PRODUCTION OF pH-STABLE LIGNIN NANOPARTICLES IN HIGH YIELD VIA AN INNOVATIVE GREEN APPROACH AND THEIR APPLICATION IN THE DYING OF TEXTILES

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CONVERSION OF LIGNIN INTO LNPs

PROPERTIES ARE DRASTICALLY IMPROVED

SOLUBILITY

UV LIGHT ABSORPTION

ANTIOXIDANT ACTIVITY

HOMOGENOUS DISPERSION IN NANOCOMPOSITES

THERMAL STABILITY OF NANOCOMPOSITES

MECHANICAL PERFORMANCE OF NANOCOMPOSITES

REDUCED **WATER SENSITIVITY** OF NANOCOMPOSITES

BARRIER TO BLOCK THE PERMEABILITY OF GAS IN FILMS

HIGHLY DEPENDENT ON THE PRODUCTION METHOD!

THERMAL STABILITY: ↓ Yang et al., 2015 ↑ Nair et al. 2014

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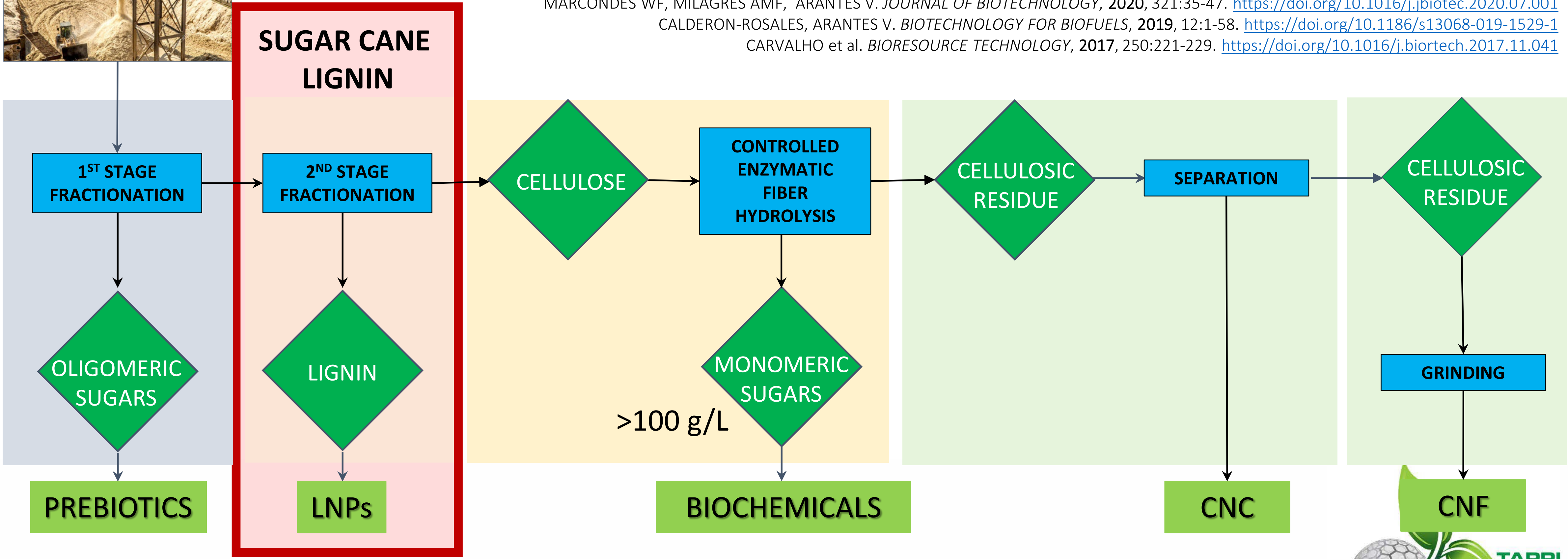
PEREIRA et al. *BIORESOURCE TECHNOLOGY*, **2021**, 342:125970. https://doi.org/10.1016/j.biortech.2021.125970
PEREIRA B and ARANTES V. *INDUSTRIAL CROPS AND PRODUCTS*, **2020**, 152:, 112377. https://doi.org/10.1016/j.indcrop.2020.112377
MARCONDES WF, MILAGRES AMF, ARANTES V. *JOURNAL OF BIOTECHNOLOGY*, **2020**, 321:35-47. https://doi.org/10.1016/j.jbiotec.2020.07.001
CALDERON-ROSALES, ARANTES V. *BIOTECHNOLOGY FOR BIOFUELS*, **2019**, 12:1-58. https://doi.org/10.1186/s13068-019-1529-1
CARVALHO et al. *BIORESOURCE TECHNOLOGY*, **2017**, 250:221-229. https://doi.org/10.1016/j.biortech.2017.11.041



PRODUCTION OF LNPs BY MECHANICAL REFINING

PROCESS SIMPLE & EFFICIENT

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PRODUCTION OF LNPs FROM SCB-LIGNIN

PROCESS SIMPLE & EFFICIENT

Table 1 Average particle size of sugarcane bagasse lignin (SCB-L) in an aqueous suspension (1% w/w) before (control) and after a different number of processing cycles in the disc ultra-refiner Supermasscolloider

Cycle	Ps_{10} [a] (μm)	Ps_{50} [b] (μm)	Ps_{90} [c] (μm)	Surface area (m^2 kg^{-1})	Zeta potential (mV)	Time (min)	Energy consumption (kW h kg^{-1})
Control	3.34	4.19	8.28	592	−27.5	0	0
1	0.012	0.018	0.035	16 500	−31.3	8.5	6
2	0.011	0.017	0.033	20 770	−32.1	17.1	11
3	0.011	0.017	0.033	22 270	−30.7	25.5	17
5	0.011	0.016	0.031	29 070	−30.4	42	28
10	0.011	0.016	0.031	28 900	−35.0	81.4	56

[a] PS_{10}: 10% of the particles have size equal or below the indicated value. [b] PS_{50} 50% of the particles have size equal or below the indicated value. [c] PS_{90} 90% of the particles have size equal or below the indicated value.

˜100 yield

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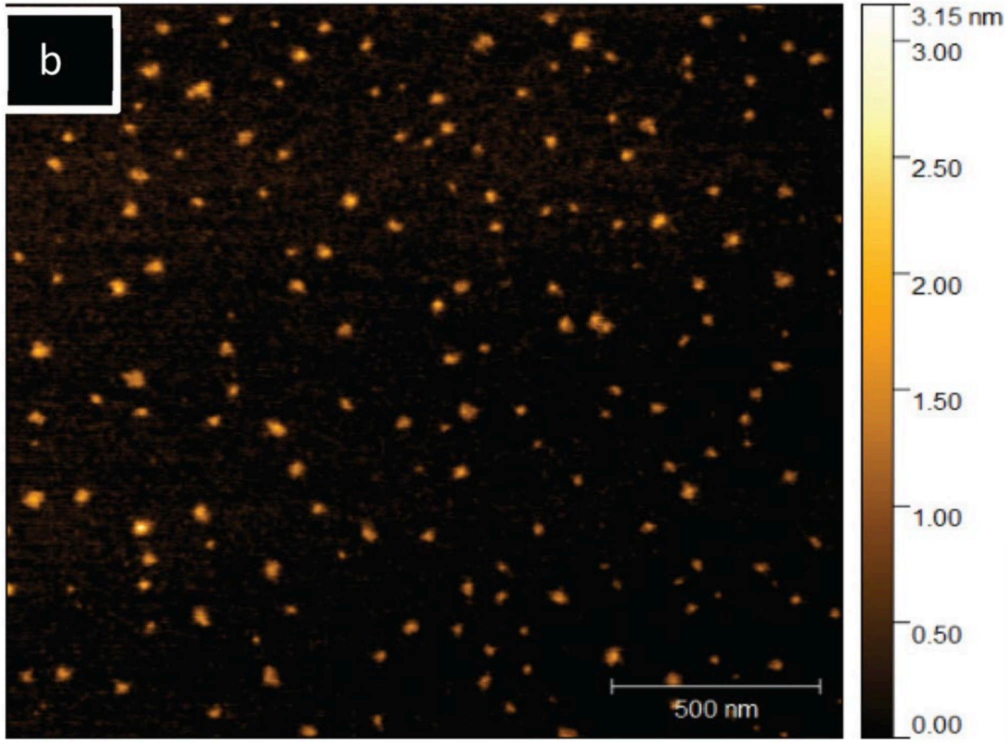
LNPs: MORPHOLOGY

SPHERICAL AND NANOMETRIC (AFM)



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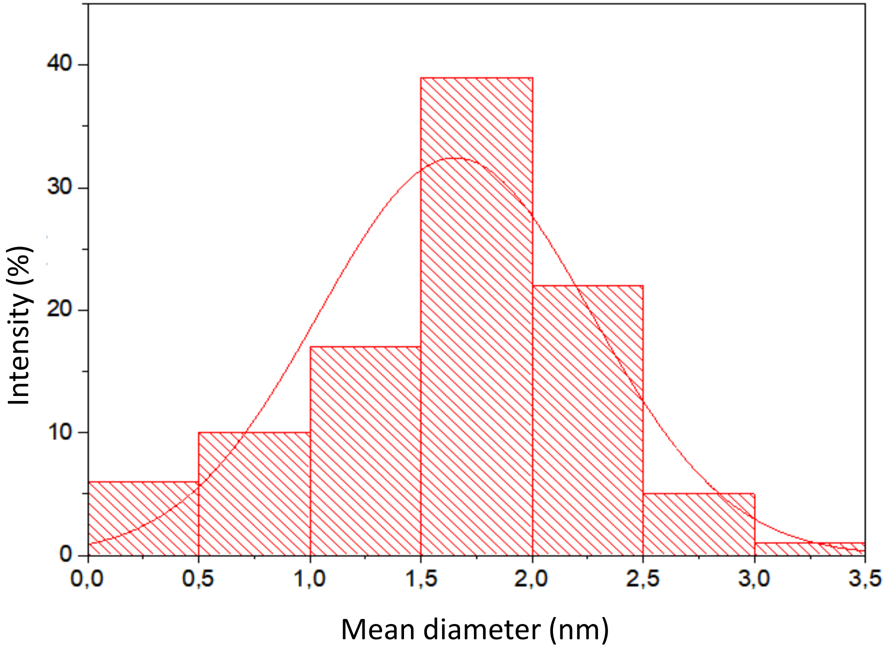


LNP: HIGHLY UNIFORM < 3,5 nm

LIGNIN: 1-100 μm

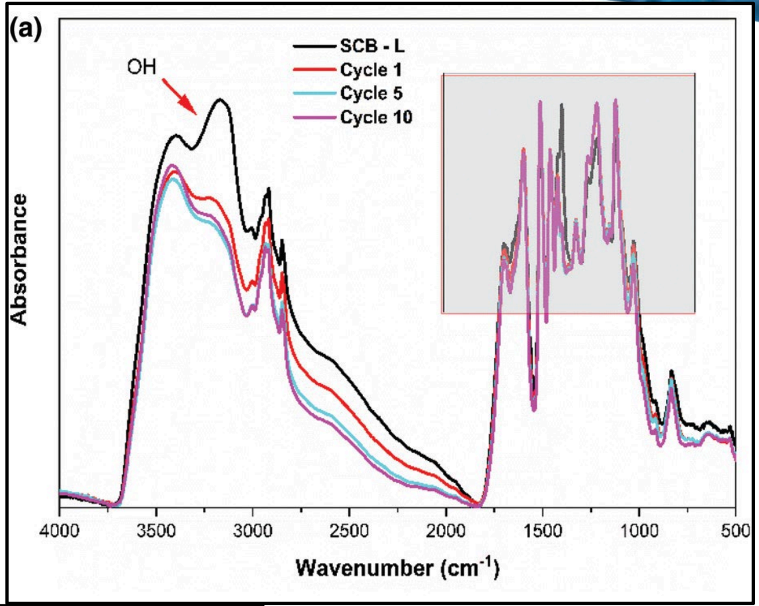
CHACTERIZATION OF SCB-LNPs

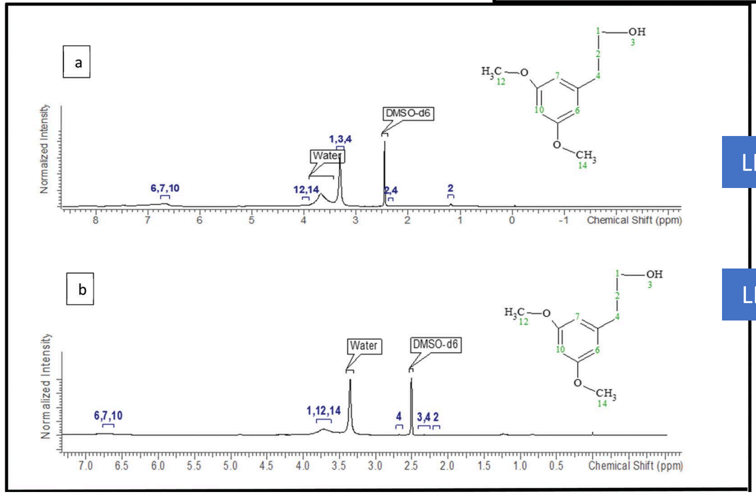
FT-IR & ^{1}H NMR

LIGNIN CHEMICAL BONDS (i.e, β-O-4) WERE DETECTED
NO SIGNIFICANT CHANGES WERE OBSERVED

INTENSITIES OF THE DIFFERENT FT-IR BANDS CHANGED
↓ HYDROXYL (3600–3200 and 1380 cm^{-1})
↑ C-O-C IN G AND S groups (1329–1130 cm^{-1})
↑ AROMATIC SKELETON (1599 and 1513 cm^{-1})

FT-IR + ^{1}H NMR
NO EVIDENCE OF CHEMICAL CHANGES
INTRINSIC PROPERTIES LIKELY MAINTAINED





LIGNIN

LNP-10C

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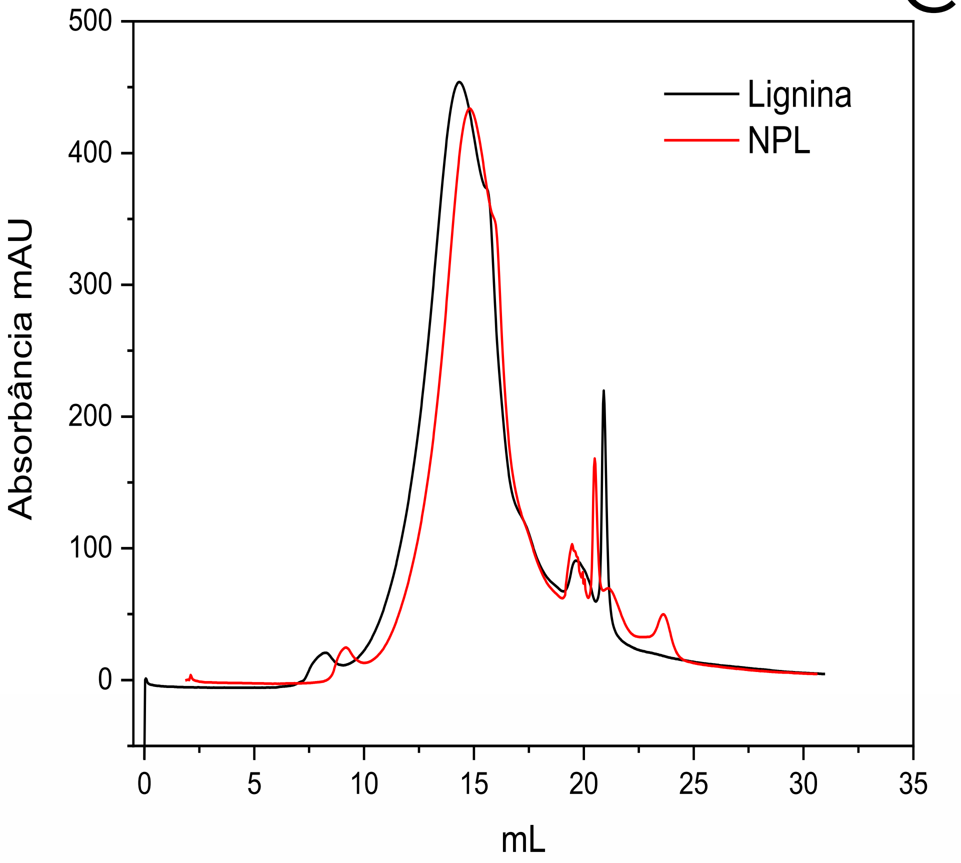
CHACTERIZATION OF SCB-LNPs

SEC



MW DISTRIBUTION OF THE LIGNIN MACROMOLECULES BEFORE AND AFTER PROCESSING WERE VERY SIMILAR (ONLY CHANGED SLIGHTLY)

NO EVIDENCE OF LIGNIN MOLECULES FRAGMENTATION

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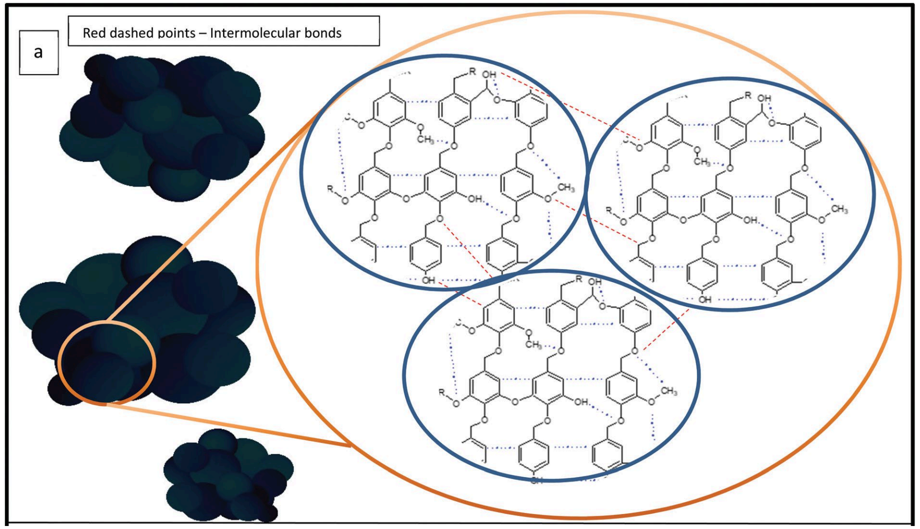
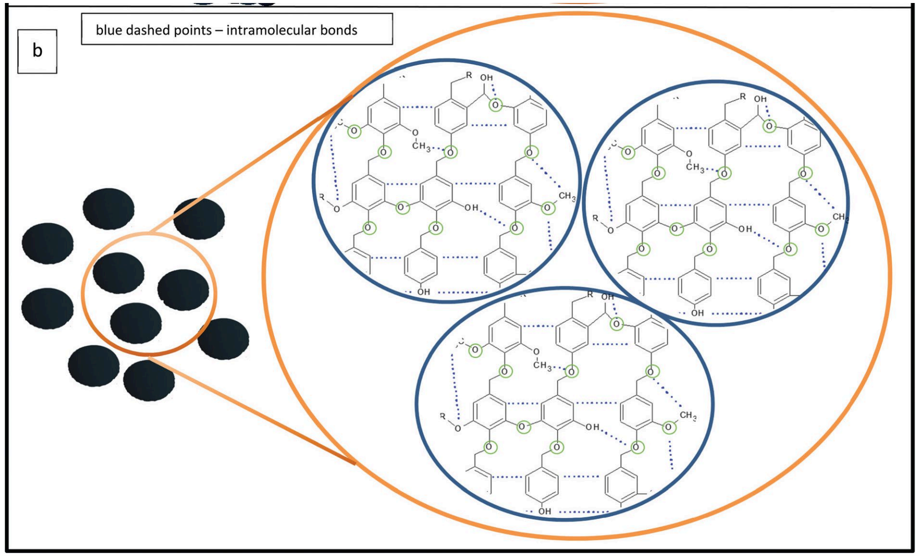
CHACTERIZATION OF SCB-LNPs

BREAKING OF WEAK INTERACTIONS
(i.e., HYDROGEN BONDS & van der WAALS)

REDUCTION IN SIZE OF LIGNIN CLUSTERS

INTRISIC PROPERTIES LIKELY MAINTAINED





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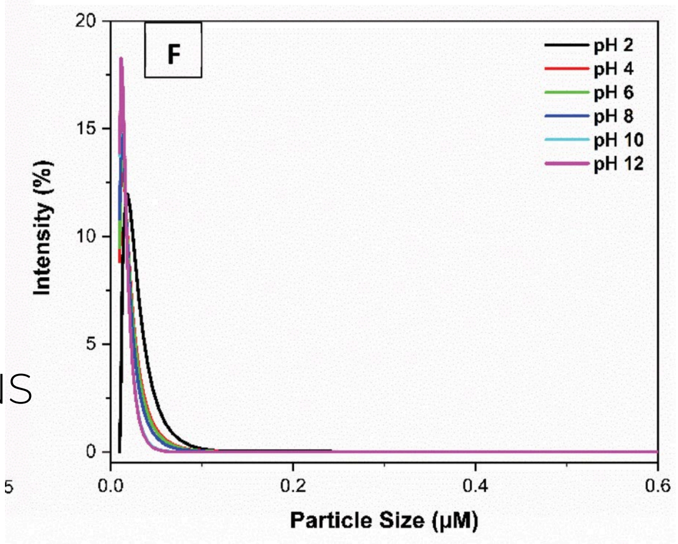
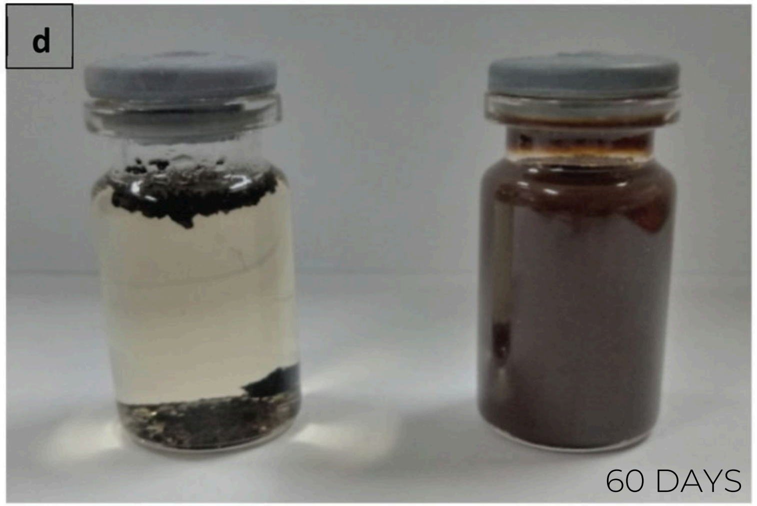
PROPERTIES OF SCB-LNPs - STABILITY

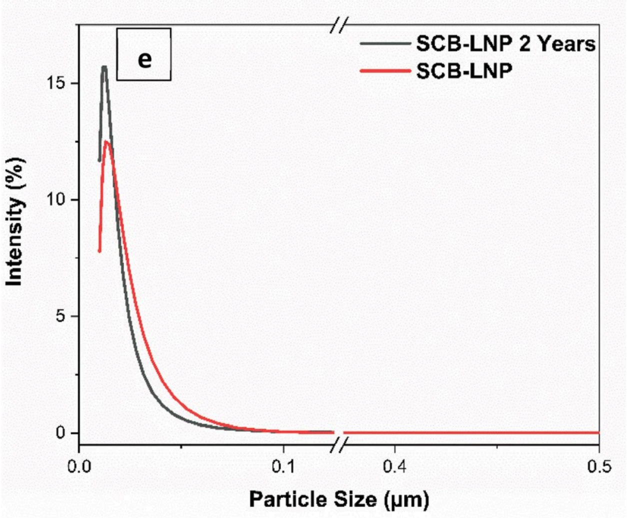
HIGH SUSPENSION STABILITY
INCREASED ZETA POTENTIAL

LONG-TERM STABILITY IN AQUEOUS MEDIUM (2 YEARS)
NO CHEMICAL CHANGED TO THE STRUCTURE (CONFIRMED BY ^{13}C NMR)

REMAINED STABLE OVER A BROAD pH RANGE, EVEN UNDER ACIDIC CONDITIONS







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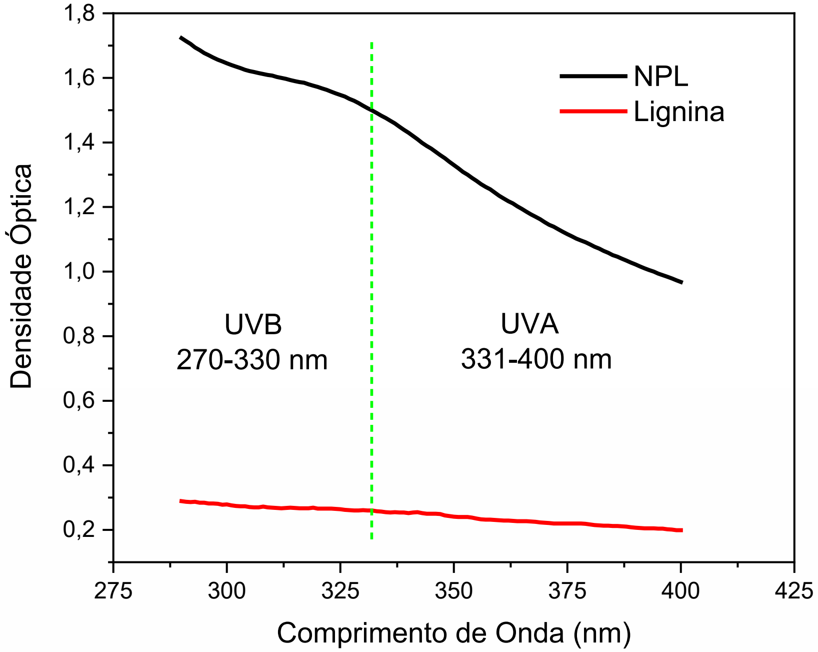
PROPERTIES OF SCB-LNPs
UV ABSORPTION

HIGHER ABSORPTION TOWARD UV-A (320-400 nm) and UV-B (290-320 nm)

INCREASE IN ~900%



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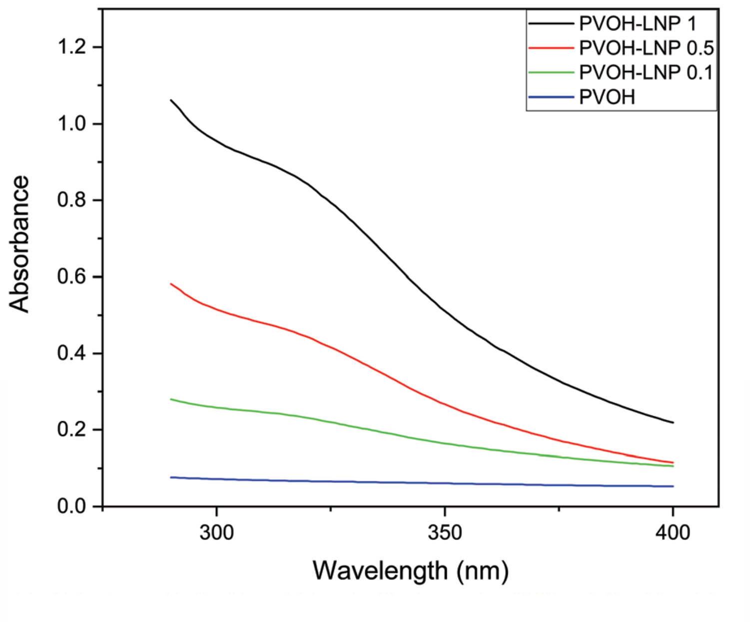
PROPERTIES OF SCB-LNPs - NANOCOMPOSITES

HIGH HOMOGENEITY OF THE PVOH-LNPs COMPOSITE FILMS

UV-SHIELDING OF PVOH-LNP NANOCOMPOSITE

UP TO 10X MORE ADSORION WITH LNPs



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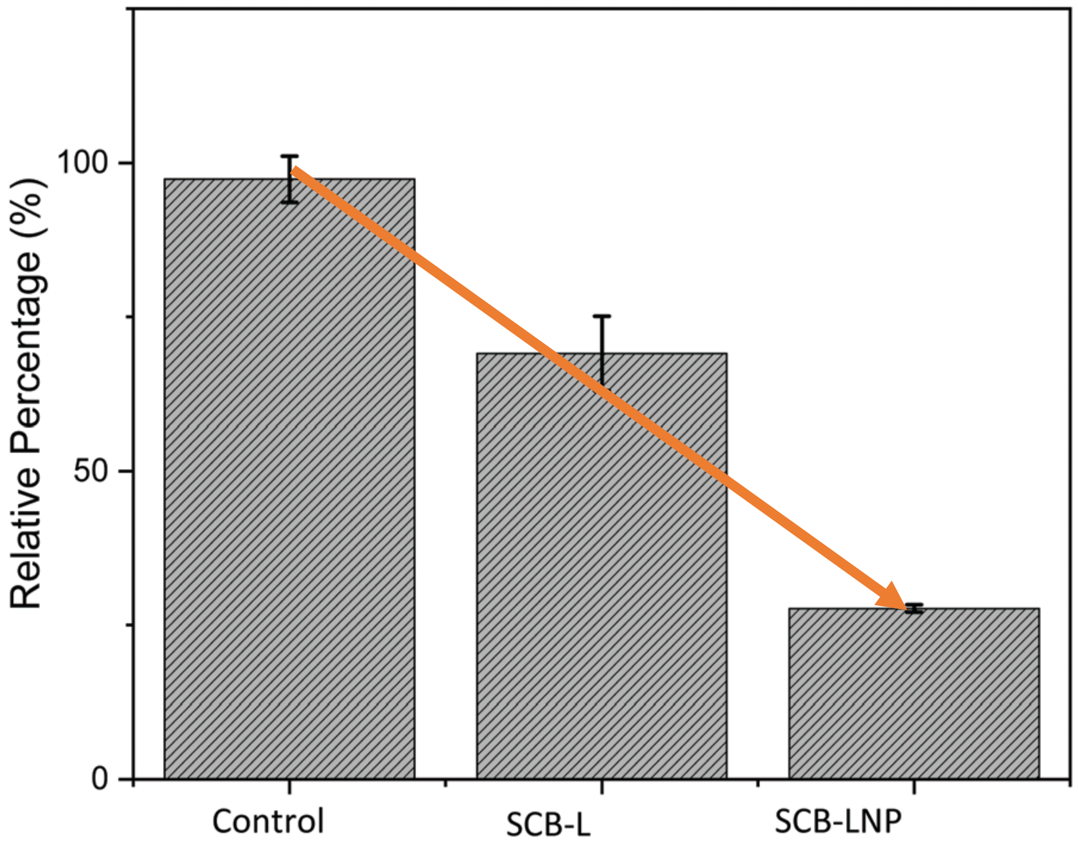
FILM THICKNESS: 0.3-0.6 mm

PROPERTIES OF SCB-LNPs

ANTIMICROBIAL ACTIVITY



GRAM-NEGATIVE *E. coli* K12711 (OPPORTUNISTIC PATHOGEN)

ANTIBACTERIAL ACTIVITY WAS DRASTICALLY ENHANCED

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PRODUCTION OF LNPs FROM KRAFT-LIGNIN

PROCESS: FEEDSTOCK AGNOSTIC

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PRODUCTION OF LNPs FROM KRAFT-LIGNIN

PROCESS: FEEDSTOCK AGNOSTIC

Table 5 Average particle size, surface area, processing time, and energy consumption of Kraft pulp lignin in an aqueous suspension (1% w/w) during processing in the disc ultra-refiner Supermasscolloider for a different number of cycles. 1% (w/w) suspension of Kraft pulp lignin before processing was used as the control

Cycle	Ps_{10} [a] (μm)	Ps_{50} [b] (μm)	Ps_{90} [c] (μm)	Surface area (m^2 kg^{-1})	Time (min)	Energy consumption (kW h kg^{-1})
Control	1.35	1.80	5.70	182.7	0	0
1	0.360	0.811	1.98	2152	5	4
2	0.242	0.356	1.35	2690	10.2	8
3	0.192	0.274	0.938	3528	15.21	12
5	0.154	0.221	0.526	4342	32.6	19
10	0.102	0.152	0.287	7912	55.2	36

[a] PS_{10}: 10% of the particles have size equal or below the indicated value. [b] PS_{50} 50% of the particles have size equal or below the indicated value. [c] PS_{90} 90% of the particles have size equal or below the indicated value.

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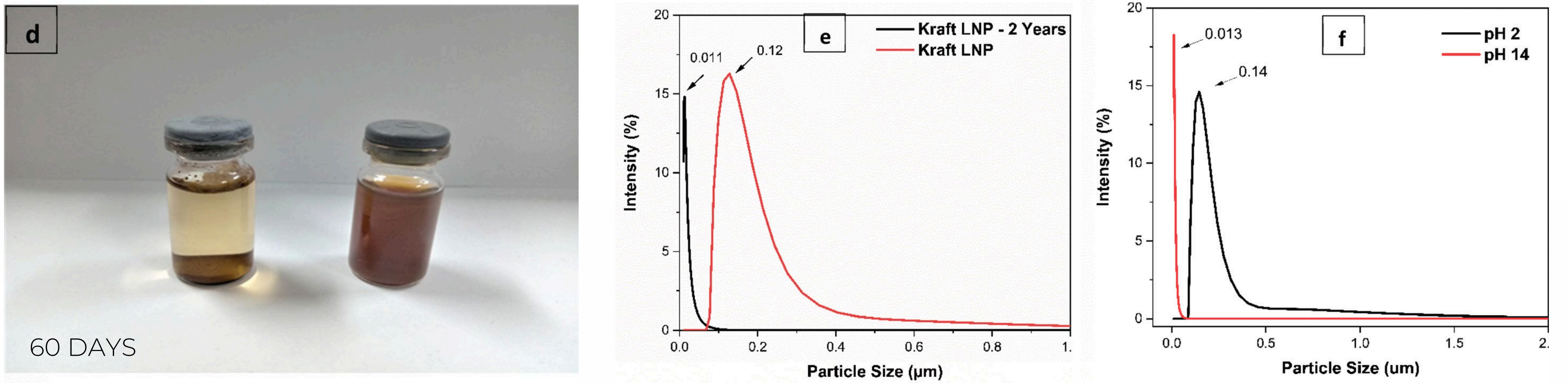
REMAINED STABLE OVER A BROAD pH RANGE, EVEN UNDER ACIDIC CONDITIONS



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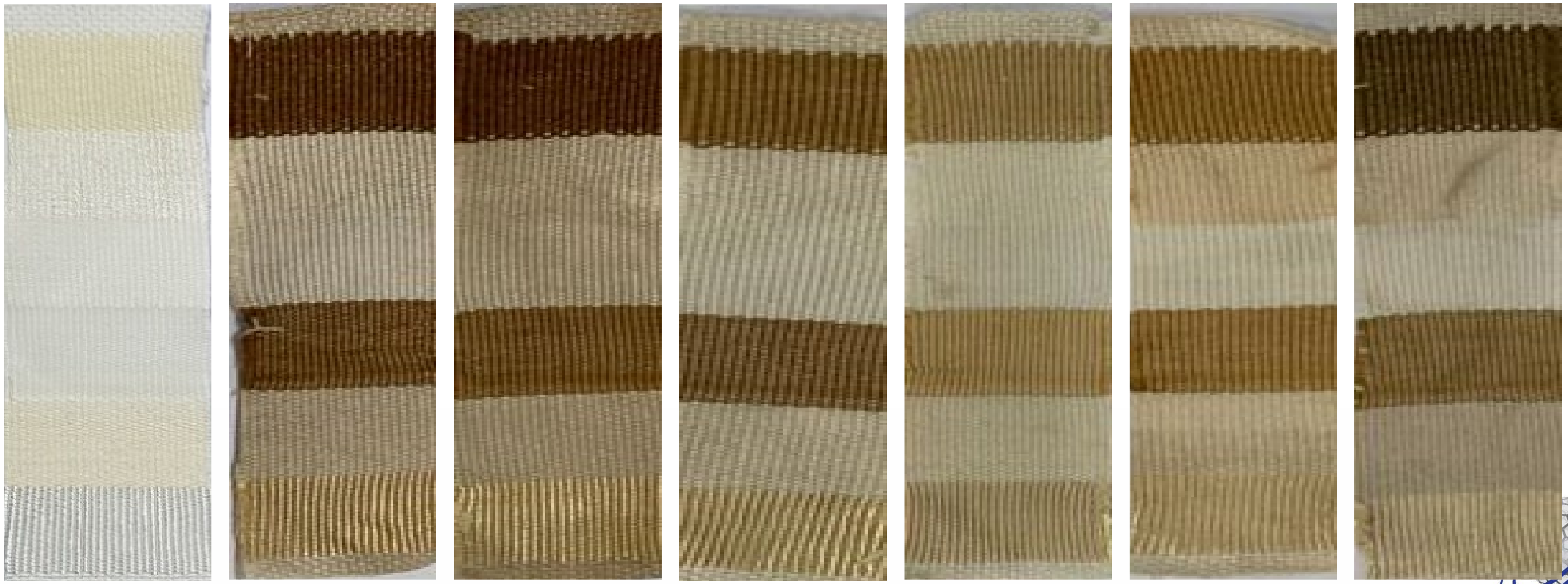
TEXTILES COATED WITH LNPs

Multifibers | pH 3,87* | pH 3.0 | pH 7.0 | pH 11.0 | Aluminum potassium sulfate | Iron (II) sulfate

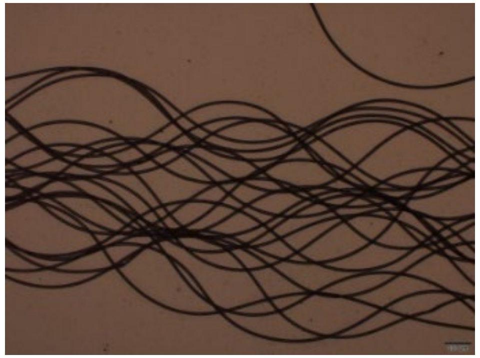


TEXTILES COATED WITH LNPs

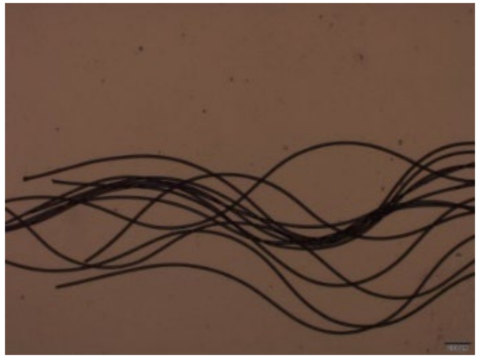
TEXTILES COATED WITH LNPs

Polyamide BF

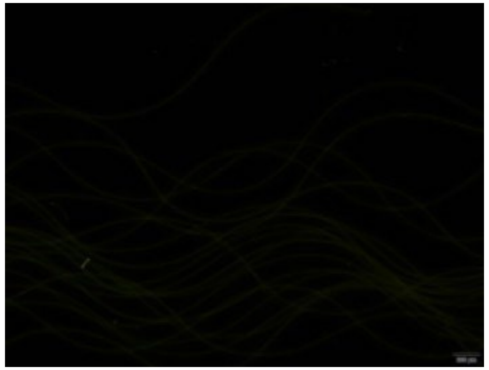
Polyamide
LNP-75-3C BF



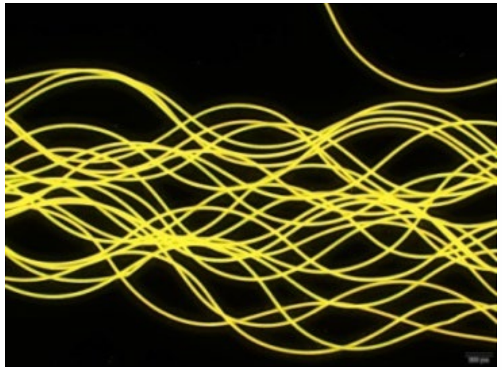
Polyamide
LNP-185-5C BF



Polyamide UFBW



Polyamide
LNP-75-3C UFBW



Polyamide
LNP-185_5C UFBW

PROPERTIES OF TEXTILE FIBERS COATED WITH LNPs

Before and after "washing"

ANTIMICROBIAL FABRIC TEST

AATCC 100-2004

COLOUR FASTNESS TO DOMESTIC AND COMMERCIAL LAUNDERING

ISO 105-C06:2010 – *Textiles – Tests for colour fastness – Part C06: Colour fastness to domestic and commercial laundering*.

COLOUR FASTNESS TO PERSPIRATION

ISO 105-E04:2013 – Textiles – Tests for colour fastness – Part E04: Colour fastness to perspiration

TENSILE PROPERTIES

ASTM D3822 (2007)

ULTRAVIOLET PROTECTION FACTOR

Sun Protective Clothing – Evaluation and Classification – AS/NZS 4399-2017
Clothing - Ultraviolet Protection Factor - Requirements and test methods – ABNT NBR 16695:2018

ANTIMICROBIAL ACTIVITY IN TEXTILES COATED WITH LNPs

Staphylococcus aureus

UPF OF THE TEXTILE FIBERS COATED WITH LNPs

TEXTILES	BEFORE WASHING		AFTER WASHING[1]	
	UPF[2,3]	UV PROTECTION CATEGORY[1,2]	UPF[2,3]	UV PROTECTION CATEGORY[1,2]
COTTON	-	-	-	-
COTTON-LNPs	25 – 39	VERY GOOD	25 – 39	VERY GOOD

[1]Textiles – Tests for colour fastness – Part C06: Colour fastness to domestic and comercial laundering – ISO 105-C06:2010
[2]Sun Protective Clothing-evaluation and Classification – AS/NZS 4399-2017
[3]Clothing - Ultraviolet Protection Factor - Requirements and test methods – ABNT NBR 16695:2018

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TAKE-HOME MESSAGES

DISC ULTRA-REFINING AS GREEN TECHNOLOGY

- PRODUCTION OF LNPs IN HIGH YIELD
- FEEDSTOCK AGNOSTIC, DOES NOT ADD HETEROGENEITY,
- HIGH UNIFORMITY, HIGH SOLUBILITY, HIGHLY pH-STABLE, REDISPERSIBILITY

APPLICATION OF LIGNIN NANOPARTICLES

NANOCOMPOSITES

TEXTILE FABRICS

- HIGH HOMOGENEITY COATING
- ANTIMICROBIAL ACTIVITY
- INCREASED UV PROTECTION (UPF)

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