Physicochemical Properties of Cellulose Nanocrystals Extracted from Postconsumer Polyester/Cotton-Blended Fabrics

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Background

- World fibre production is estimated at 111 million tonnes per annum.(EPA 2022)
- On average Europeans use nearly 26 kilos of textiles and discard about 11 kilos of them every year. (EPA 2022)
- 21% is recycled while the rest is landfilled or incinerated.(Vanzetto et al., 2021)
- Recycling is mostly downcycling to low value products such as insulation, mattress stuffing or even rugs.
- Most textile wastes comes in blends which makes recycling to original fibres difficult, currently less than 1% is recycled back to textile applications. (Kahoush & Kadi, 2022)

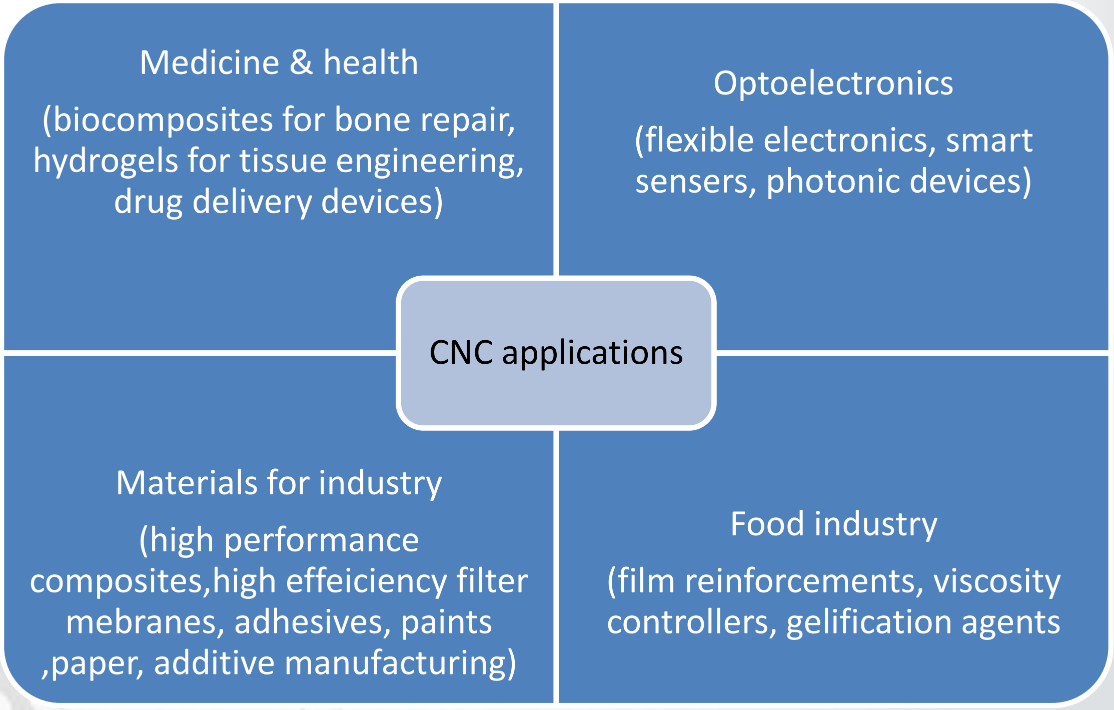
- Landfilling space, possibility of drainage blocking
- Release of microplastics
- Water pollution from dyes
- Resource inefficiency

Background

- Utilizing cotton-polyester waste for the extraction of cellulose nanocrystals is a viable option.
- Sustainable solution to address textile waste accumulation while simultaneously producing a valuable material.
- CNCs- highly crystalline cellulose nanomaterials derived from various cellulose sources –wood, cotton, agricultural residue.
- Cotton produces the highest purity CNCs
- Limited raw materials a challenge- redirect waste as a raw material source
- Array of applications depending on the grade and properties
- Vital to the pharmaceutical, textile and food manufacturing industry

Background

- Production of CNC require pure forms of cellulose
- CNCs properties allow them to be applied on various industries;
  - Biocompatibility
  - Biodegragability
  - Colloidal stability
  - High surface area
  - High mechanical strength
  - Optical properties
  - transparency



Aim & Objectives

- The main aim of this study is to examine the properties of cellulose nanocrystals (CNCs) obtained from post-consumer polyester-cotton waste and assess the effect of different fabric structures on the extraction and these properties

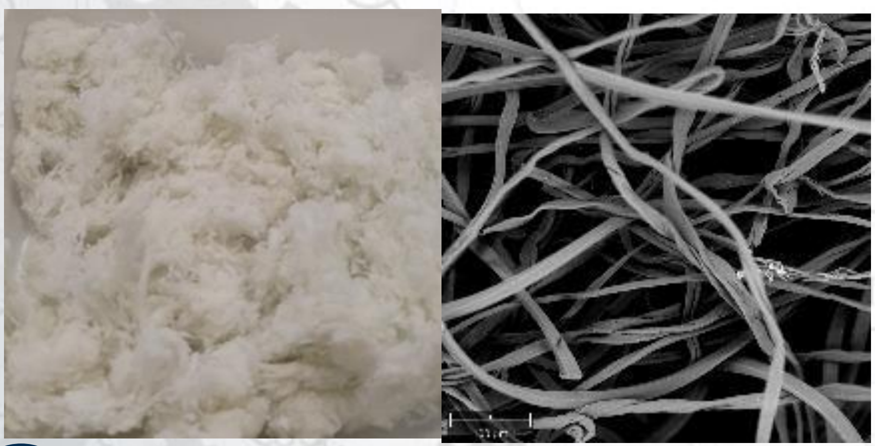
- Objectives
  1. Separation of the polyester/cotton material into CNC and pure polyester fibres.
  2. Optimise the reaction conditions-time (50-75min), temperature (60-90 °C) and chemical concentration (55-70%) for the maximum extraction of CNCs and recovery of polyester fibres.
  3. Characterisation of the extracted CNCs for their properties

Materials

a

b

c



Sample	Fabric density (g/m^2)	Average composition of polyester (%)	Average composition of cotton (%)
Woven fabric	263	23.70	76.30
Knitted fabric	203.25	17.42	82.58
Mixed shoddy	-	39.53	60.67

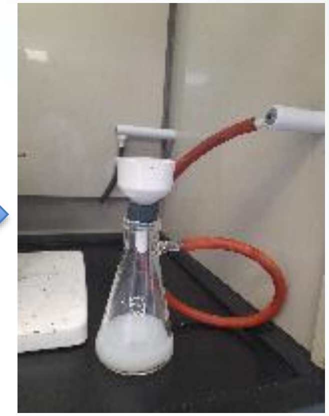
Figure 1: Visual appearance (left) and SEM-micrographs (right) of decolourised materials a) woven fabric waste[b) knitted fabric waste and c) mixed shoddy waste

Experimental Procedure-CNC extraction

Blended textile waste (decolourised)

Acid hydrolysis
55-70% H_2SO_4,50° C,
500rpm, 50-75min

Quenching
10x DI water



Separation
350µm
mesh

Recovered PET fibres

Centrifugation 3x
15min,9000rpm

Dialysis MWCO
12000Da

500W, 60%
amplitude, 5min

CNC suspension

Characterisation
TEM,
FTIR, XRD,
hydrodynamic, TGA

Results- CNC preparation

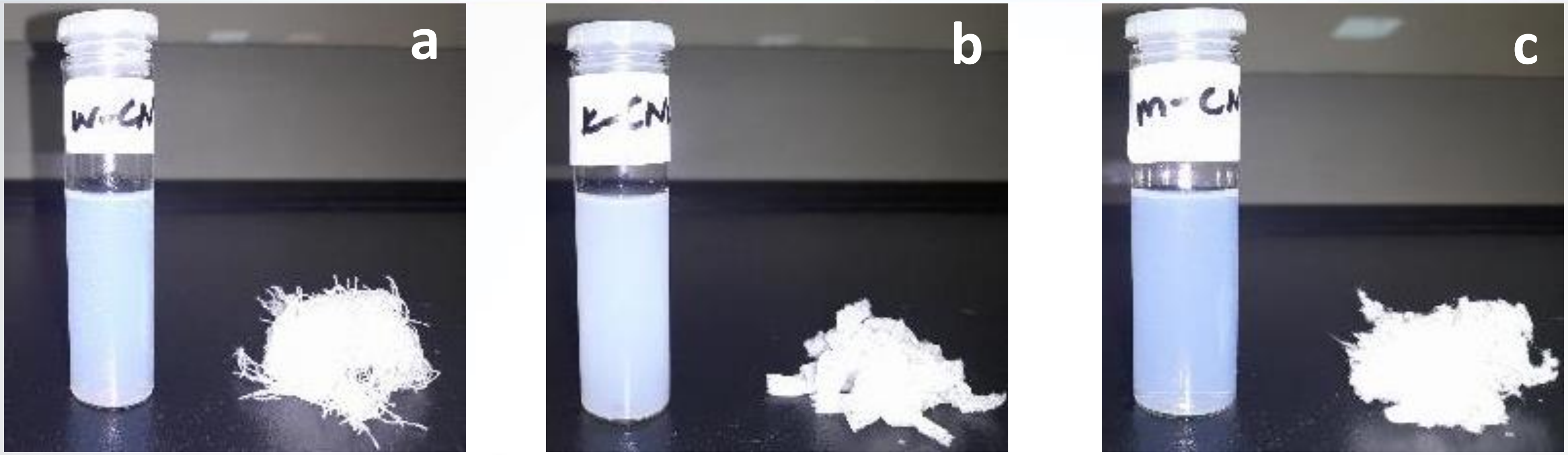


Figure 2: Visual appearance of CNC suspensions a) WCNC, b) KCNC, c) MCNC

- Low concentration (<64%) and low time (below 60min)- not effective-settlin of suspension due to large particles
- Higher temperatures (>64%)and longer times (>60 mins) resulted in lower yield or total cellulose hydrolysis
- Optimum conditions for CNC extraction 64% H_2SO_4,60min, 50° C

Sample ID	Yield		PET recovered (wt %)
	g CNC per g sample	g CNC per g cotton	
WCNC	44.8	69.5	99.8
KCNC	53.5	66.3	98.7
MCNC	22.4	38.1	88.3

Results- Hydrodynamic properties

Table 1. Yield and hydrodynamic properties of CNCs extracted from post-consumer waste

Sample	Hydrodynamic size (nm)	Zeta potential (mV)	Polydispersity Index (PDI)
WCNC	158.4 ± 0.85	- 33.2 ± 0.55	0.277 ± 0.015
KCNC	163.1 ± 1.50	- 31.5 ± 0.65	0.217 ± 0.019
MCNC	153.8 ± 2.01	- 31.8 ± 1.37	0.244 ± 0.006

Results- Morphological Properties

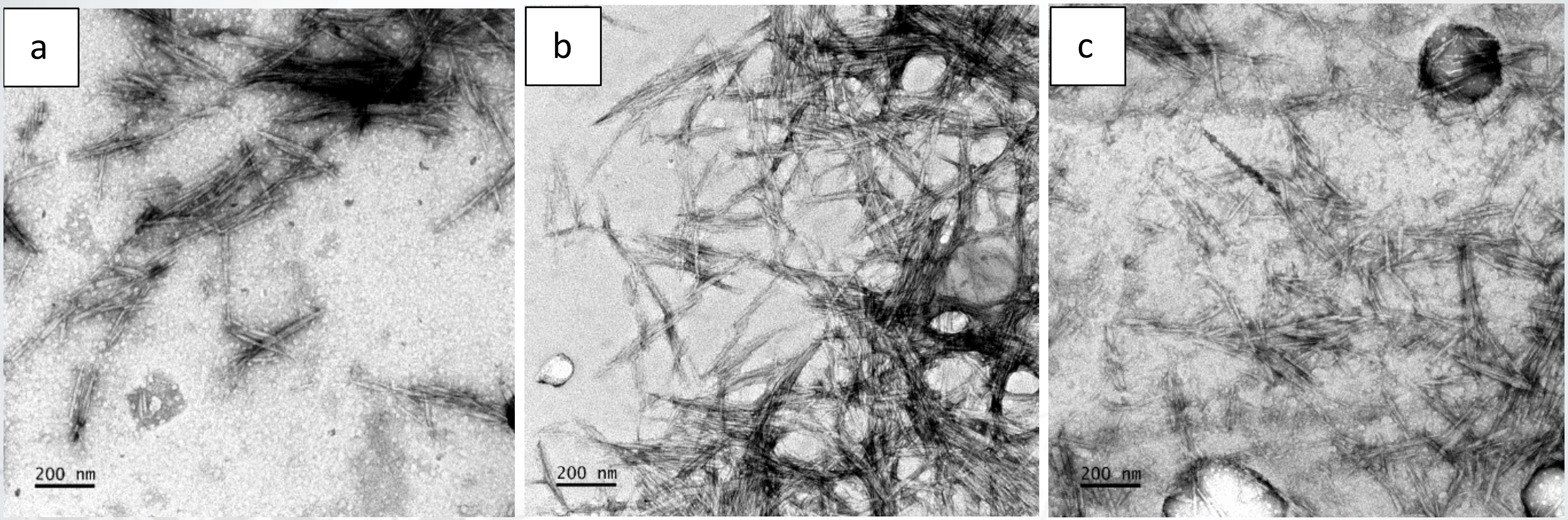
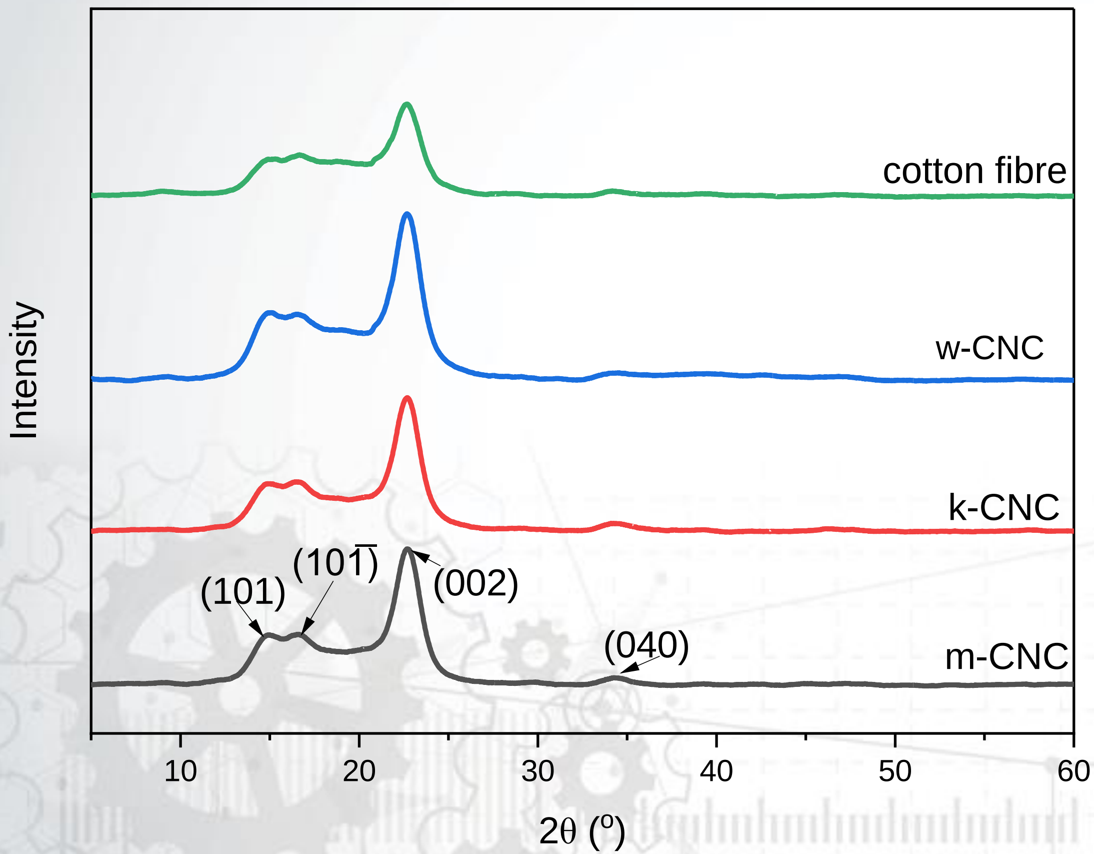


Figure 3. TEM images of extracted cellulose nanocrystals a) WCNC, b) KCNC and c) MCNC

Sample ID	WCNC	KCNC	MCNC
Length (nm)	135-358	78-194	165-295
Width (nm)	6.30-15.96	5.40-9.66	12.23-16.25

Results-Crystallinity properties



Sample	Crystallinity Index	Average crystallite size (nm)
Cotton fibre	70.3	-
w-CNC	86.1	3.52
k-CNC	89.9	3.81
m-CNC	74.5	3.85

Figure 4: XRD patterns of extracted CNCs as compared to the pattern of cotton fibre

Crystallinity- Peak deconvolution method (Hermans et al. 1948, Salem et al. 2023)
Crystallite size-Scherrer equation (Patterson, 1939)

Results-Surface functional group analysis

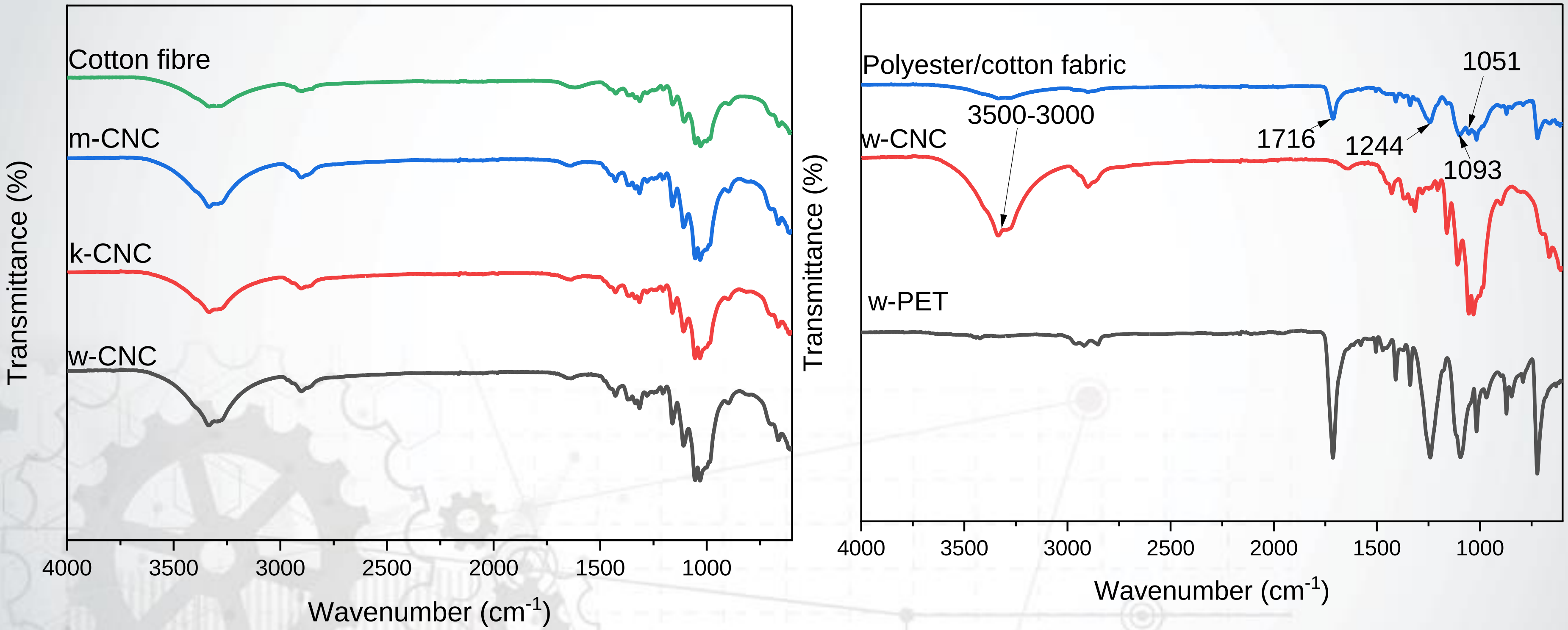


Figure 4. (a) FTIR spectra of waste cotton fibre and the extracted CNCs, (b) spectra of woven polyester/cotton fabric, w-CNC and polyester fibre recovered from the woven fabric

3300–3600 cm^{-1} (OH group)
1160 and 1109cm^{-1} (C-O groups)
896cm^{-1} (O-S3Na)

2900 & 1311 cm^{-1} (C-H groups)
1033cm^{-1} (S=O stretches)

Results-Thermal properties

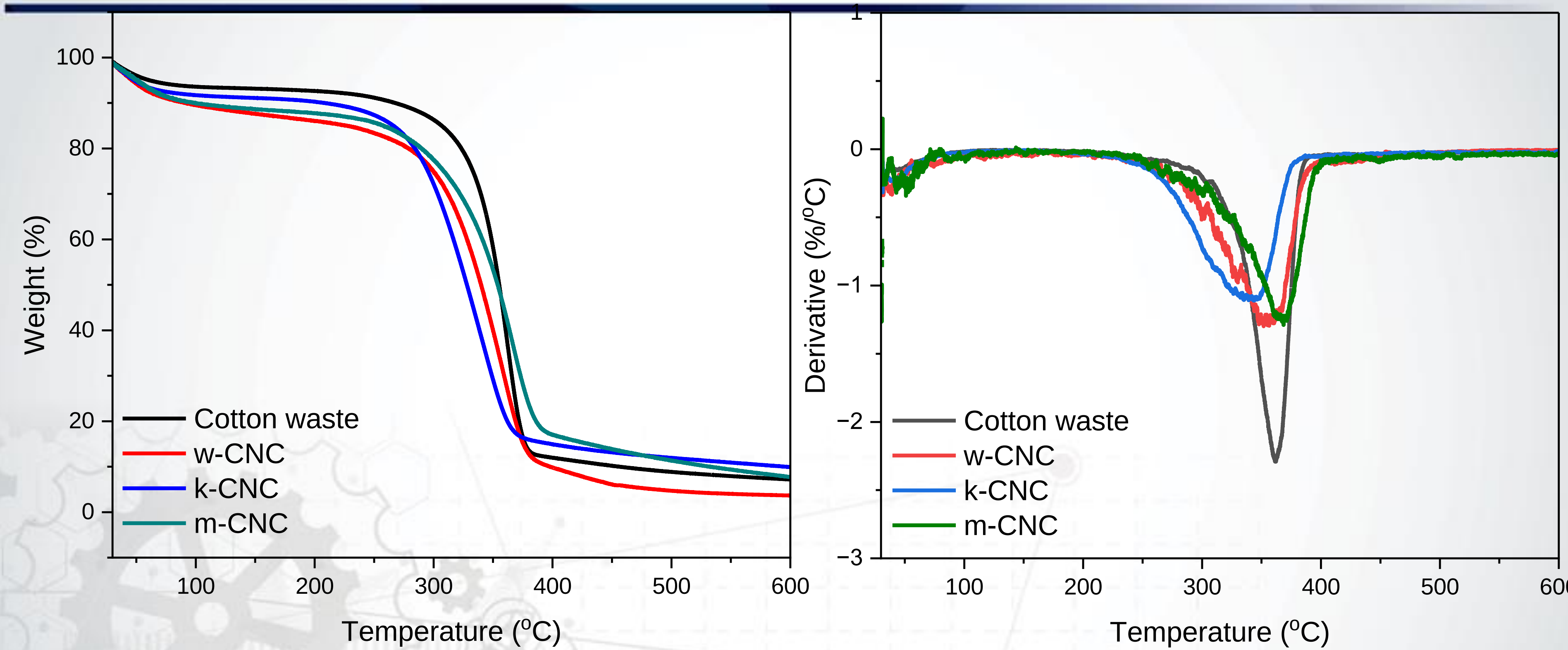


Figure 5. (a) TGA and (b) DTG curves for cotton and extracted CNCs

- Two-step degradation profile
- 5% weight loss @ 100° C due to volatiles
- Approx 87%weight loss from 344 to 366 ° C, breakdown of primary cellulose chains
- Lower thermal stability of CNC than cotton due to reduce surface area & presence of sulphate groups.

Conclusions and Recommendations

- This study effectively isolated CNCs from woven fabric, knitted fabric and mixed shoddy through hydrolysis with sulfuric acid followed by dialysis and ultrasonication
- The CNCs showed similar rod-like structures, ranging from 5 to 16nm and a length of 78 to 358nm.
- The efficiency of the obtained CNC yield (38-69% cotton basis) surpassed that of the CNC mentioned in the literature from cotton waste
- The CNC properties were significantly influenced by the acid-to-cellulose ratio, as indicated by the results obtained from the samples with different cotton/polyester ratios
- However, when handling the mixed shoddy, it is recommended to further break down the fibres to prevent the tangling of the yarns during mixing, consequently diminishing the efficiency of the acid hydrolysis process
- The CNC yield, reproducibility across different batches, purity level, high crystallinity, thermal and hydrodynamic properties of the CNCs highlight the potential of blended post-consumer textile waste as a viable source for CNCs possibly as reinforcements in plastic packaging, medical applications and paper industry.
- Recovered polyester fibres have the potential to be recycled back into the textile industry.

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Thank you!