# Transforming Biowastes into Advanced Materials for Diverse Applications

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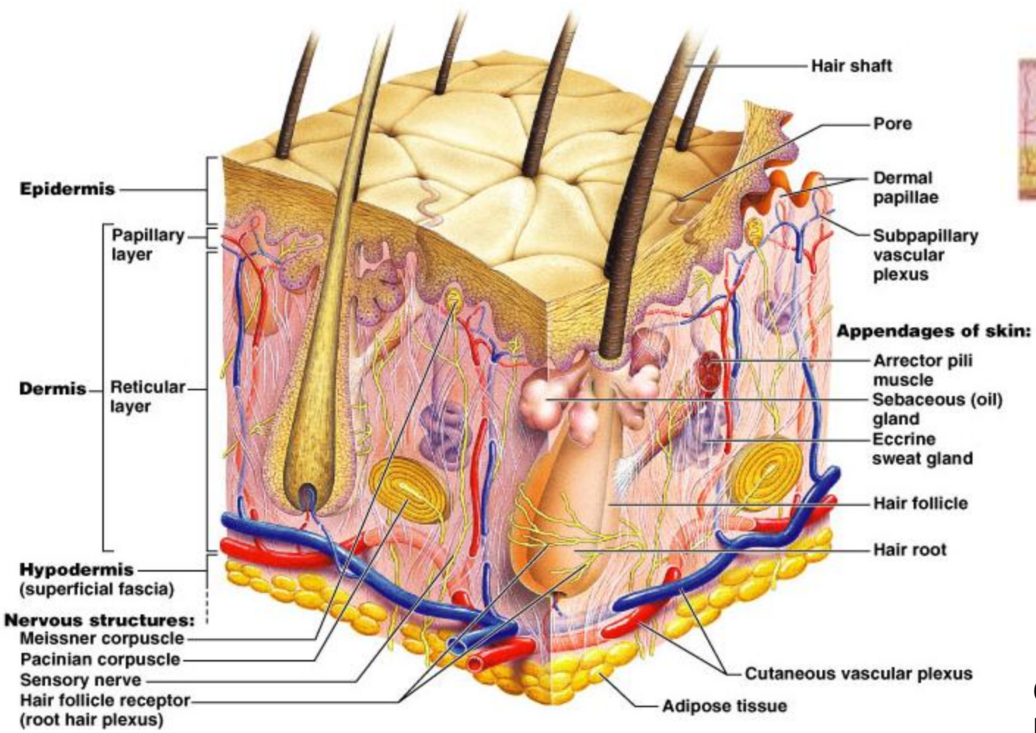
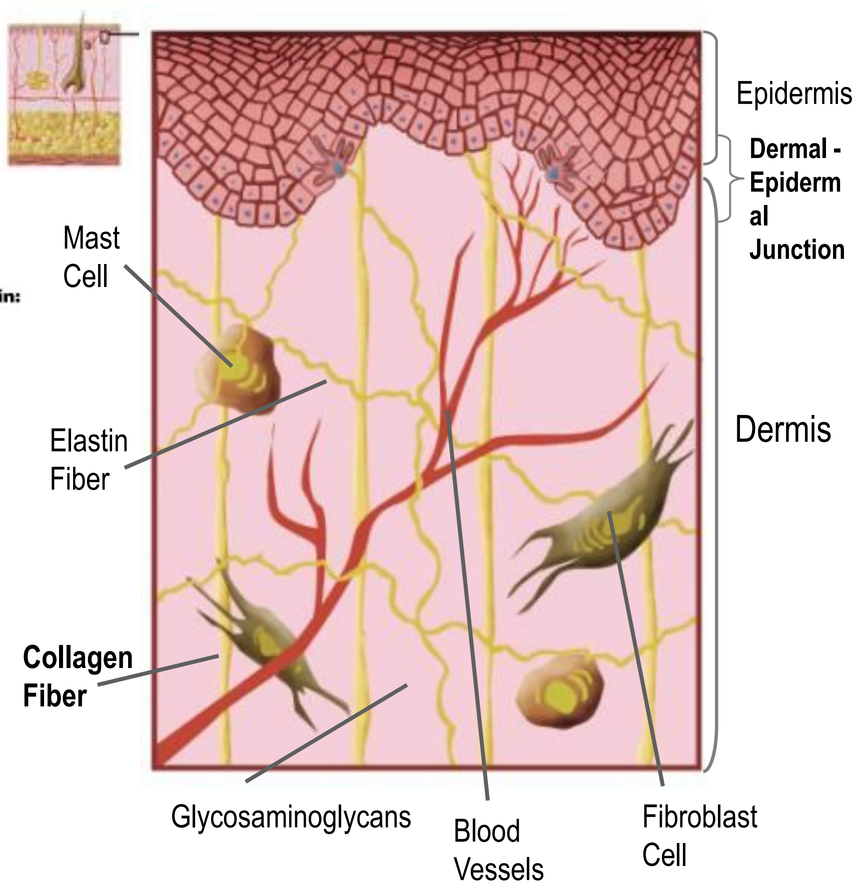
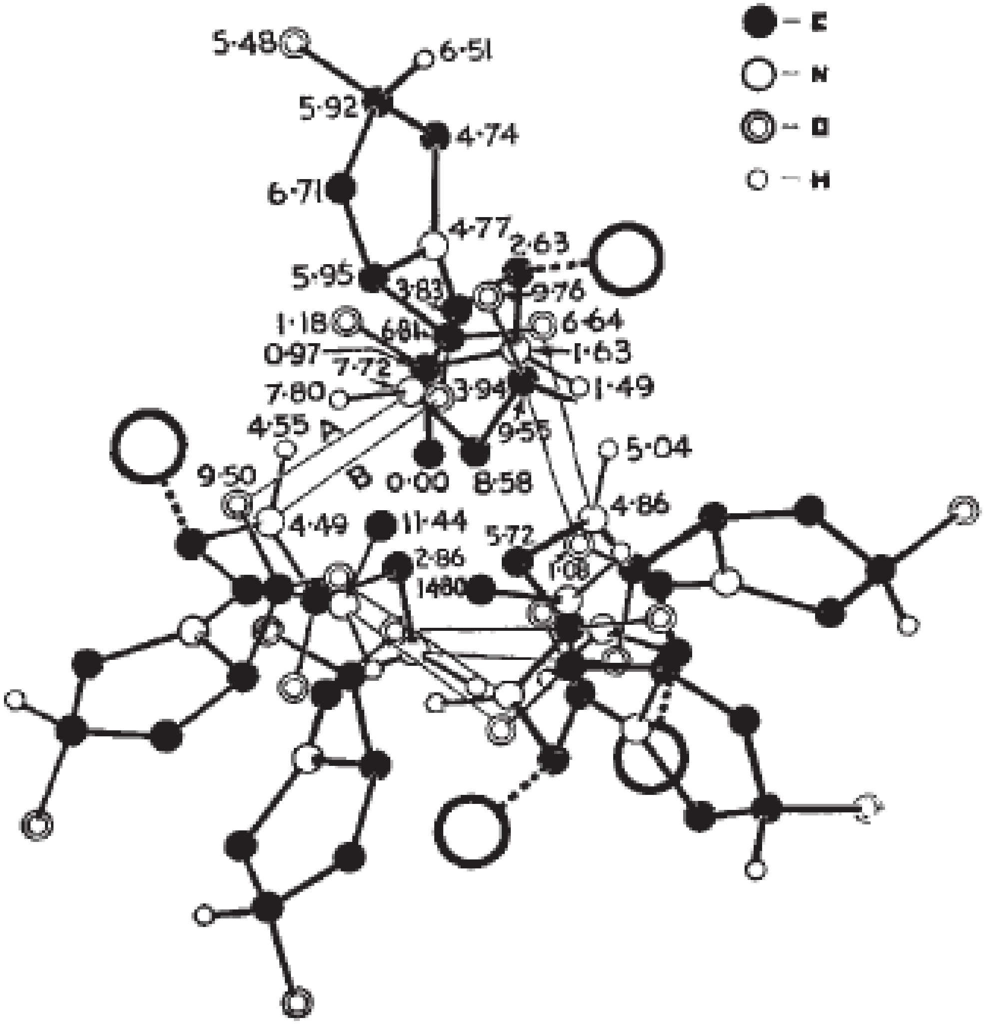
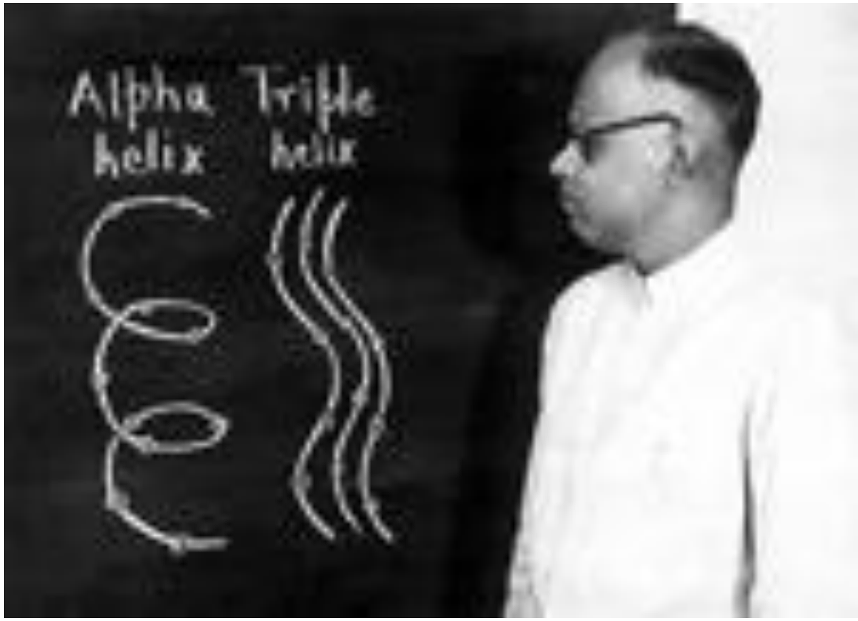
# Collagen: A Skin Protein



# Collagen Structure: G.N. Ramachandran

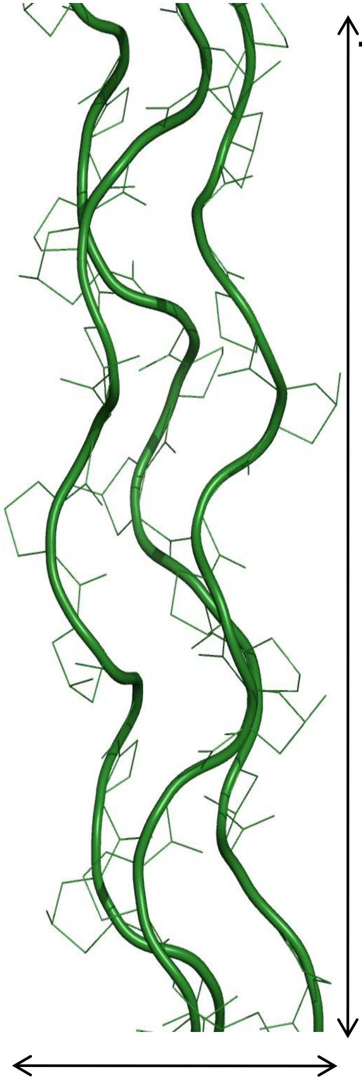


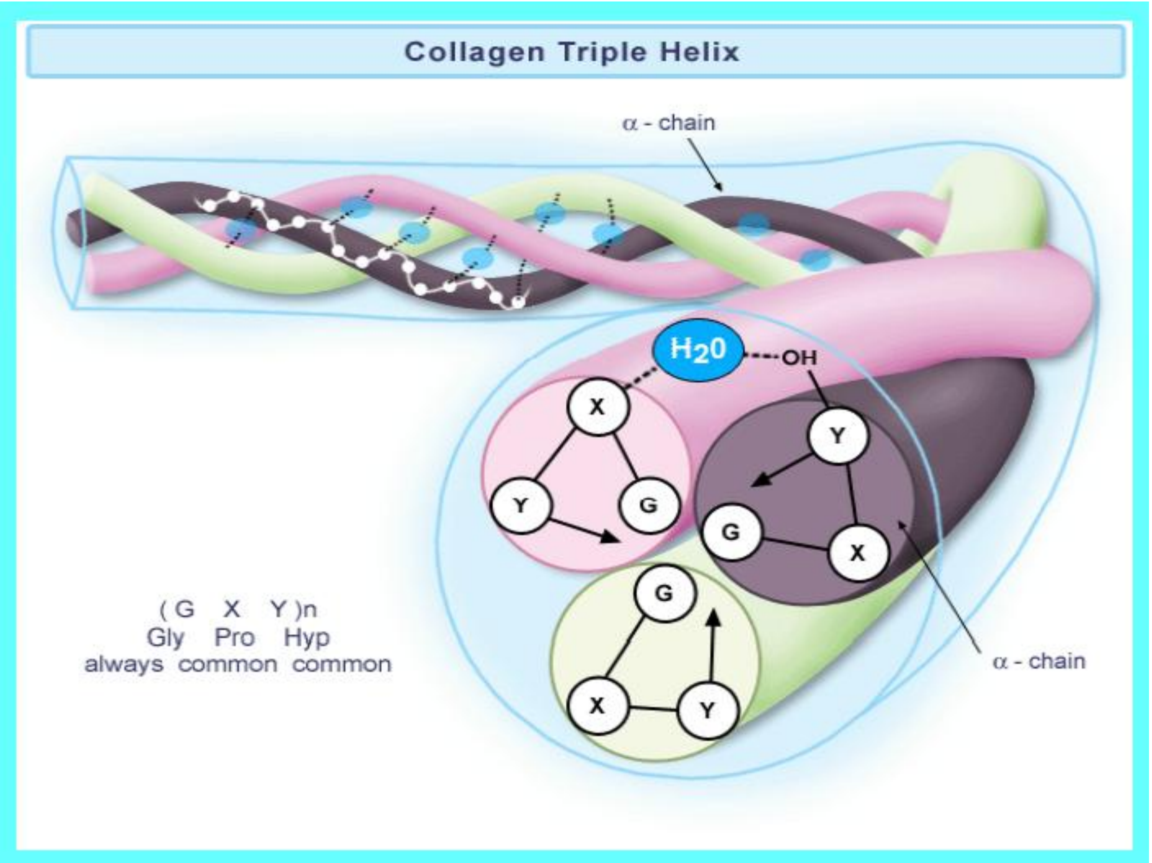






*Nature*, **174**, 269 (1954); *Nature* **176**, 593 (1955)

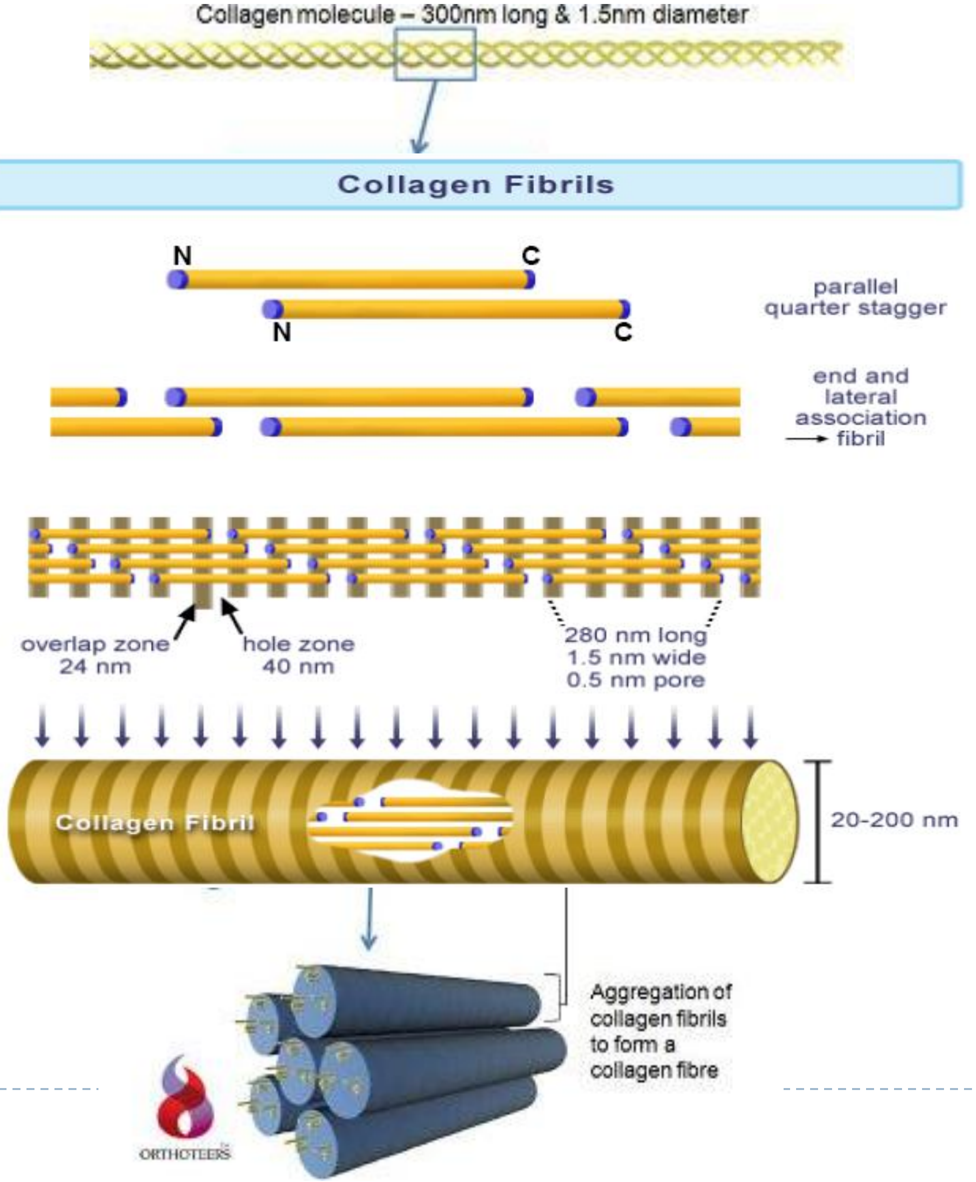
# Collagen Triple Helical Structure



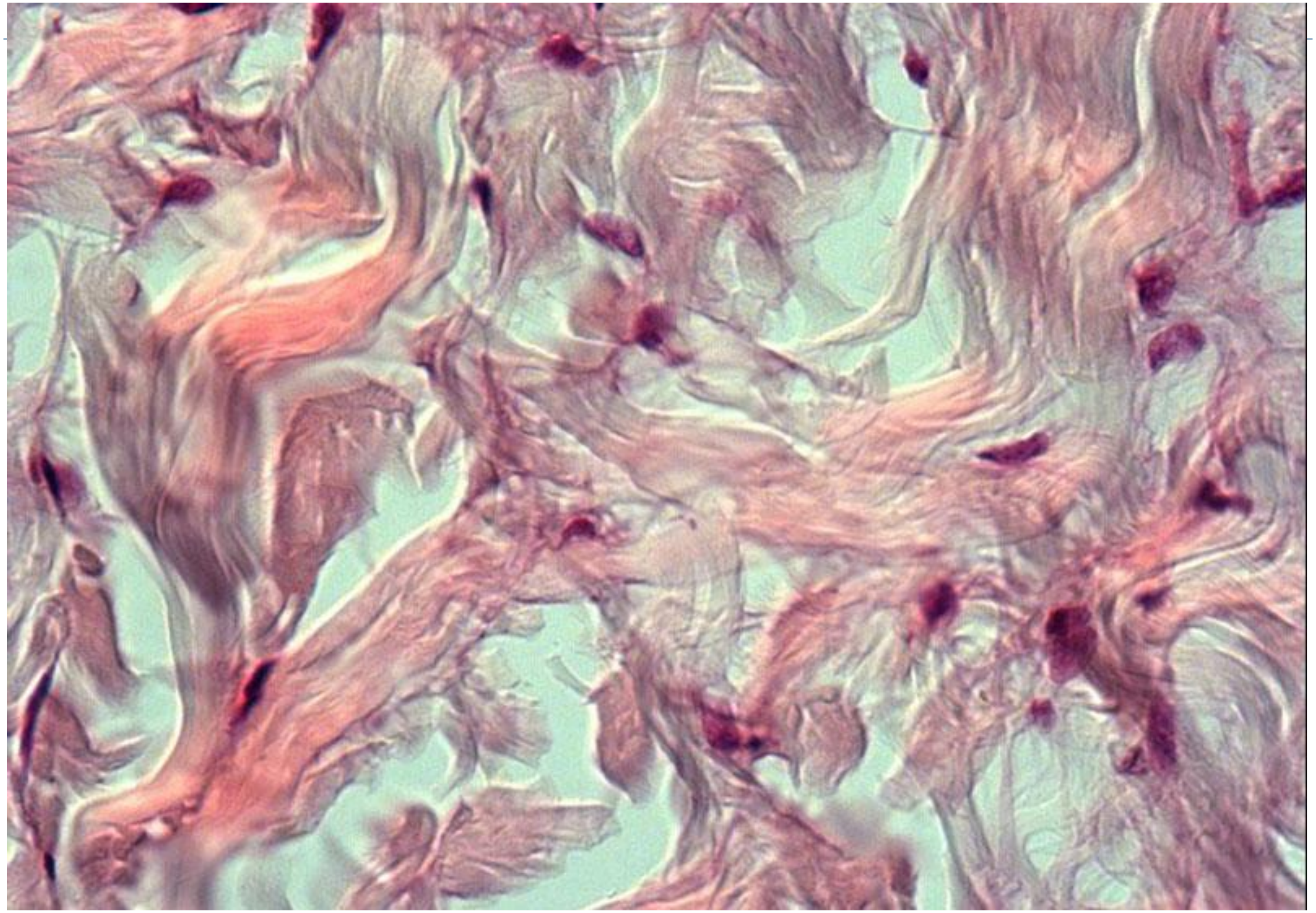




**A long cylindrical protein consisting of three poly peptide chains wound around each other in a suprahelical fashion**

# Collagen Architecture



# Animal Skin



This image of the dermis layer of skin shows **fibroblasts and thick collagen bundles** (connective tissue).
Credit: Ivor Mason, Wellcome Images

# Food Chain: Collagen

**Skins/hides**

**Agriculture**

**Meat industry**

**Leather industry**

- Global meat industry: 1 trillion USD in 2024 expected to cross over 1.3 trillion dollars by 2027
- Animal skins and hides are by-products of meat industry: Feeding to leather/product industry having market size of 450 billion USD in 2024

# *Leather Processing: Global Status*

- Employs large amounts of chemical inputs with utilization levels lower than 10% and generates solid, liquid, gaseous wastes and environmental problems
- The amount of waste discharged from leather processing (per ton of Skins/Hides) includes
  - **Solid wastes: about 600-700 kg**
  - Liquid wastes: about 40,000 lit
  - Total Dissolved Solids (Salt): 350-450 kg
- Significant amount of **proteins**, which have great economic potentials, are generated in the **solid wastes**

# *Protein Based Solid Wastes*

Proteinaceous collagen wastes

Cr-free collagenous wastes

Raw hide/skin trimmings, limed split wastes, green/limed fleshings, etc.

Cr-containing collagenous wastes

Cr shavings, buffing dust, crust trimmings, finished leather trimmings, leather scraps, etc.

**Various attempts have been made to overcome the problems of solid waste disposal.**

# Protein Wastes: Current Applications

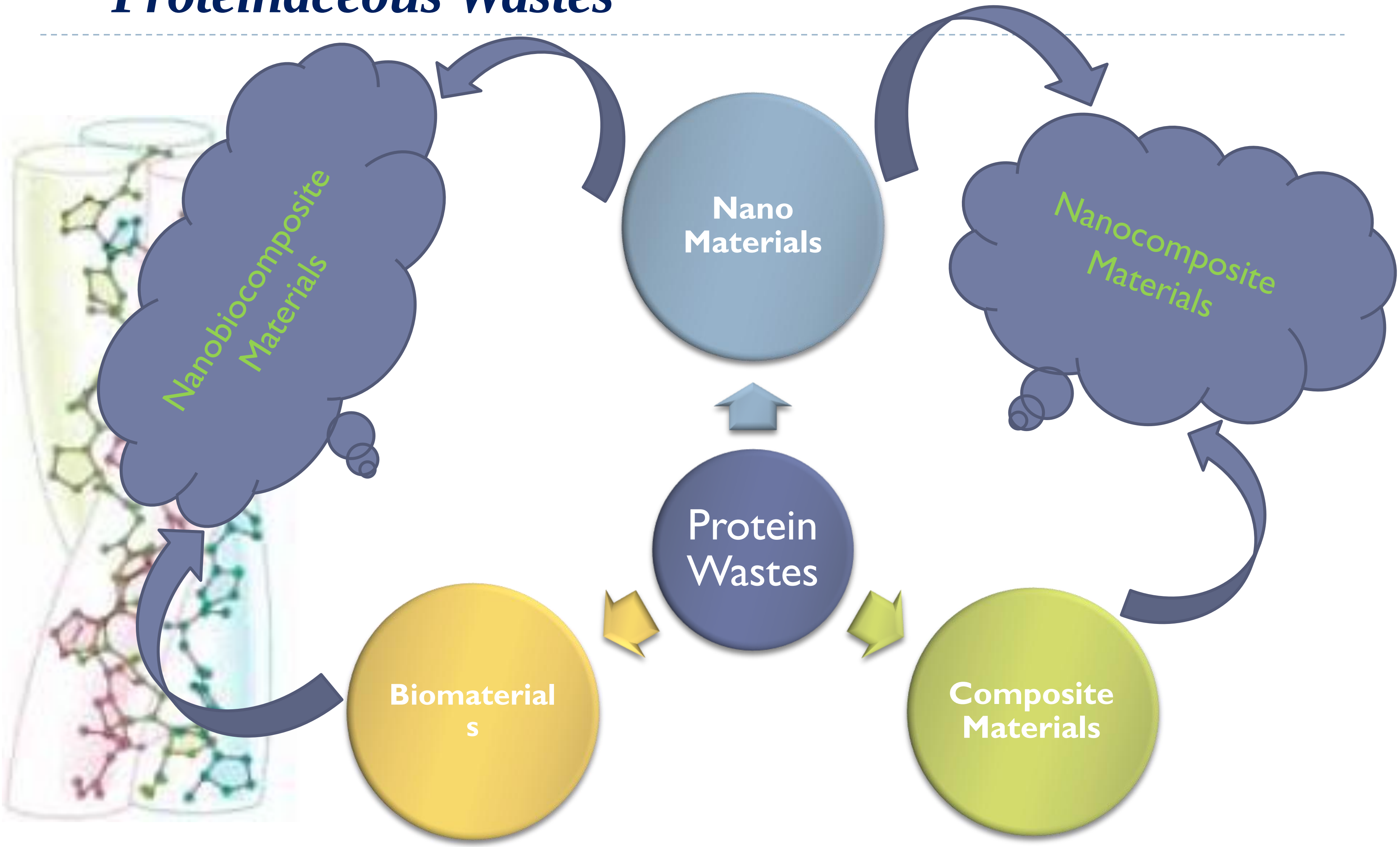
Skin trimming wastes

Chrome shaving wastes

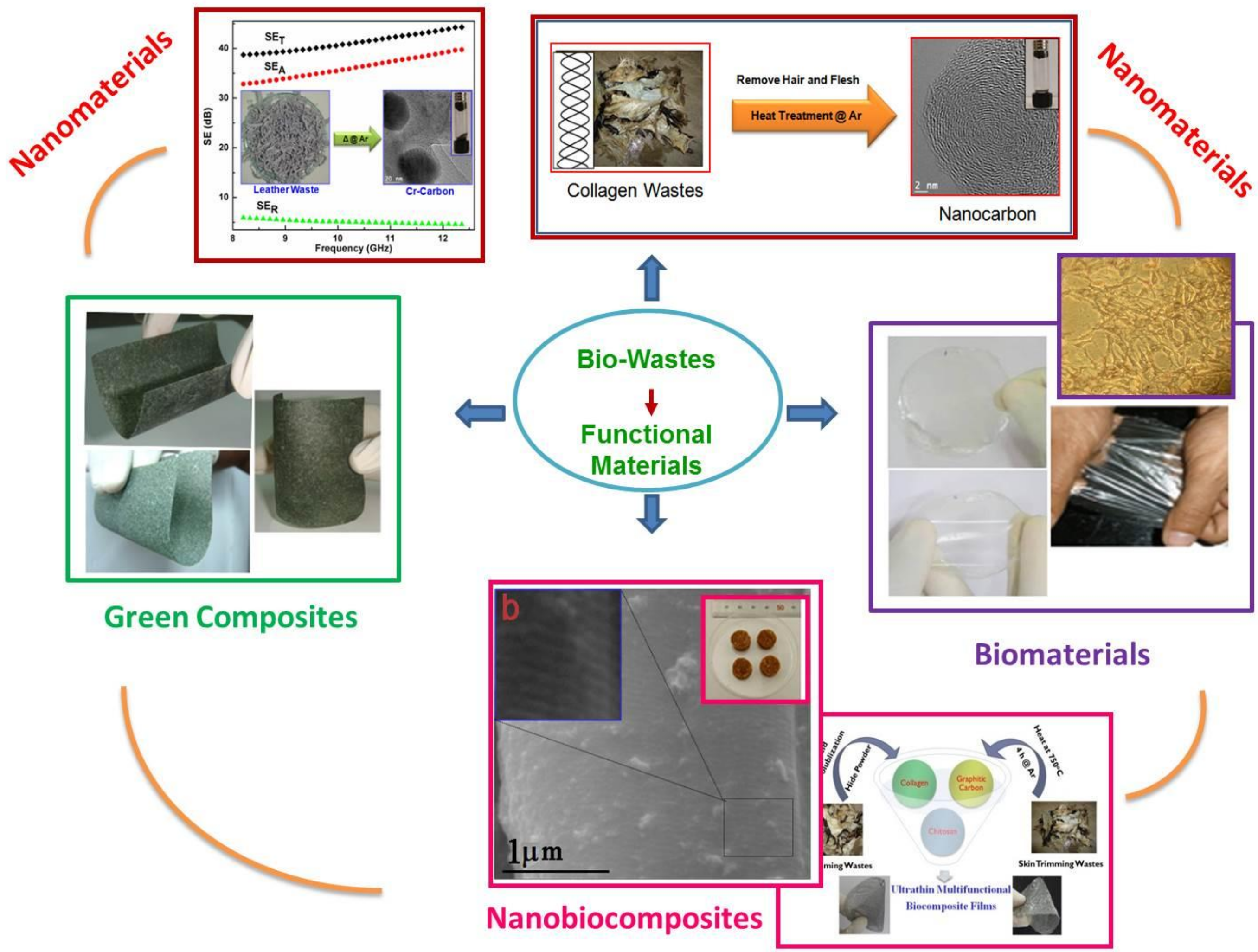
- Present applications include glue, gelatin, adhesive manufacture, etc.
- Except gelatin, all the other products obtained from skin wastes are found to have low value applications
- Hydrolyzed using enzymes/alkali - isolate Cr-free protein and chrome cake - used as ingredients for fertilizer, feeds, cosmetics and pigments.
- Incorporated with different polymeric matrices to form boards/sheets/insole - used in footwear and clothing industries

**An opportunity do exist to convert these solid wastes into high-value advanced materials**

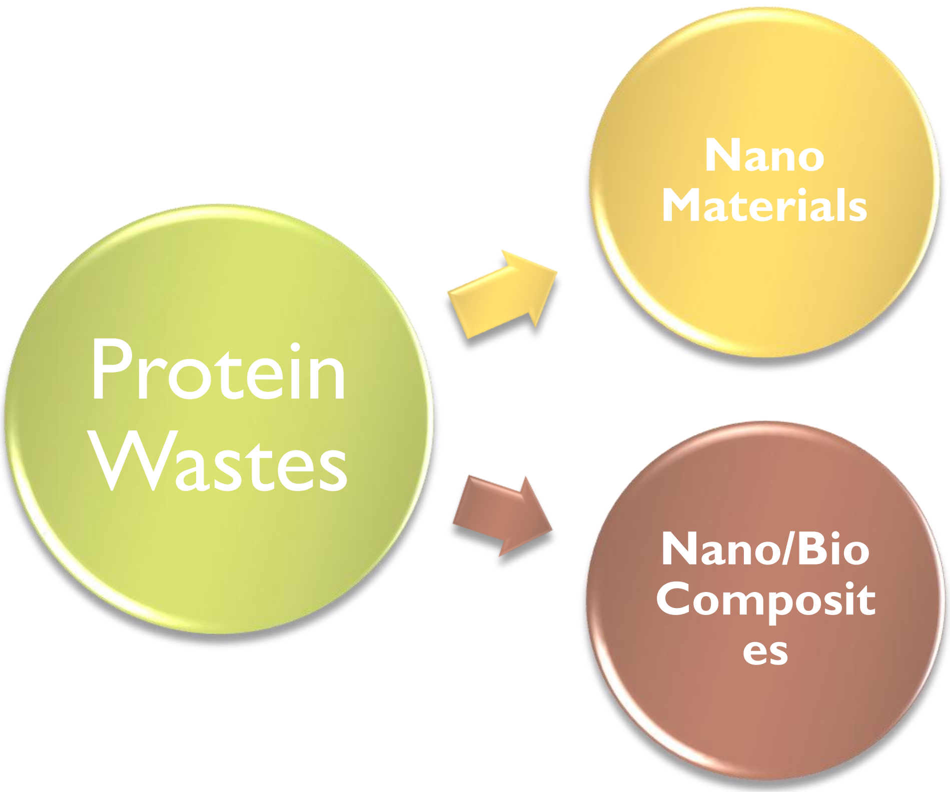
# *Development of Innovative Materials from Proteinaceous Wastes*



# Bio-Wastes to Advanced Functional Materials



# Development of Innovative Materials




*J. Clean. Prod.* 2019, **210**, 190
*Carbon* 2018, **130**, 645
*ACS Sustainable Chem. Eng*. 2013, **1**, 619
*Green Chem*. 2012, **14**, 1689

*Sci. Rep.* 2019, **9**, 1040
*ACS Omega* 2017, **2**, 5260
*ACS Appl. Mater. Interfaces* 2016, **8**, 14836
*RSC Adv.* 2016, **6**, 63071
*Mater. Chem. Phys*. 2015, **157**, 8
*RSC Adv.* 2015, **5**, 20939
*RSC Adv*. 2014, **4**, 19507
*Carbon* 2012, **50**, 5574
*Sci. Rep*. 2012, **2**, 230

# Doped Nanocarbon Materials from Collagen Wastes

Green Chemistry

Dynamic Article Links

Cite this: *Green Chem.*, 2012, **14**, 1689

www.rsc.org/greenchem

PAPER

## Transforming collagen wastes into doped nanocarbons for sustainable energy applications†

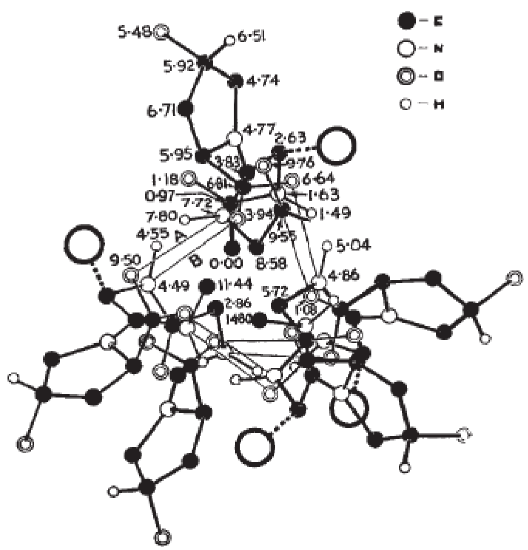
**Meiyazhagan Ashokkumar,[a] Narayanan Tharangattu Narayanan,[b] Arava Leela Mohana Reddy,[b] Bipin Kumar Gupta,[c] Bangaru Chandrasekaran,[a] Saikat Talapatra,[d] Pulickel M. Ajayan*[b] and Palanisamy Thanikaivelan*[a]**

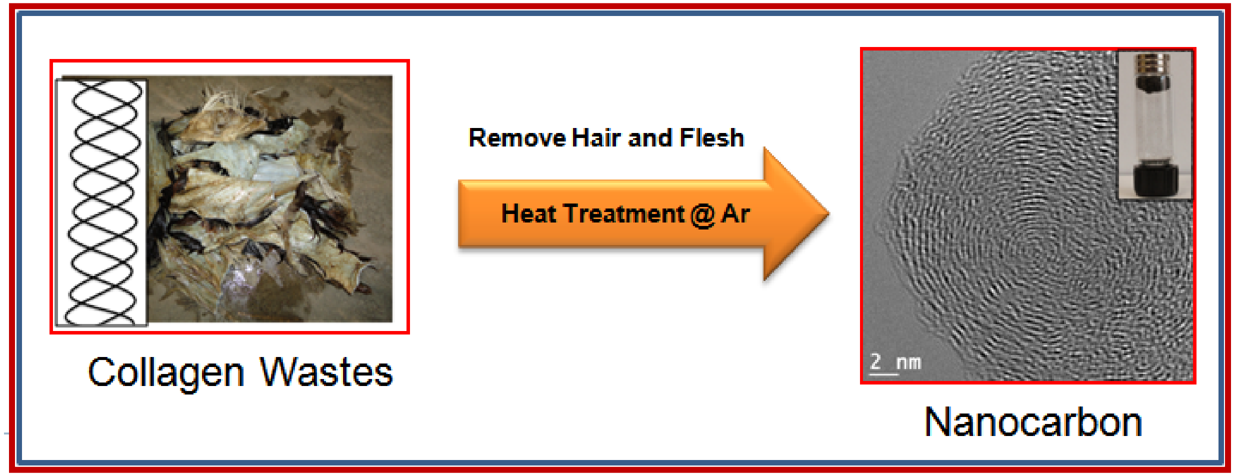
***Received 21st February 2012, Accepted 19th March 2012***
**DOI: 10.1039/c2gc35262a**

Leather industry produces huge quantities of bio-waste that can be used as raw material for the bulk synthesis of carbonaceous materials. Here we report the synthesis of multifunctional carbon nanostructures from pristine collagen wastes by a simple high temperature treatment. Our studies reveal that the nanocarbons derived from the bio-waste have a partially graphitized structure with onion-like morphology and are naturally doped with nitrogen and oxygen, resulting in multifunctional properties

# Experimental

- ***Synthesis of carbon nanomaterials from collagen wastes:***



# *Atomic composition of carbon materials*

| Carbon materials: Temperature (°C) | Carbon materials: Type of analysis | % C | % O | % N | % H | % Other elements (Na, S, Ca, Cl) |
|---|---|---|---|---|---|---|
| 500 | XPS | 77.94 | 6.17 | 12.79 | - | 3.10 |
| | CHNS | 72.21 | - | 13.56 | 3.05 | 4.94 |
| 750 | XPS | 81.58 | 9.84 | 7.21 | - | 1.37 |
| | CHNS | 78.72 | - | 8.45 | 1.60 | 1.40 |
| 1000 | XPS | 79.68 | 15.41 | 2.86 | - | 2.05 |
| | CHNS | 78.56 | - | 3.12 | 0.55 | 1.41 |

# *Raman Spectroscopy & XRD*

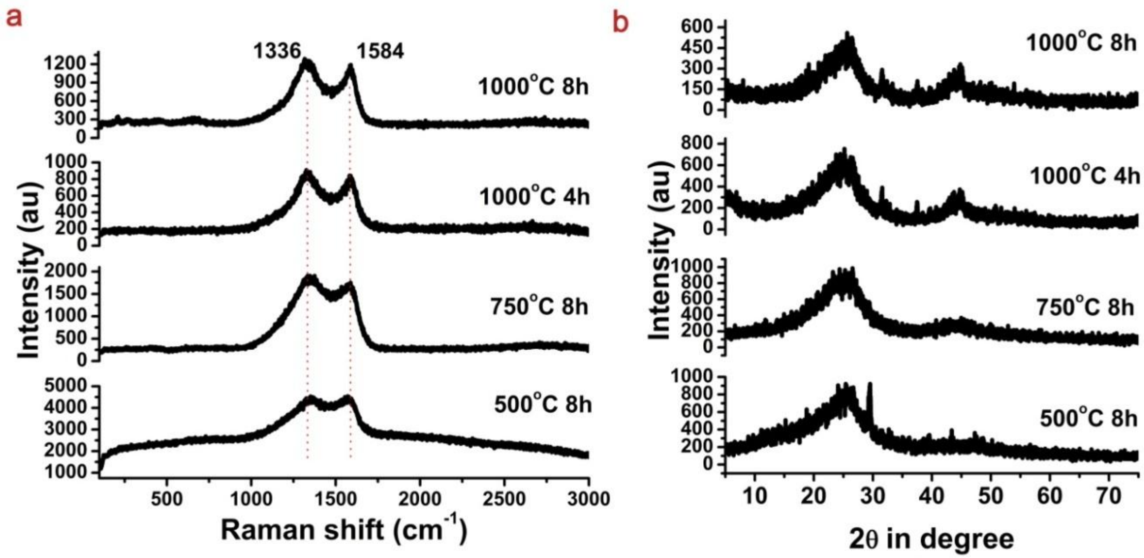



Fig 1a, Raman and Fig 1b, XRD characterization of the select carbon materials.

➢2 broad bands - 1336 symmetric disorder (D mode) occurring in diamond like carbon materials

➢1584 cm$^{-1}$ corresponds to single-crystalline graphite originating from the sp$^2$ bonded carbon atoms in a 2D hexagonal lattice.

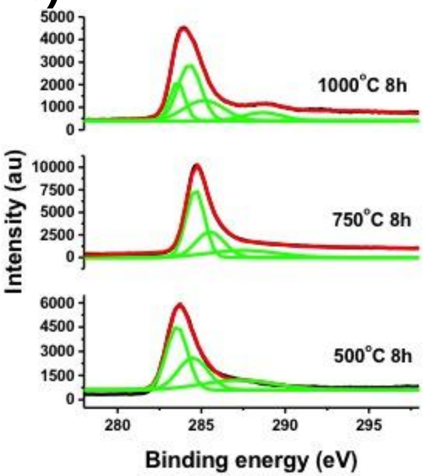
➢Powder X-ray diffraction patterns of the carbon materials exhibit peaks around 2θ = 25 and 45$^{o}$, which can be assigned to (002) and (101) diffraction peaks of crystalline graphitic carbon.

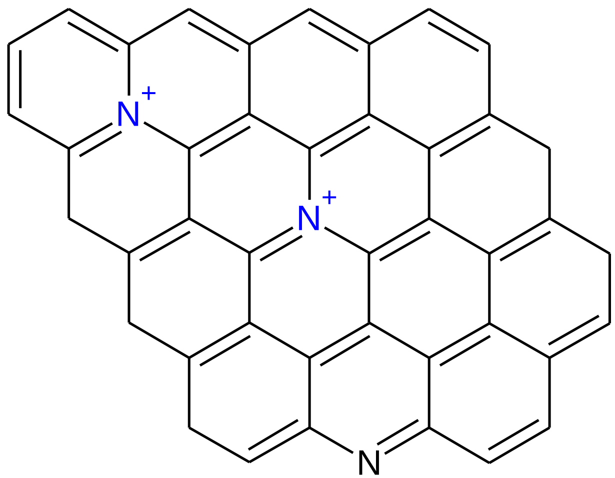
➢Broadening decreases and the crystallinity increases as temperature increases.

# *X-ray Photoelectron Spectroscopy*

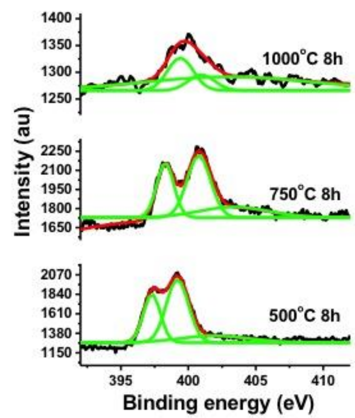
- It was seen that the formation of graphitic nitrogen substituting inner C atoms on the graphene layer increases while the pyridinic nitrogen contribution decreases as the treatment temperature increases.

C(1s)

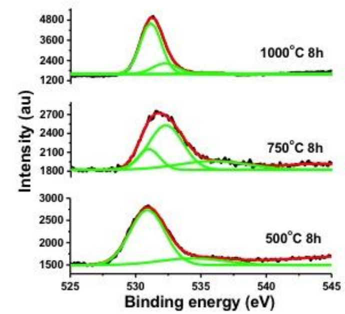



N(1s)

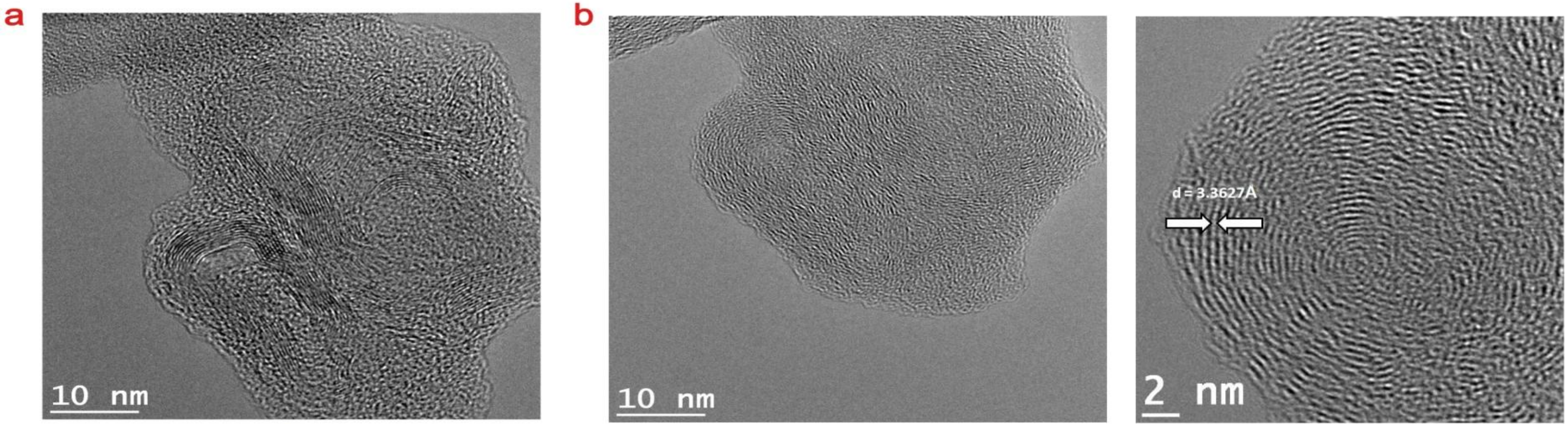



O(1s)




- The pyridinic nitrogen might be converted into $N_2$ gas in anoxic conditions at higher temperature and escape out during pyrolysis.

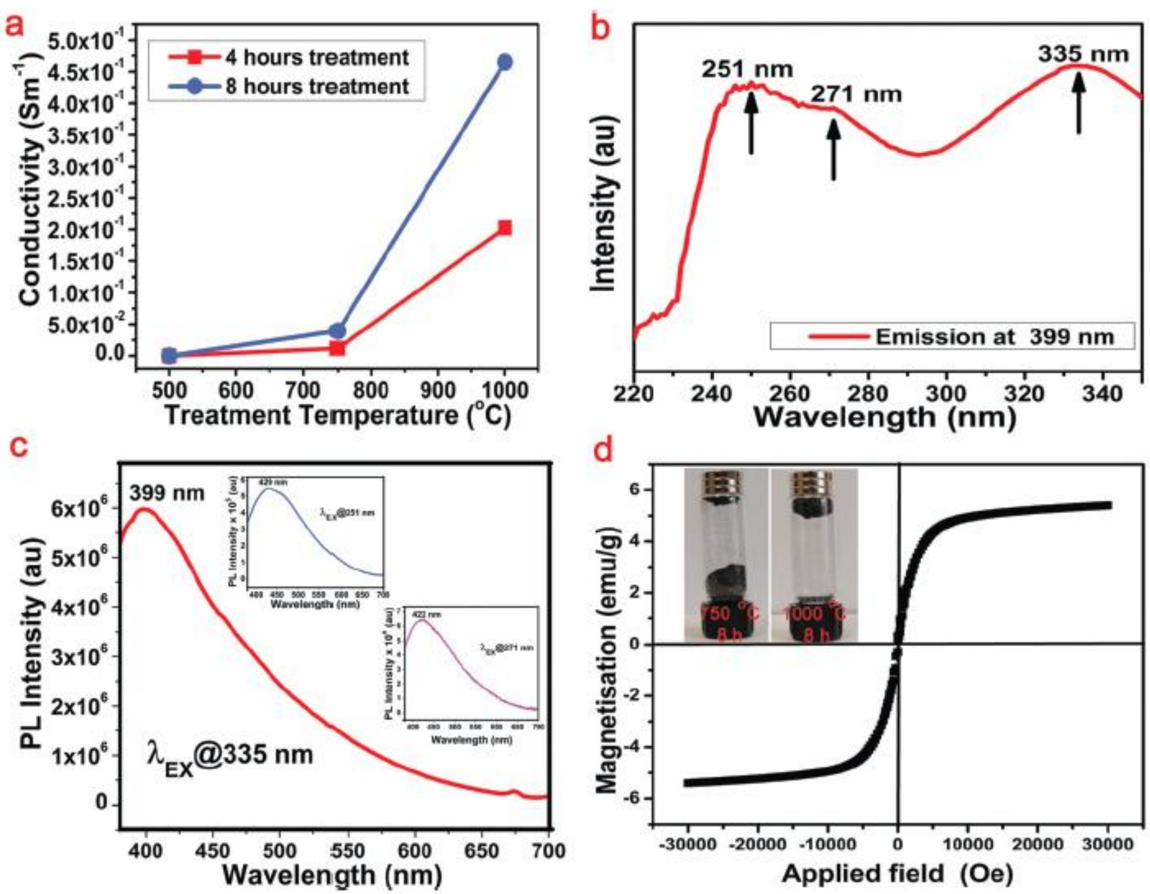
# HR-TEM




➢HR-TEM images of the nanocarbons show the presence of graphitic layers and carbon onions. Each onion, measures ~20 nm - separated from the adjacent layers by approximately 3.363 Å.

➢Slight increase in the interplanar distance d spacing from 3.354 Å to 3.363 Å could be due to the structural as well as ordering defects owing to the presence of nitrogen and oxygen atoms

# *Multifunctional properties of the derived Carbon nanostructures*




**(a) Electrical conductivity of the carbon materials as a function of treatment (b) Photoluminescence excitation (PLE) spectrum (c) Photoluminescence (PL) spectrum of carbon derived from high temperature treatment (1000 ° C for 8 h) (d) Room temperature M(H) curve of carbon derived from collagen waste (1000 ° C for 8 h**

# *Collagen derived nanocarbons for Li-Ion Battery Applications*

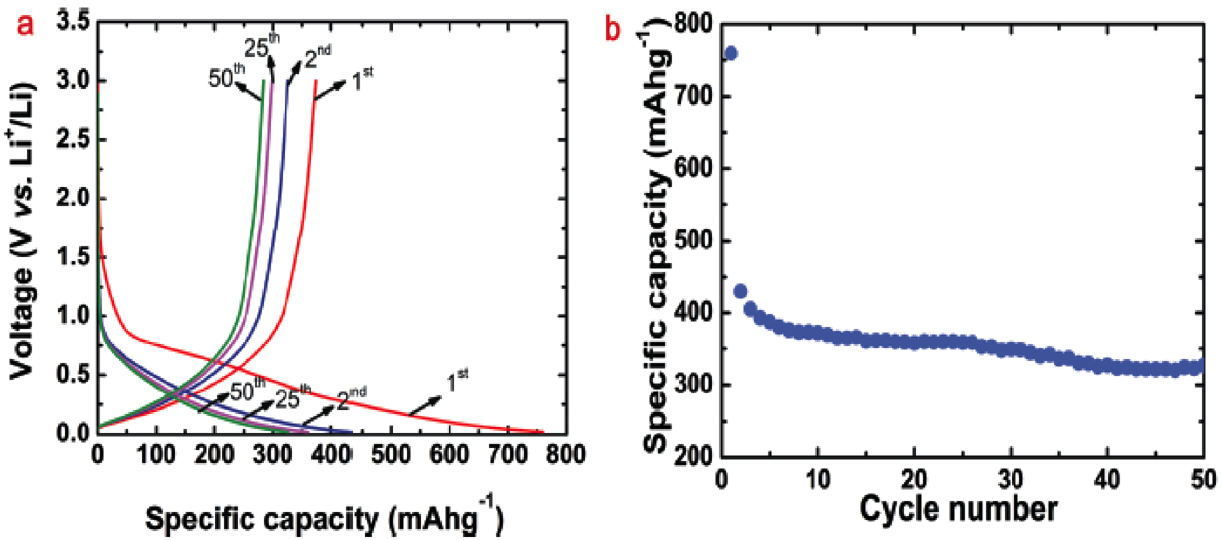


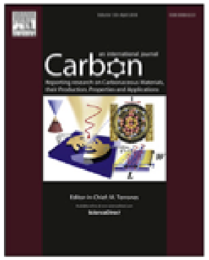

**Fig. 5** Electrochemical characterization of the carbon materials derived from collagen waste (1000 °C for 8 h) as positive electrode in Li ion battery. (a) Charge–discharge voltage profiles cycled at a rate of 50 mA $g^{-1}$ between 3.0 V and 0.02 V *vs.* $Li/Li^{+}$. (b) Cycling stability as a function of discharge capacity demonstrating fairly stable reversible capacity of 327 mA h $g^{-1}$ even after 50 cycles.

Carbon 130 (2018) 645–651

Contents lists available at ScienceDirect

Carbon

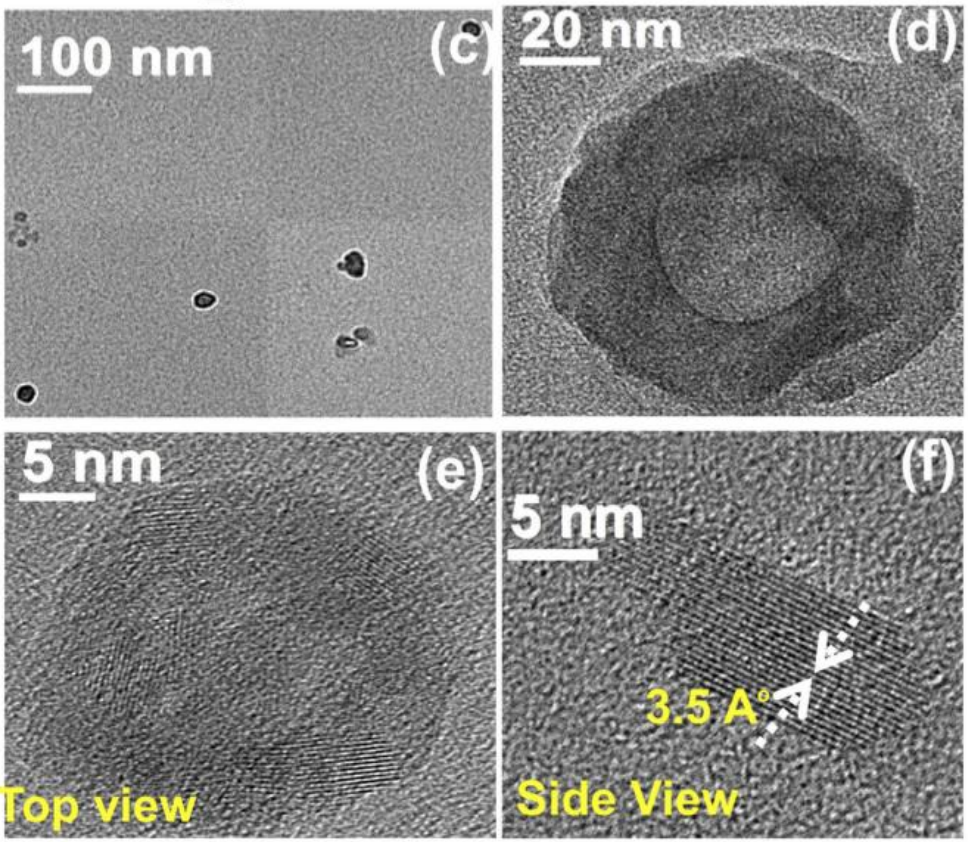
journal homepage: www.elsevier.com/locate/carbon

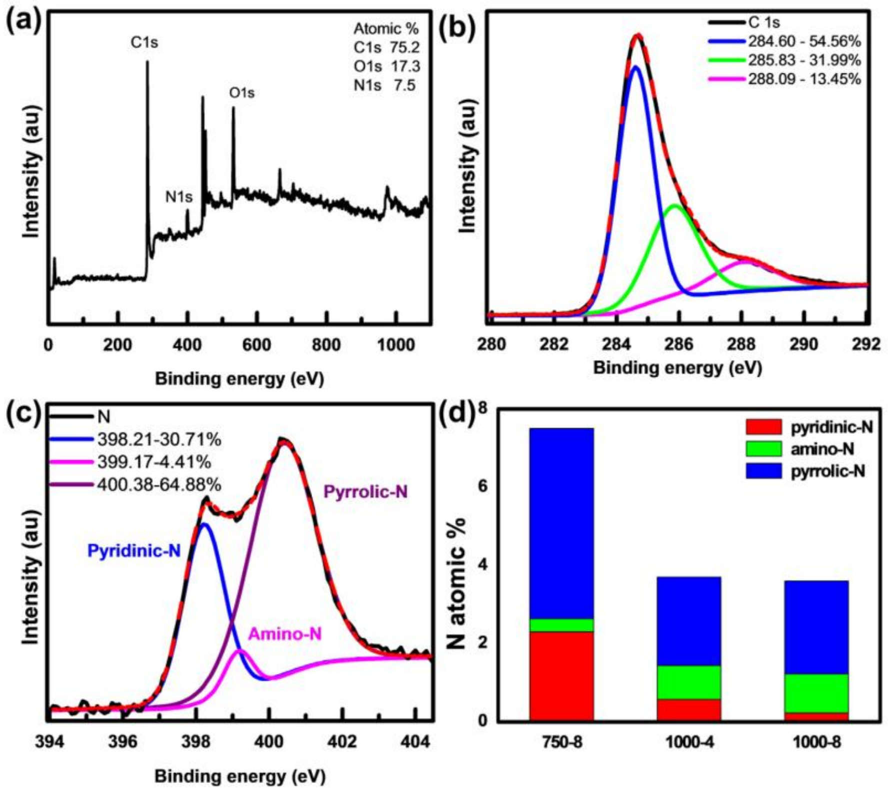



# Nitrogen-rich carbon nano-onions for oxygen reduction reaction

Kuntal Chatterjee [a, b, **], Meiyazhagan Ashokkumar [a, *], Hemtej Gullapalli [a], Yongji Gong [a], Robert Vajtai [a], Palanisamy Thanikaivelan [c], Pulickel M. Ajayan [a, ***]

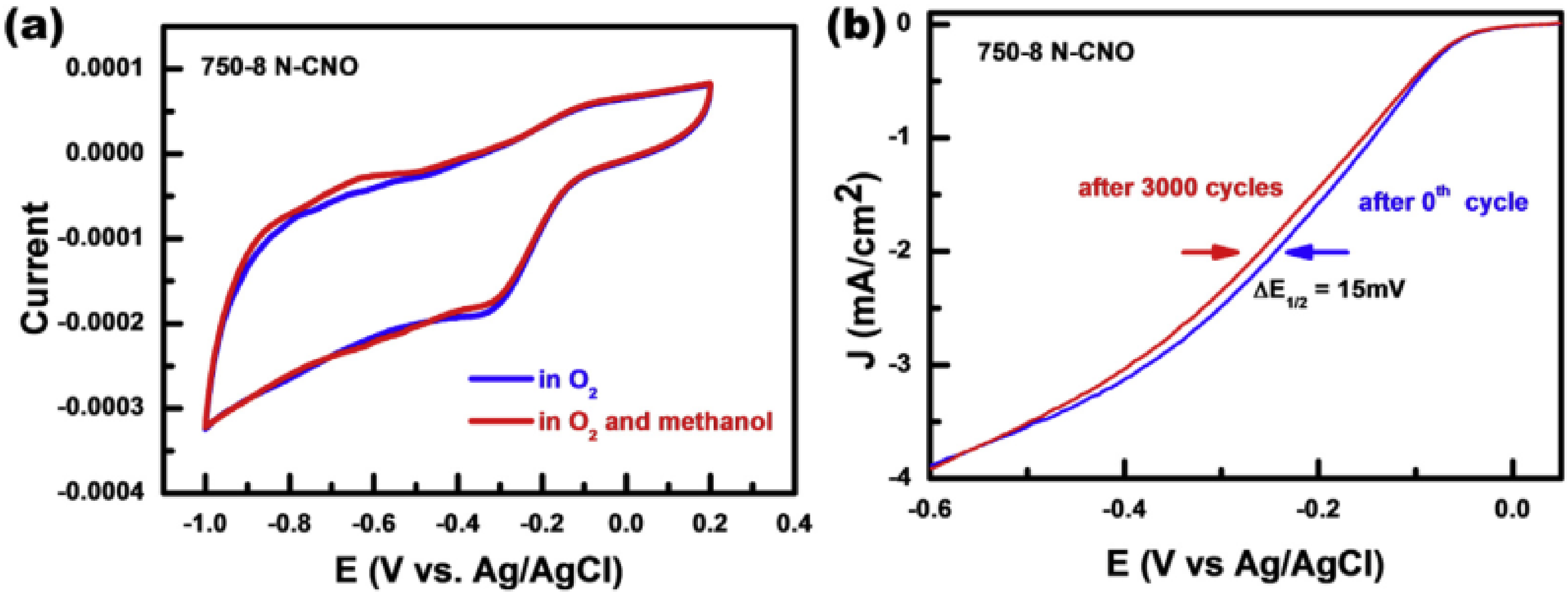
# N-rich Carbon Nano-Onions




- ***High percentage of nitrogen (7.5%) integrated into the carbon molecular skeleton***

# Carbon Nano-Onions for Oxygen Reduction Reaction (ORR)




- ***Exhibits outstanding ORR electrocatalytic activity with***
  - ***low onset potential,***
  - ***high current density,***
  - ***superior methanol crossover immunity***
  - ***better durability***

***than the benchmark Pt/C catalyst in alkaline medium***

# Biowastes to Bi-functional Fe@C Nanomaterials for Energy and Environmental Applications

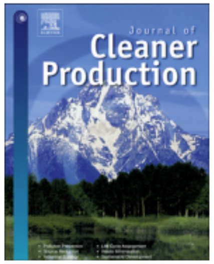
Journal of Cleaner Production 210 (2019) 190–199

ELSEVIER

Contents lists available at ScienceDirect

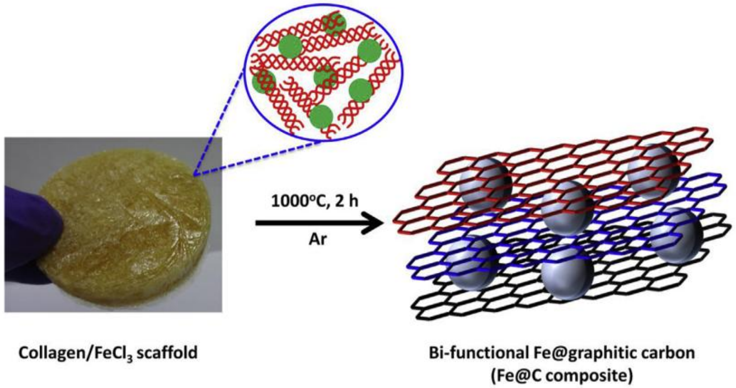
Journal of Cleaner Production

journal homepage: www.elsevier.com/locate/jclepro

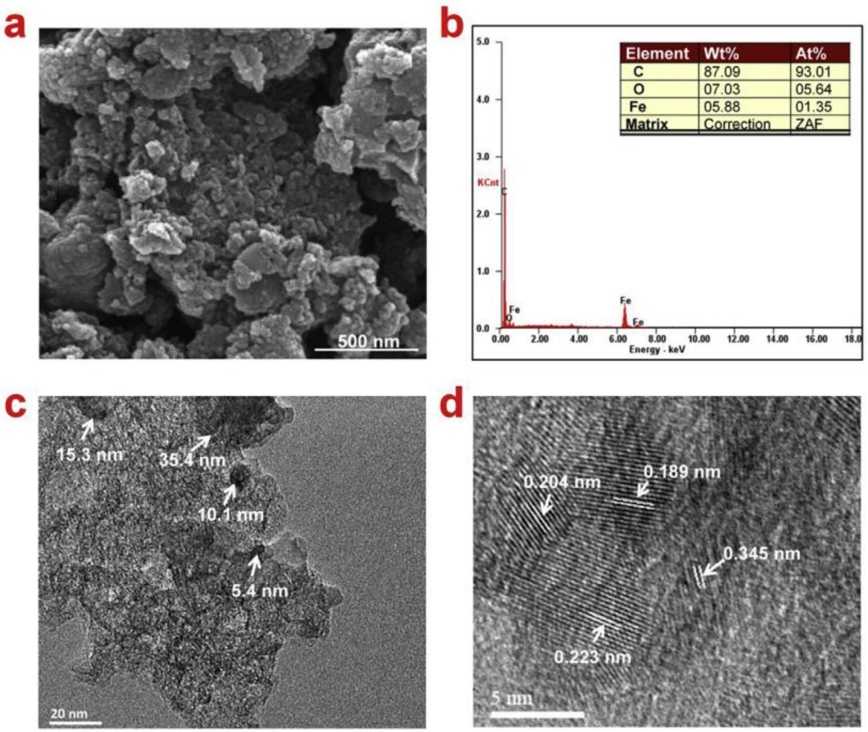


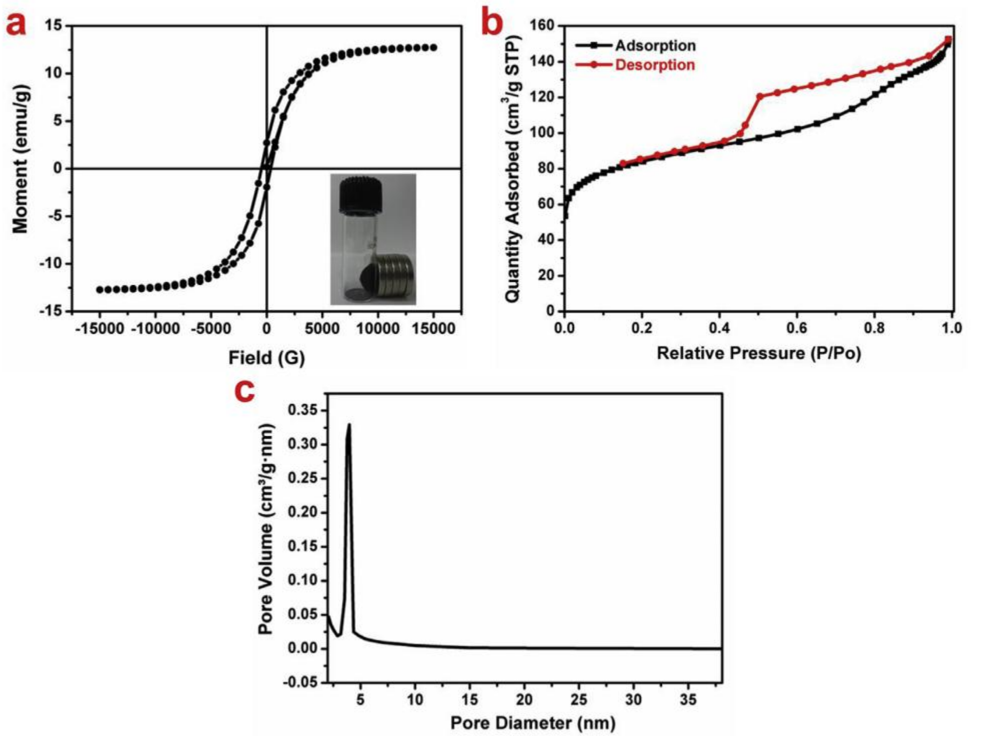

## Bi-functional iron embedded carbon nanostructures from collagen waste for photocatalysis and Li-ion battery applications: A waste to wealth approach

Berhanu Telay Mekonnen [a, b, c], Ashokkumar Meiyazhagan [d], Murali Ragothaman [a], Cheirmadurai Kalirajan [a], Thanikaivelan Palanisamy [a, b, *]



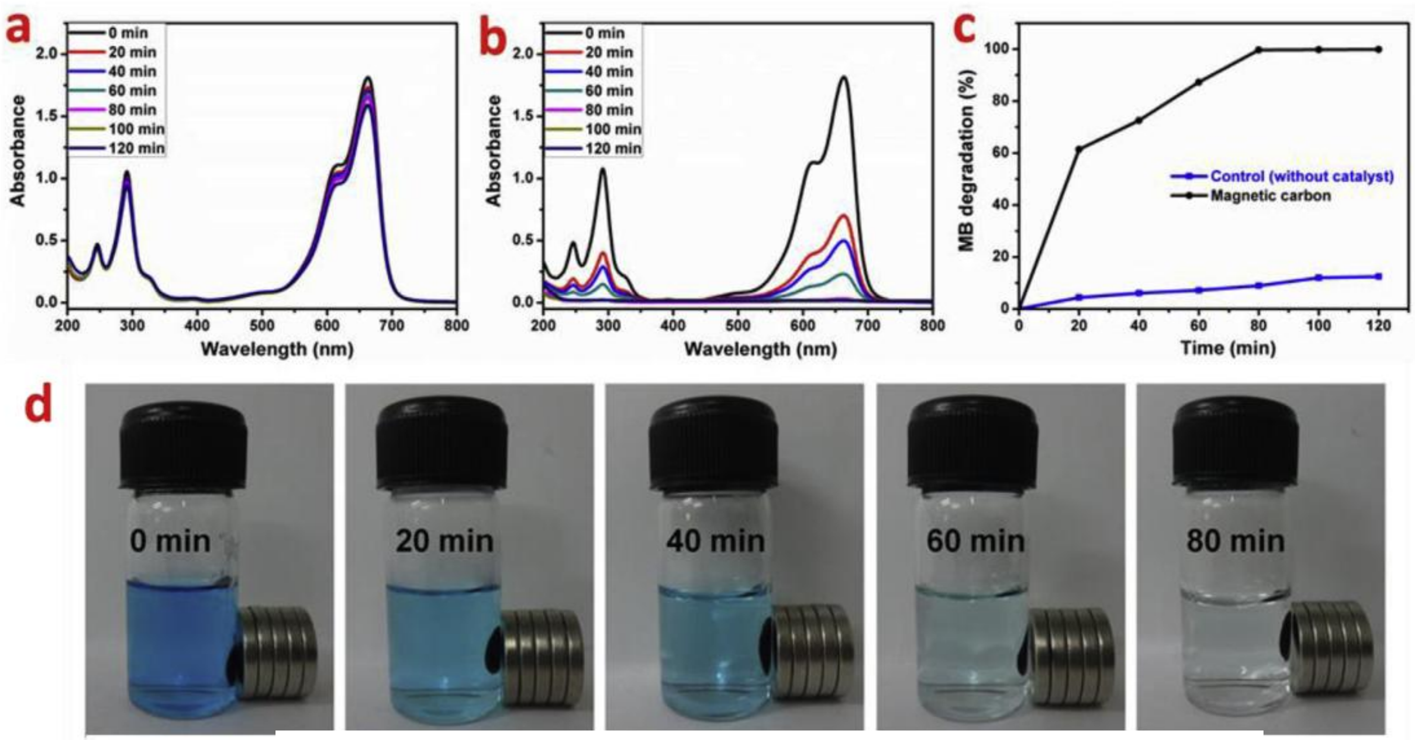
# Bi-functional Fe@C Nanomaterials from Biowastes

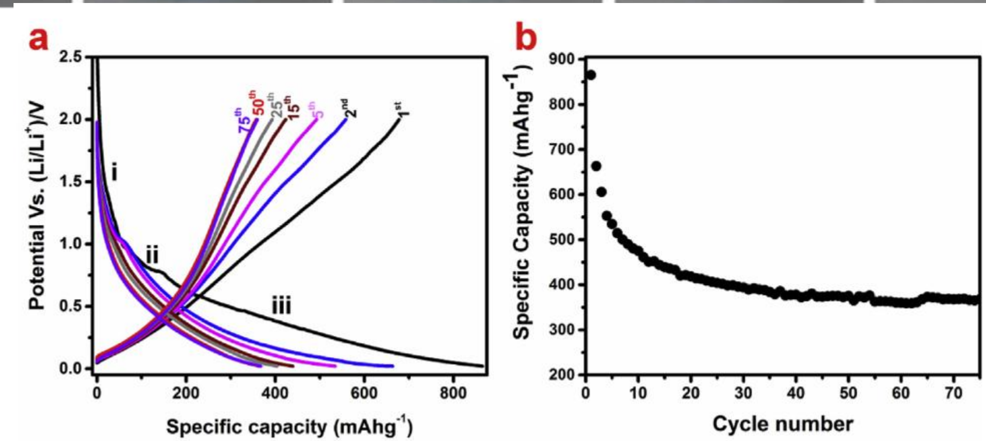



***Transformed highly insulating and paramagnetic collagen-FeCl$_3$ scaffolds into perfectly conducting and ferromagnetic bi-functional Fe@C nanoparticles***

# Bi-functional Fe@C Nanomaterials for Dual Applications



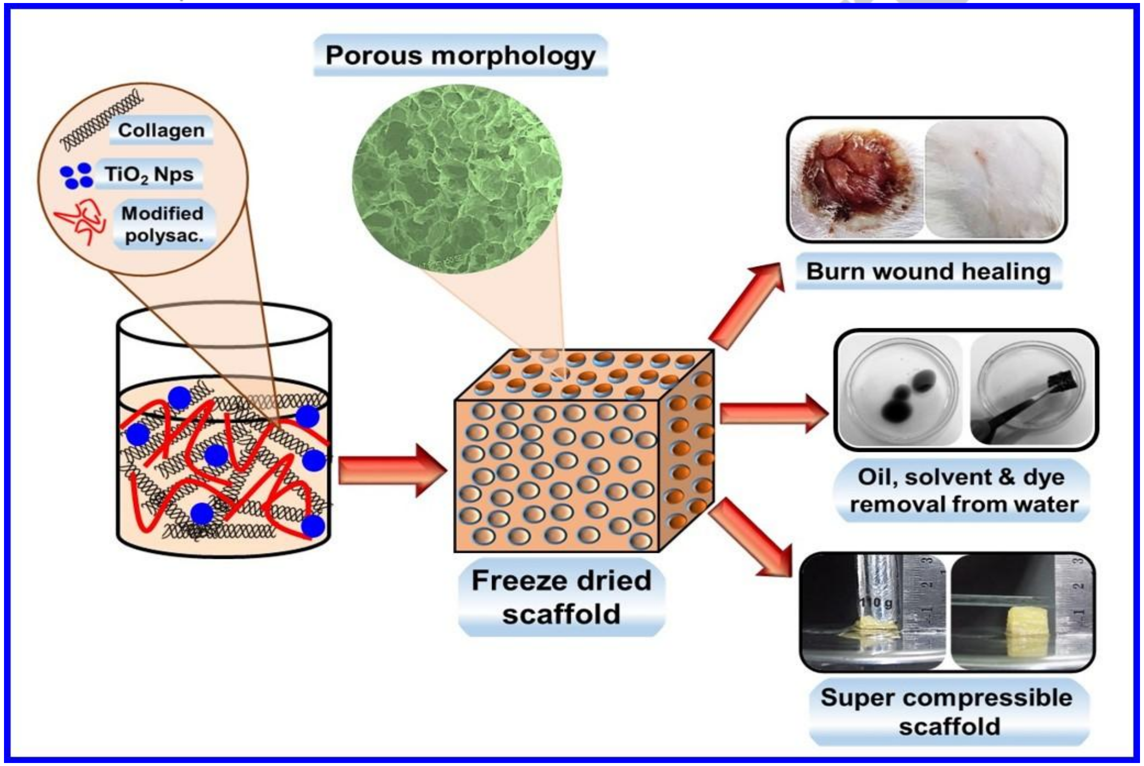

***Bi-functional Fe@C nanoparticles can be used for photocatalytic dye degradation and Li-ion battery applications***

SCIENTIFIC REPORTS

OPEN

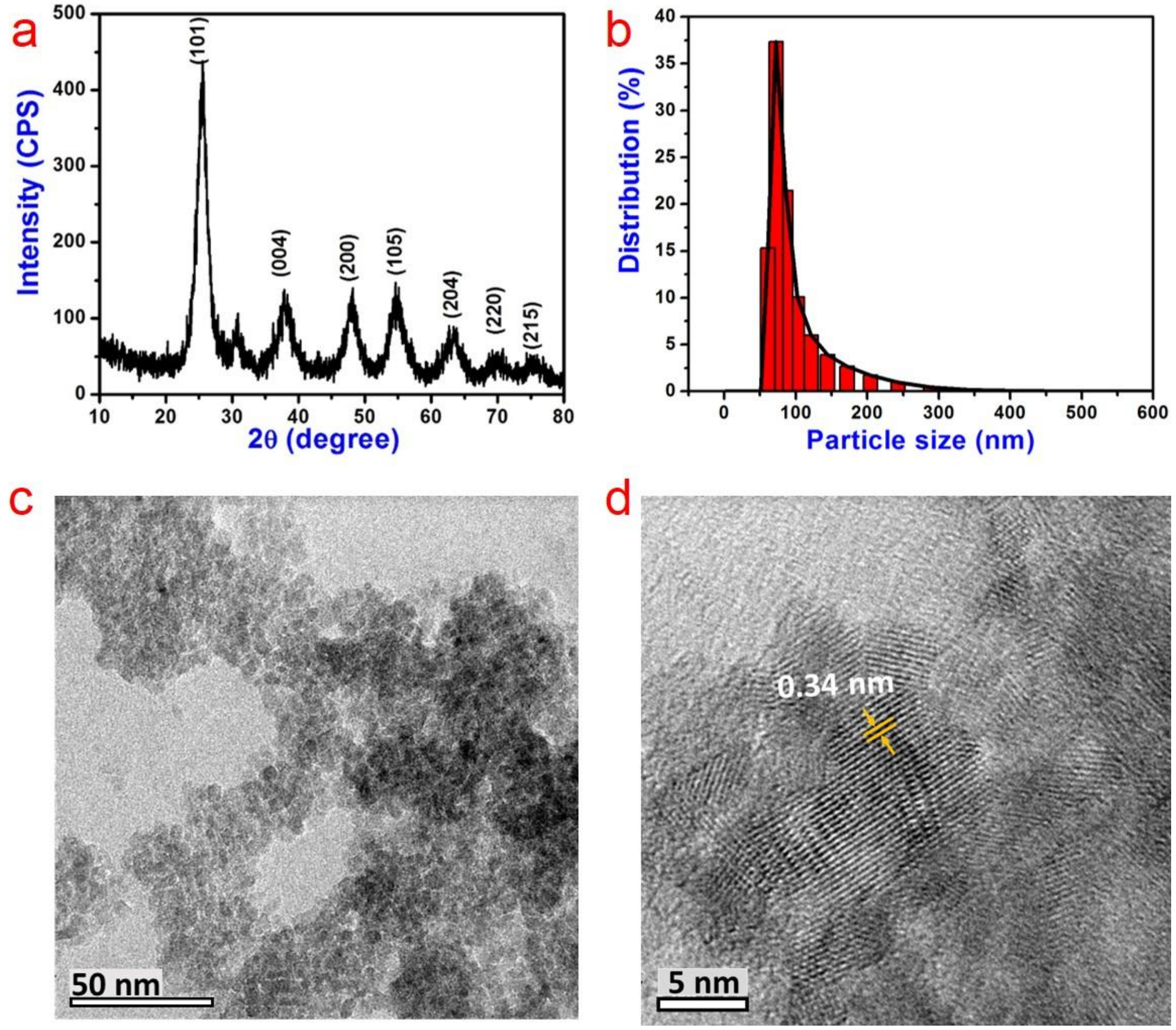
# A Facile Approach to Fabricate Dual Purpose Hybrid Materials for Tissue Engineering and Water Remediation

Received: 1 June 2018
Accepted: 14 December 2018

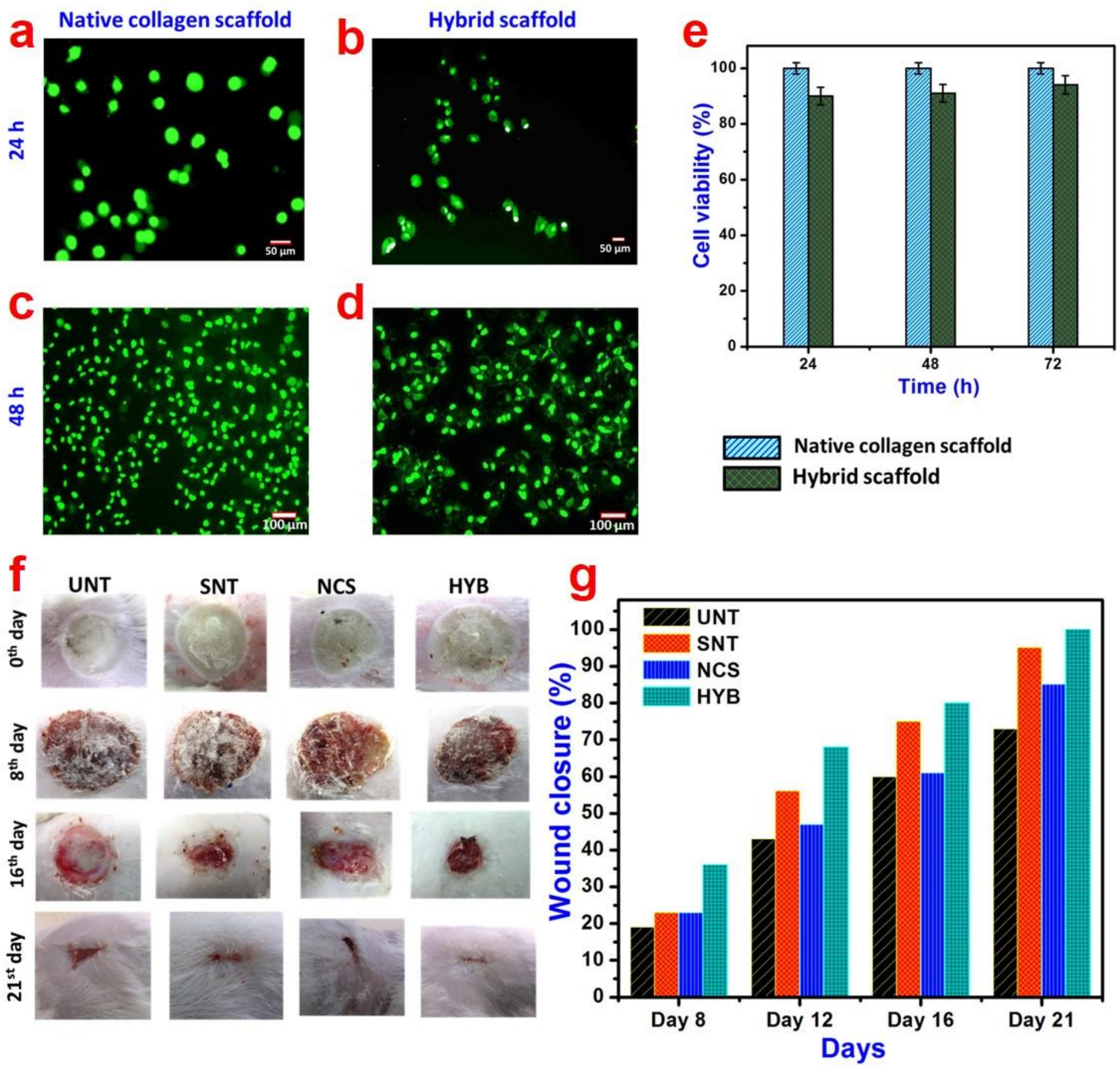


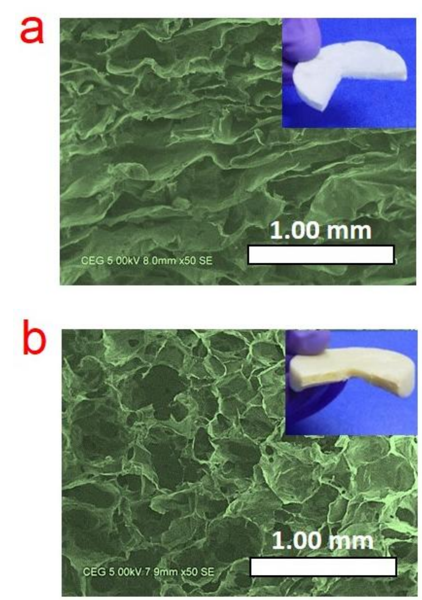

## $TiO_2$ integrated hybrid collagen scaffolds

# Structure and morphology of $TiO_2$ nanoparticles



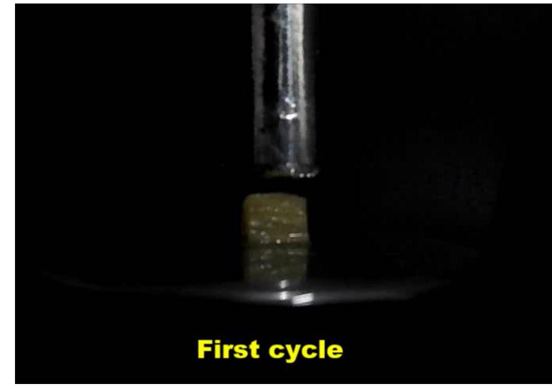
# *In-vitro* and *in-vivo* application studies of nano hybrid scaffolds



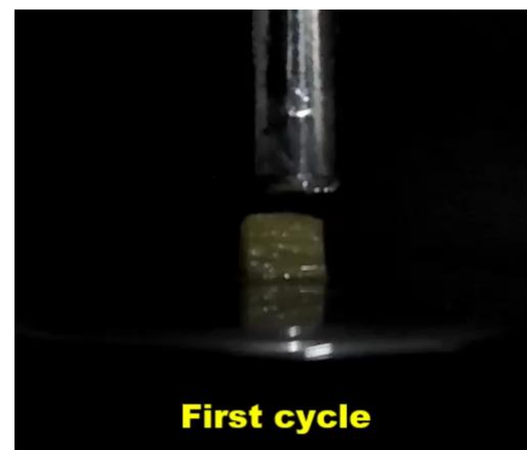




**Biocompatibility, thermal stability and antimicrobial property of hybrid scaffold enabled its application in burn wound healing**

# Water Remediation Application of Hybrid Scaffolds





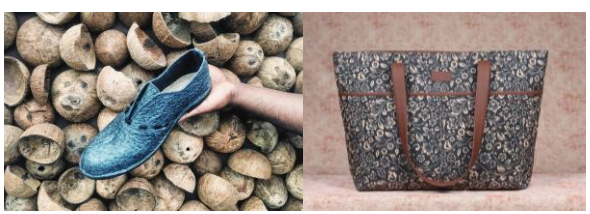
# ALTERNATE MATERIALS: LABORATORY TO INDUSTRY

## CURRENT SYNTHETIC LEATHER ALTERNATIVES ARE POORLY BIODEGRADABLE

# Vegan materials: National and international status quo

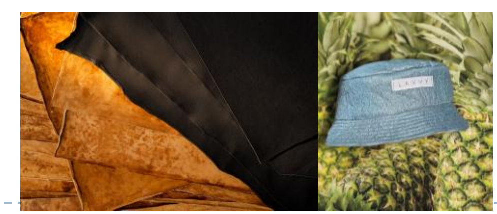
**Indian Market**

- Aulive – products made from pineapples, coconuts, and cork leather sourced from Italy
- Arture – cork fabrics from Europe
- Alternate – Faux leather
- Zouk – Jute Khadi and Ikat fabric
- A Big Indian Story (ABIS) – finest kilims, Pinatex, Cactus leather, Texon Vogue and PU
- **Malai** – bacterial cellulose from Coconut water
- **Phool** – Fleather from flower waste (dev. stage)



**International Markets**

- **Presstoff** – paper pulp
- Biocouture – bacterial cellulose
- Mycoworks and Mylo – Mycelium
- **Pinatex** – 80% pineapple leaf fibre and 20% PLA
- Modern Meadow – gene-editing yeast cells
- **Desserto** – cactus leaves
- **Sorona** – plant based 1,3-Propanediol & TPA
- **Vegea** – Wine leather from grape marc
- **Fruitleather Rotterdam** – mango wastes with natural additives & polyester fabric



# Vegan materials: Micro Structures

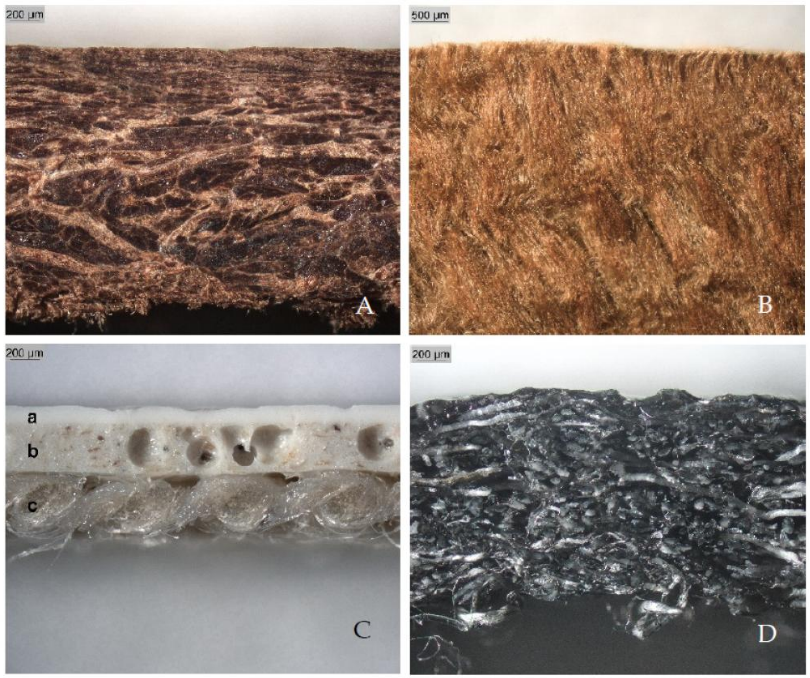
Leather

Material with a naturally grown fiber structure (Muskin® Mushroom)

Topcoat

Foamed middle layer

Textile support




Structure of a typical coated textile (Desserto)

Non-woven made of natural fibers (Pinatex)

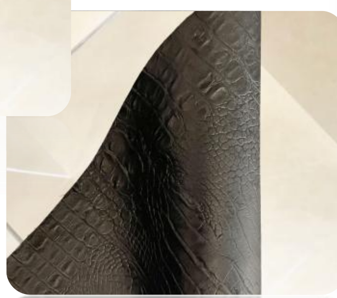
| Meyer et al., Coatings 2021, 11, 226 – FILK, Germany

# Alternate Materials: Laboratory to Industry

- Due to the growing population, limited raw materials, and environmental concerns, alternatives to leather from natural resources are high in demand.
- The available leather alternatives are predominantly synthetic materials of different chemical combinations (PVC/PU) which are eco-sensitive
- We recently developed leather-like materials from various agrowastes and transferred the technology to 3 start-ups and 1 MSME company

# Alternate Materials: Mango Leather-likes

| Properties | Fruitleather Rotterdam (The Netherlands) | CSIR-CLRI - Mango leather-likes |
|---|---|---|
| Tensile strength (MPa) | 8 | 11 |
| Tear strength (N) | 15 | 50 |
| Colour fastness – Dry rub | 4 | 4 |
| Colour fastness – Wet rub | 3 | 3/4 |
| Flexural resistance (no of cycles) | 7000 | 8000 |



**€10/sq.ft.**

**€1.5/sq.ft.**

**Patent filed and technology transferred to a start-up company on an exclusive basis**

# Plant Leather-likes : Laboratory to Industry

- CSIR-CLRI recently developed leather-like materials from agro wastes such as rice/wheat straw
- Patent filed in India and technology transferred to 2 start-ups and 1 MSME company

**A viable solution for the burning of agro-wastes**

# Bio-Content in VEGAN Leather SIMILARS: A Comparison

| VEGAN LEATHER SIMILARS | UNIT | BIOMASS | BIO-CONTENT |
|---|---|---|---|
| DESSERTO BIO+ | Percentage (%) | NK | 61* |
| SORONA | Percentage (%) | NK | 28* |
| APPLE SKIN | Percentage (%) | NK | 34* |
| CSIR-CLRI MANGO LLM | Percentage (%) | 50 | >50 |
| CSIR-CLRI PLANT LLM | Percentage (%) | 20 | 50 |

*https://www.biopreferred.gov/

# PLANT-BASED LEATHER ALTERNATIVES

- Up-cycling of plant waste
- Eco-friendly
- Easily biodegradable
- Practicable
- Market: >100 million USD in 2030
- Way forward for future

# Outcomes

- Possible ways to convert bio-wastes into useful multifunctional advanced nano/composite materials have been shown
  - Self-doped carbon nanomaterials,
  - N-rich carbon nano-onions
  - Bi-functional Fe@C nanomaterials
  - Nano-hybrid super-compressible scaffolds
  - Leather-likes
- Uses of these nanomaterials have been shown such as
  - Li ion battery applications as an electrode
  - As a catalyst in organic reactions and for EMI shielding applications
  - Environmental remediation applications
  - Lifestyle products
- Our results show that nanoscience and nanoengineering principles can be applied to transform industrial/agro bio-wastes into advanced materials for various high-value applications

# Acknowledgment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- Mr. Sachin Sharma
- Dr. M. Ashokkumar, Rice Univ., USA
- Dr. M. Murali, Univ. Oklahoma
- Dr Berhanu Telay, Ethiopia
- Dr. K. Cheirmadurai, VIT
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Thank you

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