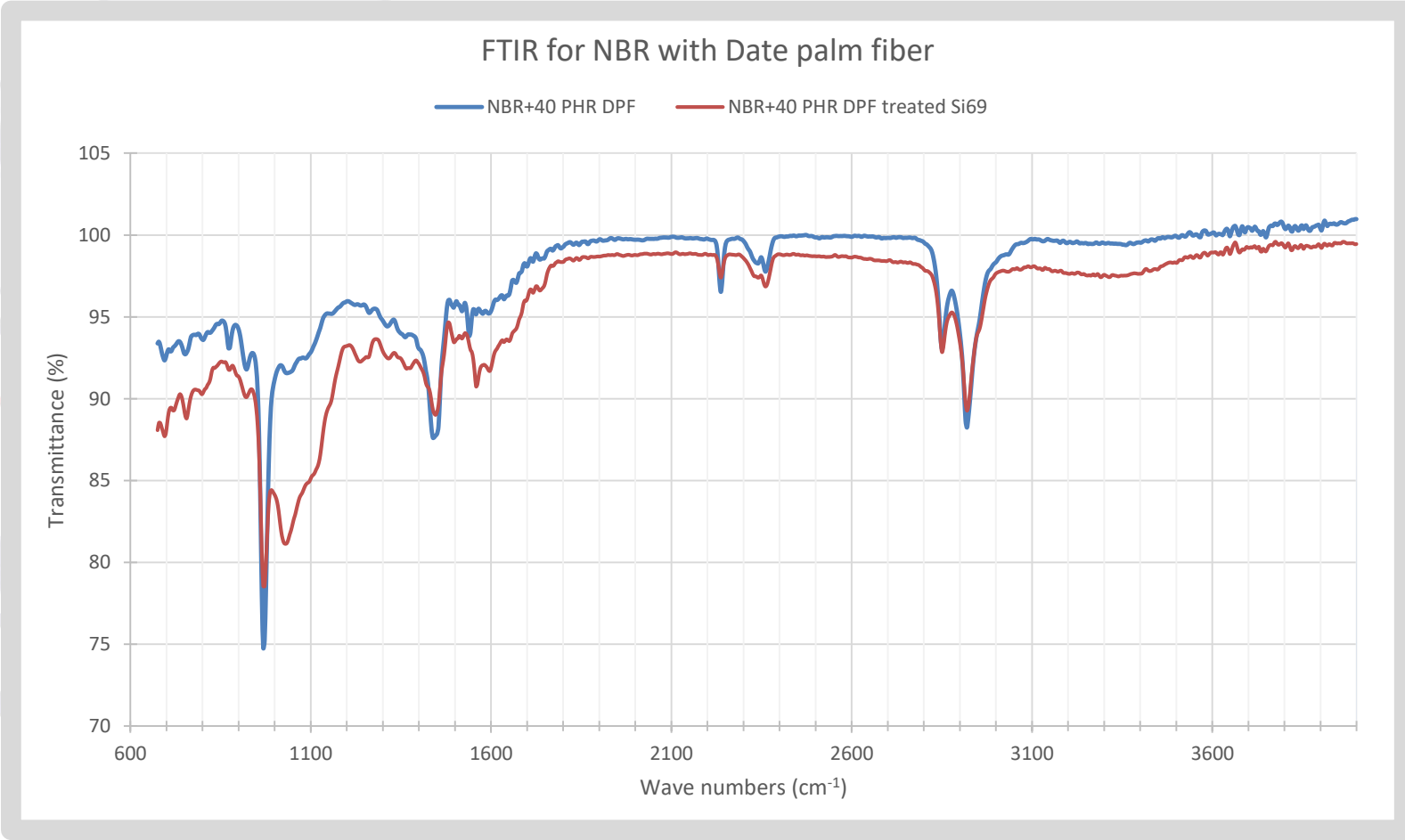
- Morphological Results:
  - FTIR



Results and Discussion

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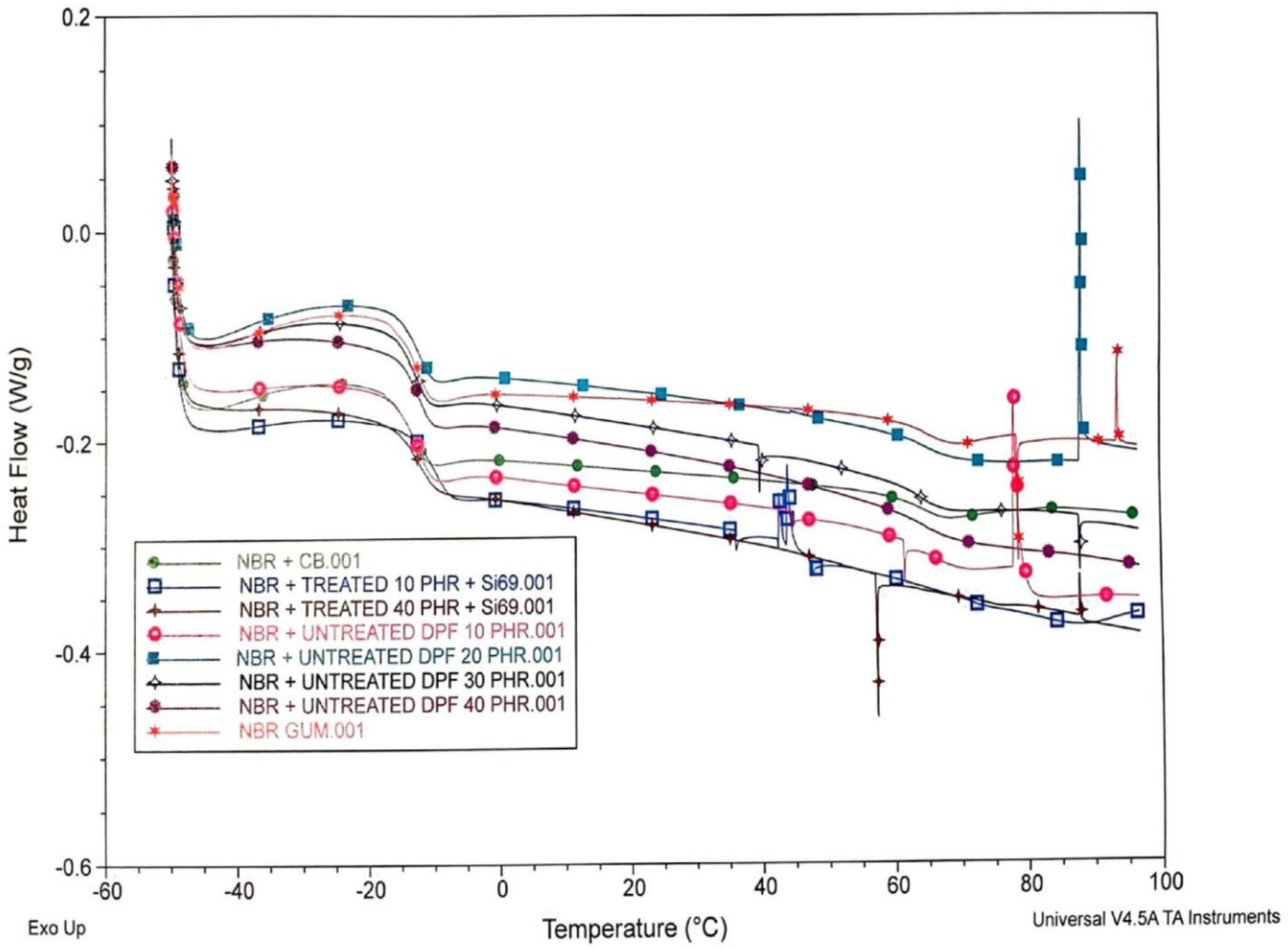
- Thermal Analysis Results :
  - DSC



Results and Discussion

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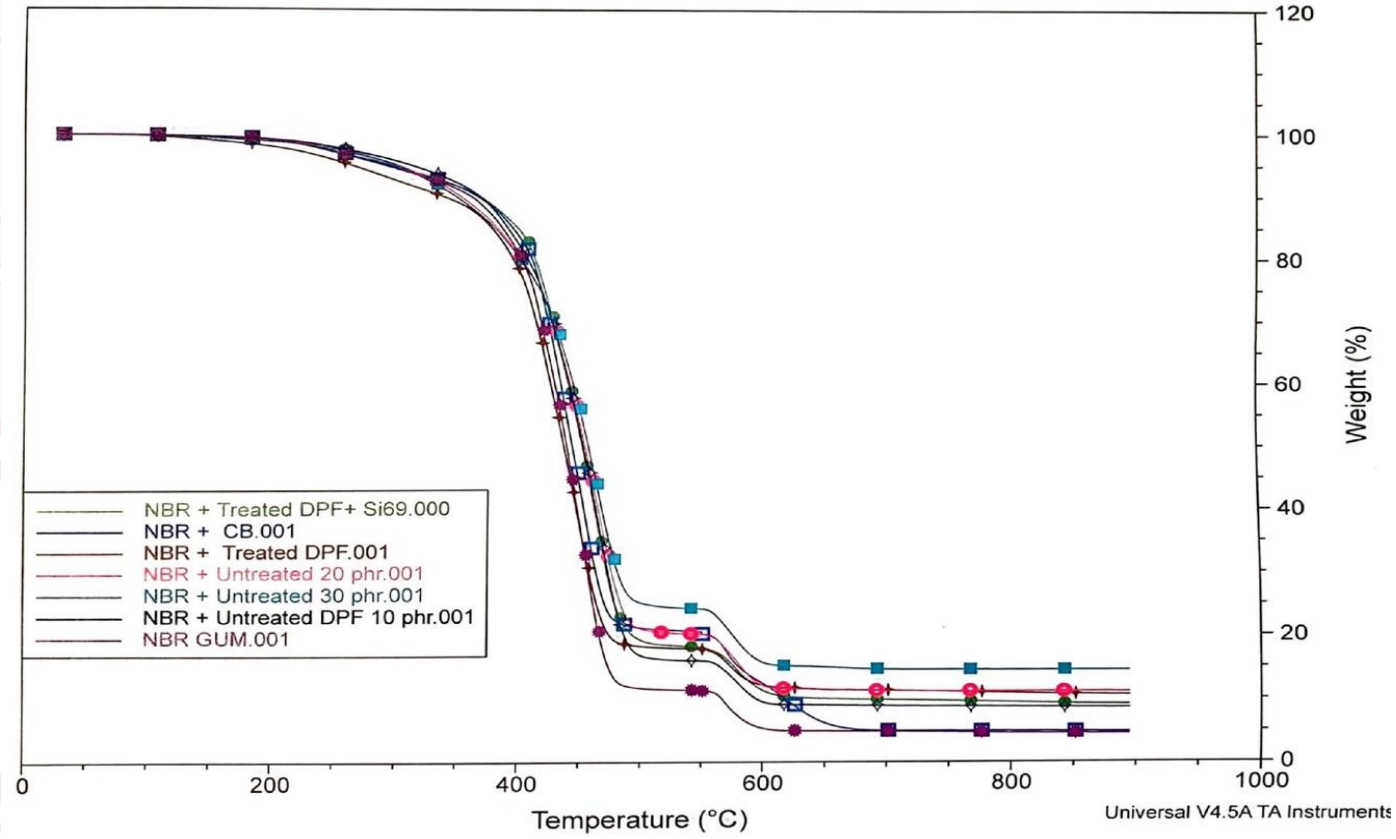
- Thermal Analysis Results :
  - TGA



Results and Discussion

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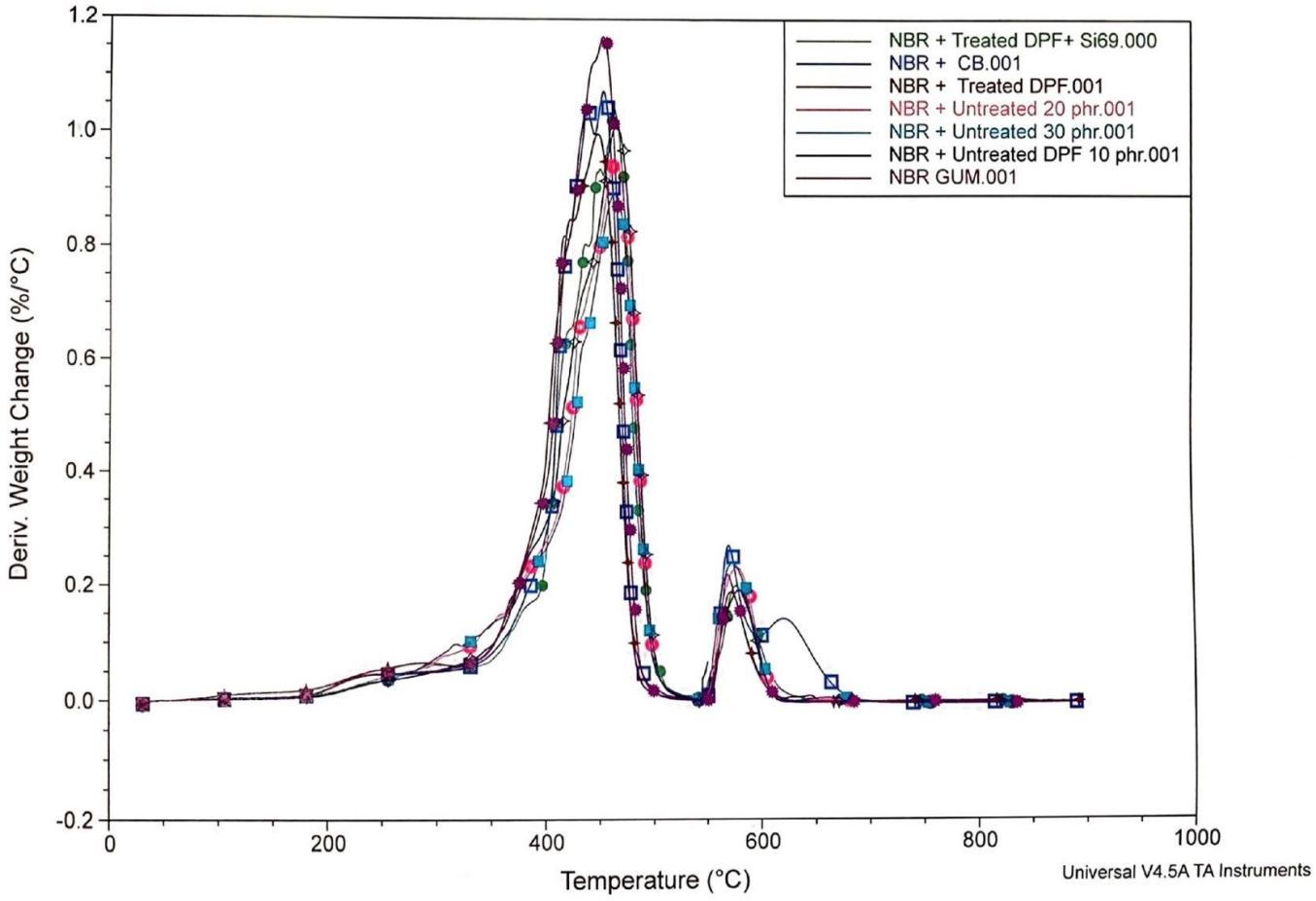
- Thermal Analysis Results :
  - DTGA



Conclusions

- Treating the fibers causes the cure time to increase significantly.
- treating the fibers resulted in an increase in the tensile strength
- Oil aging deteriorates the tensile strength of DPF/NBR composites, unlike hot air aging and ozone aging.
- Composites with treated fibers showed better mechanical properties than those with untreated fibers

Conclusions

- DPF/NBR composite exabits a pull out. Although the composite with treated fibers exhibites moderate interaction between the fibers and the rubber matrix.
- treating the fibers results in a small shift in the glass transition temperature.
- date palm fibers give a stability to the composite, increasing the degradation temperature.
- simple with untreated date palm fibers exabit a pull out. Where the treated fibers exhibited moderate interaction between the fibers and the rubber matrix.

Recommendations

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- Studying various types of fibers (Stem, leaves, bunch,..) instead of mixed fibers
- Improving the methods of fiber sizing.
- Investigating green method of fiber treatment.
- Exploring other methods of processing.

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