Preparation and characterization of chitosan/bamboo charcoal/poly(methacrylate) composite beads and its Adsorption to Creatinine

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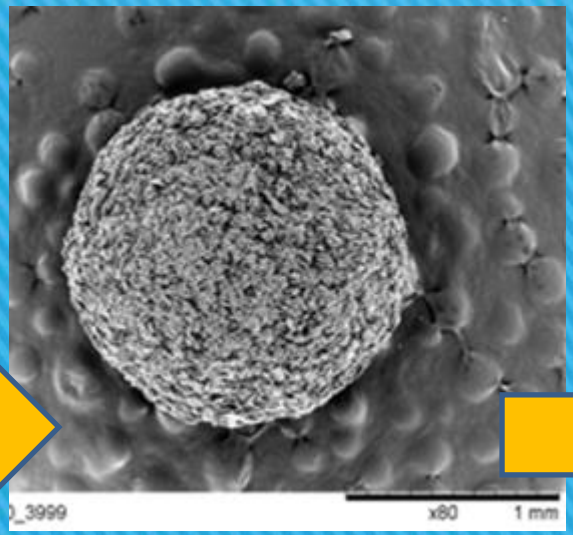
13th Philippine National Health Related System
August 13,2019
Luxe Hotel, Limketkai, Cagayan de Oro City

Outline

Introduction

& Motivation



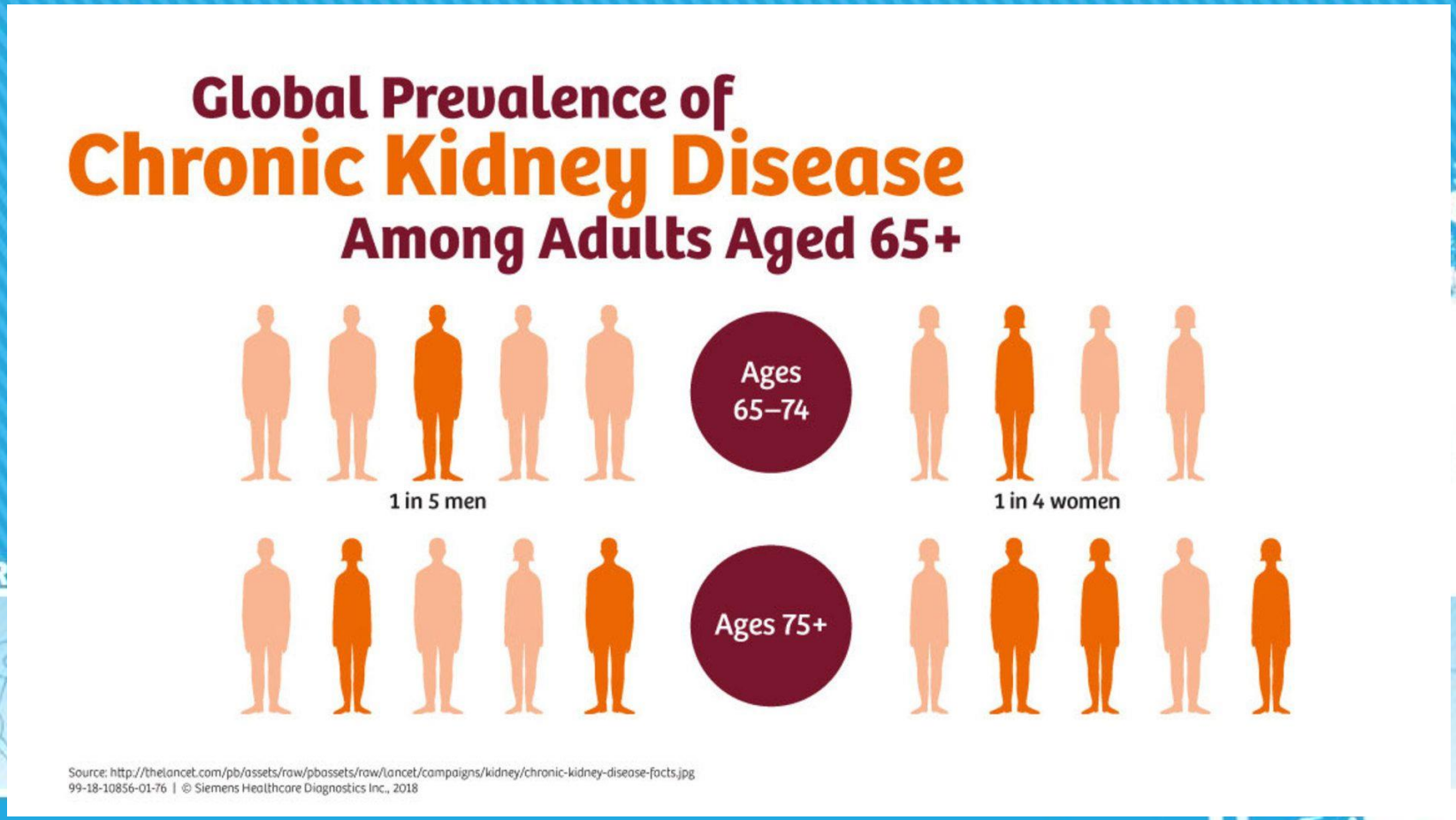
Preparation

Surface Properties

Application

Recommendations

10% Global Population has CKD



http://global-disease-burden.healthgrove.com/l/67119/Chronic-Kidney-Disease-in-Philippines

Prevalence of Chronic Kidney disease in the Phil

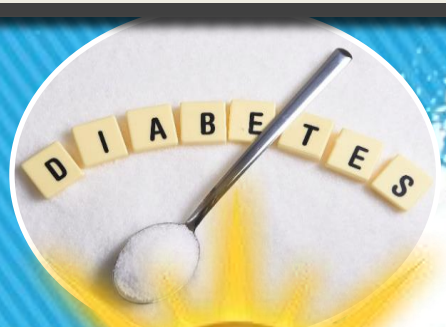
mortality rate has increased from 11 K (2013) to 14 K (2014)

CKD in 2005

2.6% (1.2M)

CKD in 2014

9.4% (6M)



41%

Now, Cause of CKD

Cause of CKD in the Past: Chronic glumerolunephritis

- NNHeS 2003-2004 Renal Report
- http://www.nkti.gov.ph/kidney_health.do.Kidney Health Plus
- National Statistics Office
- abs-cbnnews.com

The Philippine College of Physicians
Philippine Society of Nephrology

Current treatment for CKD

Bamboo Plants

Low cost

Renewability

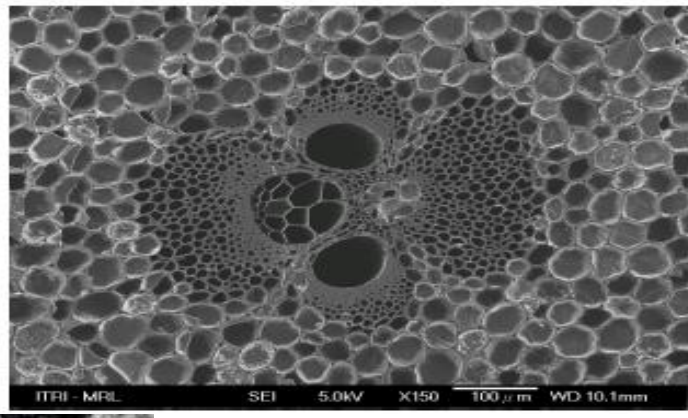
Eco-friendly

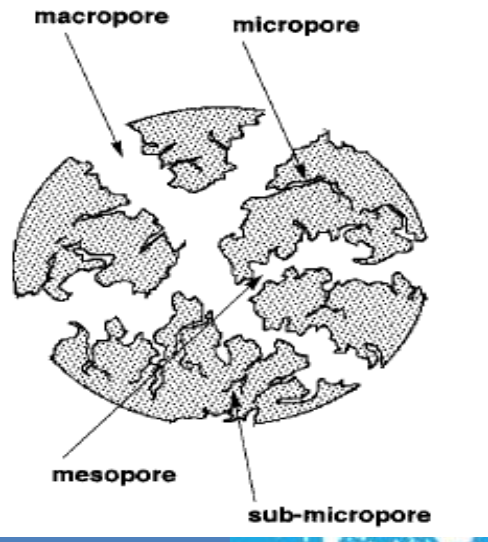
Asada T, et al. *Journal of Health Science*, 48(6): 473--479.
Mizuta K, et al. *Bioresource Technology*, 95(3): 255--257.

Bamboo charcoal (BC)

Applications: Adsorbent

- ✓ Organic, inorganic, toxic contaminants from aqueous solutions,
- ✓ Nanotechnology,
- ✓ Biomedicine, etc.





Properties

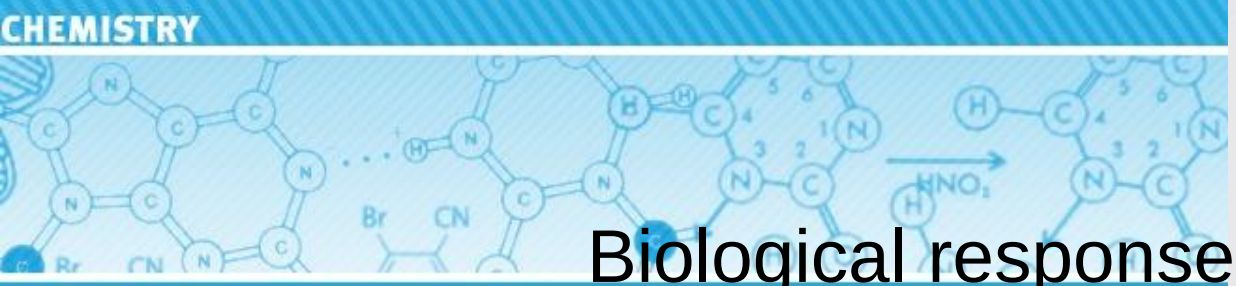
- ✓ High surface area
- ✓ High adsorption potential
- ✓ Acid-base functional groups
- ✓ Unique multicellularity

Asada T, et al. *Journal of Health Science*, 48(6): 473—479, Mizuta K, et al. *Bioresource Technology*, 95(3): 255—257; Ming, Fa Hsieh et al. (2007)

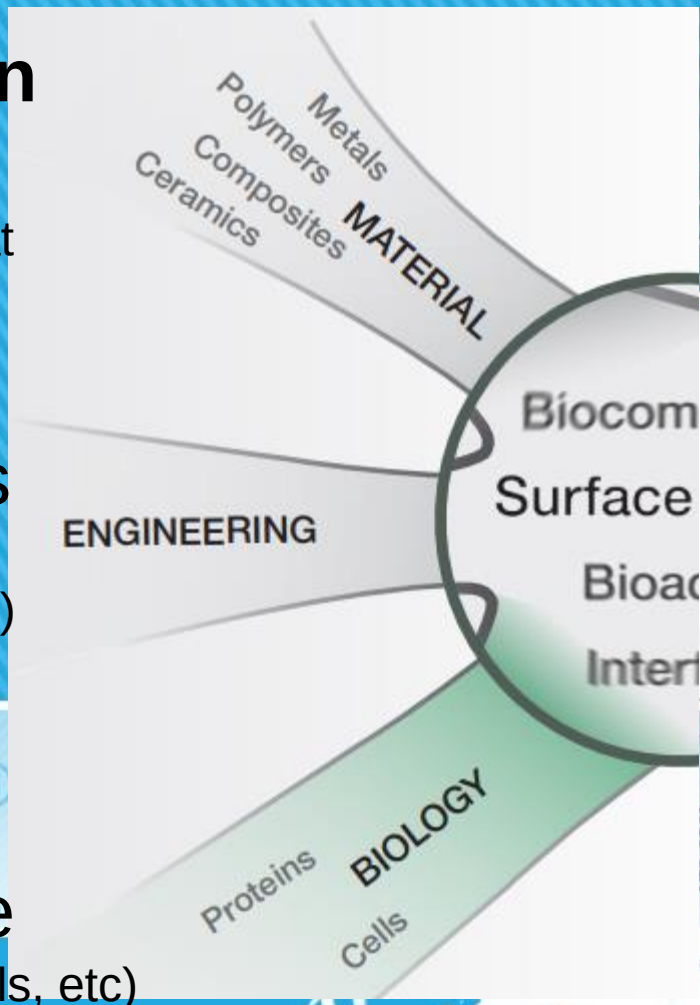
Understanding the Surface Properties of Biomaterials

Performance evaluation
Chemical properties
(Surface chemistry is distinct from that of the bulk material)

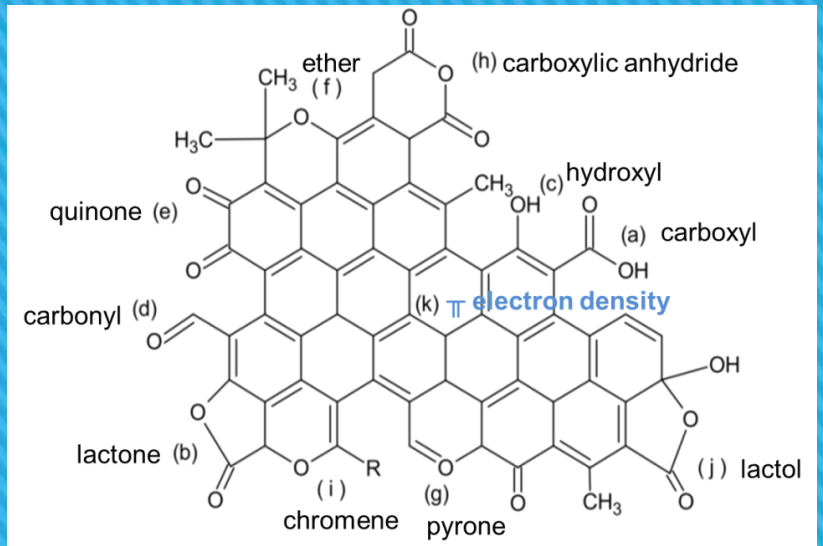
Fabrication protocol, design process
(Tailoring and controlling surface properties is therefore a major challenge in biomaterial engineering)



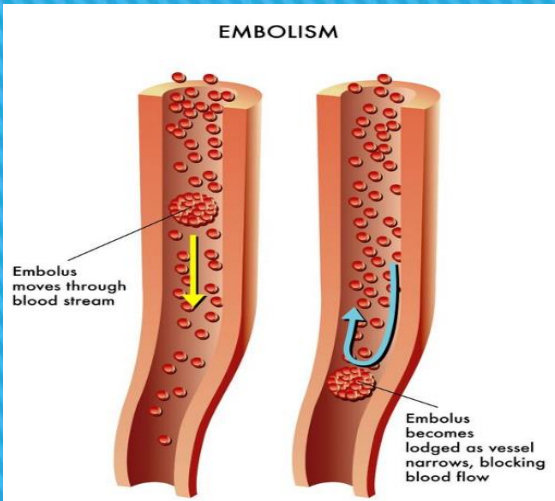
Biological response
(Materials interact with proteins, cells, etc)



Studies in the past…Bamboo charcoal powder



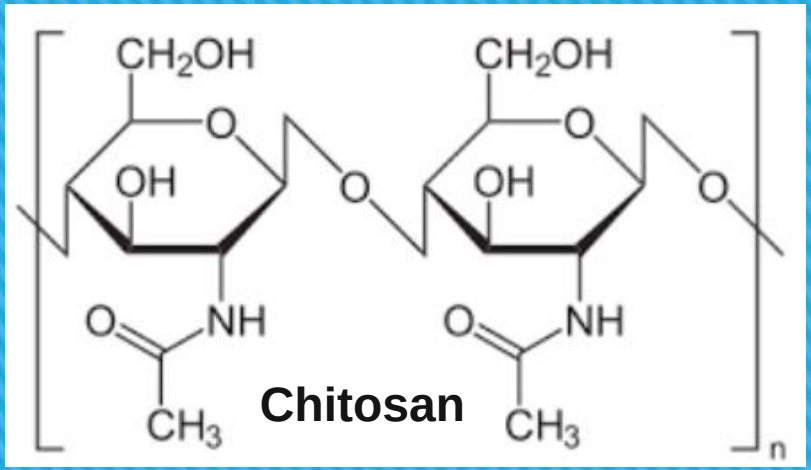
- ✓ BC surface: non-polar
- ✓ Solution was highly basic (pH12)



- ✓ BC Powder when used as adsorbent for hemoperfusion – risk for embolism

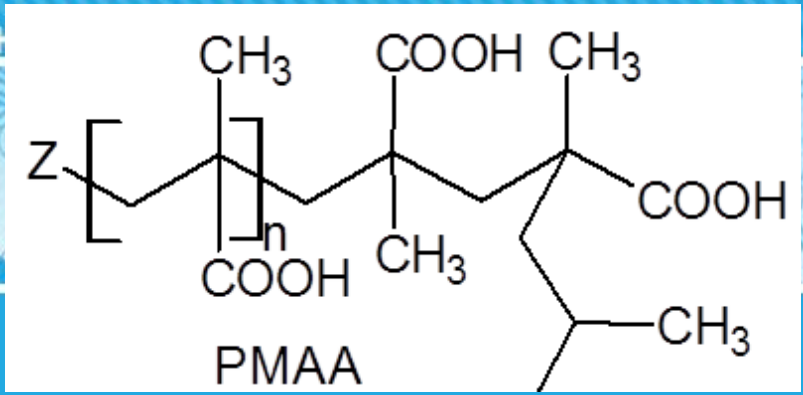
Hsieh, M.Fet.al. Journal of Medical and Biological Engineering. 2007, 27(1), 47-51.

Studies in past …Bamboo charcoal / AC Beads….



Chitosan

- Biodegradable & biocompatible
- Nontoxic polymer
- Used as coating to BC (CTS/BC) beads
- **Minimizes emboli**
- **Adsorbed Phenylalanine > albumin**



PMAA

- Coating to pharmaceuticals and AC
- **Smooth coating**
- **Mechanical strength**
- **Accessible to polar substances**

- Jager M, and Wilke A 2003 *J. Biomater. Sci., Polym. Ed.* **14,** 393 1283
- Hsieh, M.F.et.al. Journal of Biomedical Materials Research. Part A. 2010, 94(4), 1133-1140.

OBJECTIVES

1

Prepare beads

Surface characteristics

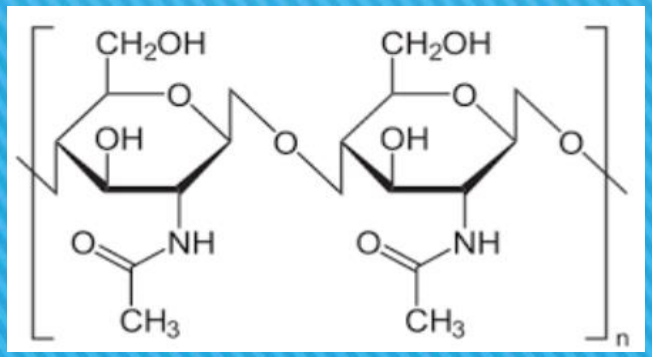
2

Application:

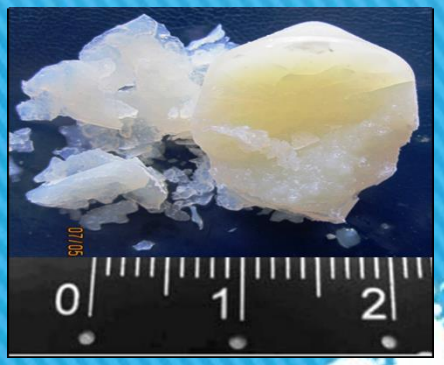
Dynamic Adsorption of Creatinine

Composition of Beads….

BC



Chitosan



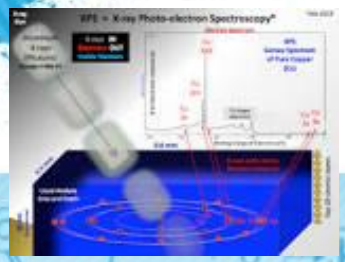
Poly(methacrylate)

Surface Properties of beads revealed by

SEM

BET

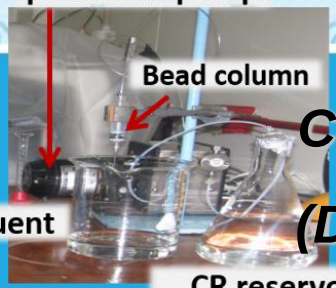
DSC & TGA



XPS

Boehm Titration

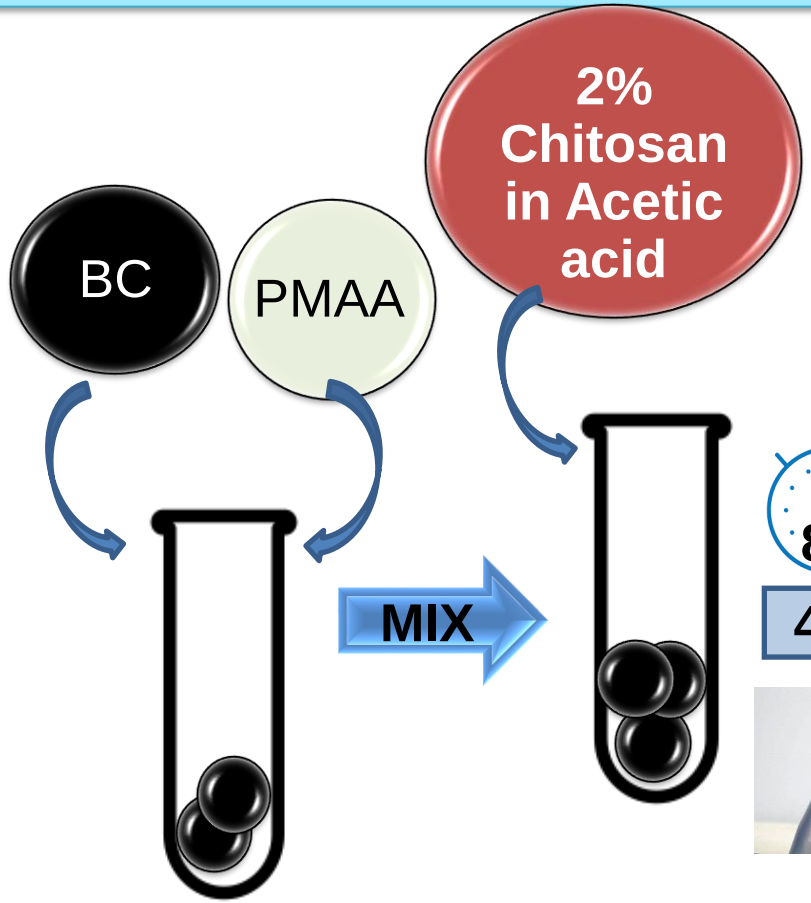
pHpzc

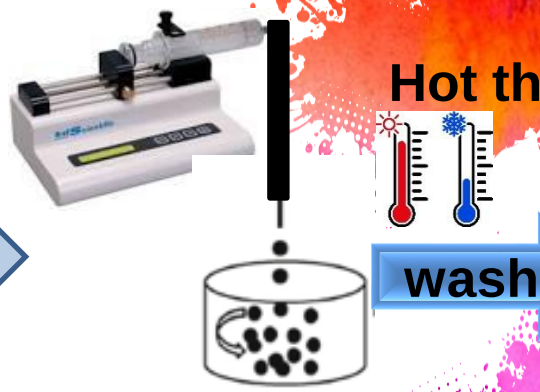


Creatinine Capture

(Dynamic Process)

Preparation of the Beads





Hot then Cold

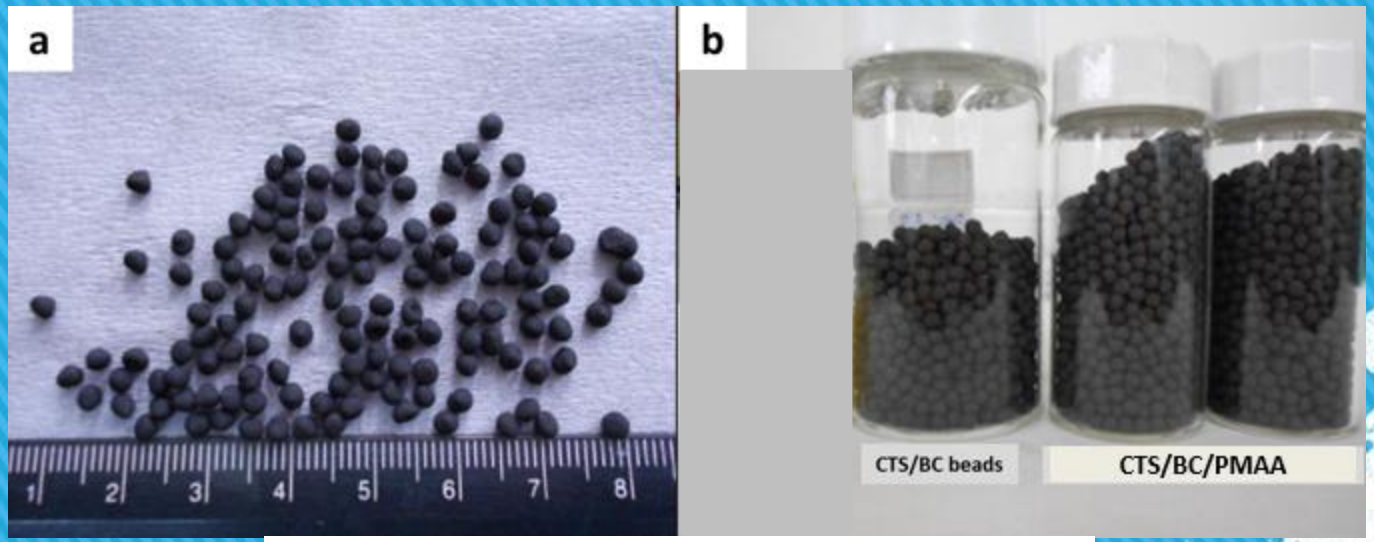
wash

Dried 65 °C

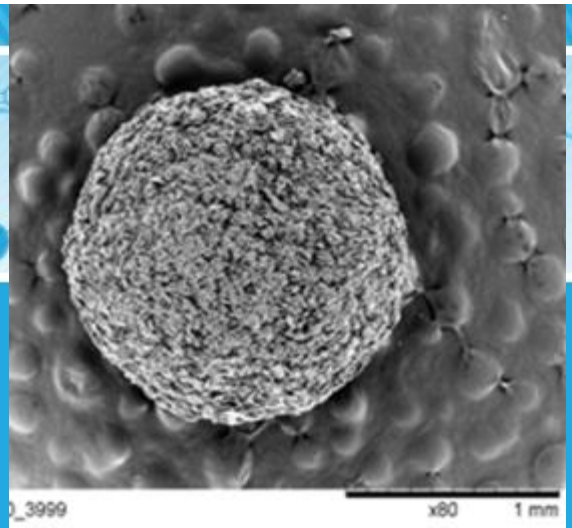
1.49 mm diameter

The Prepared Beads…

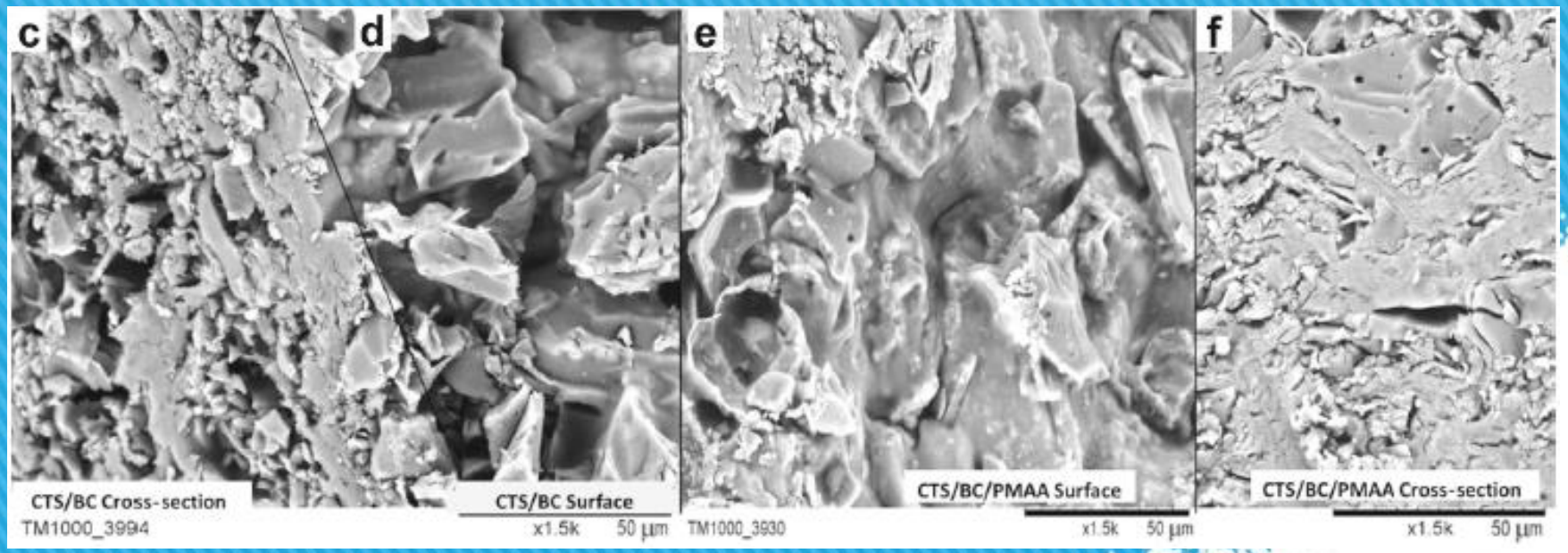
Optical photos



SEM Image@ x80

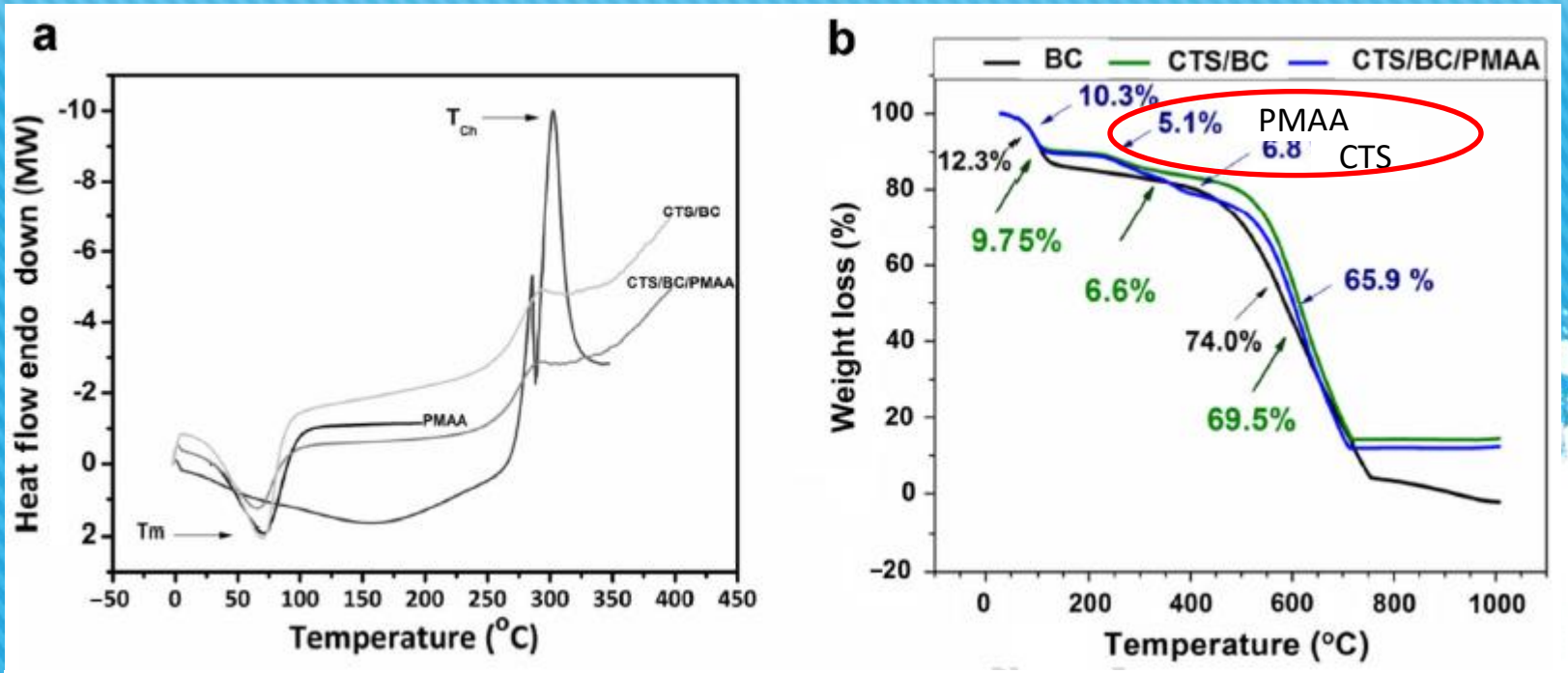


Surface Morphology



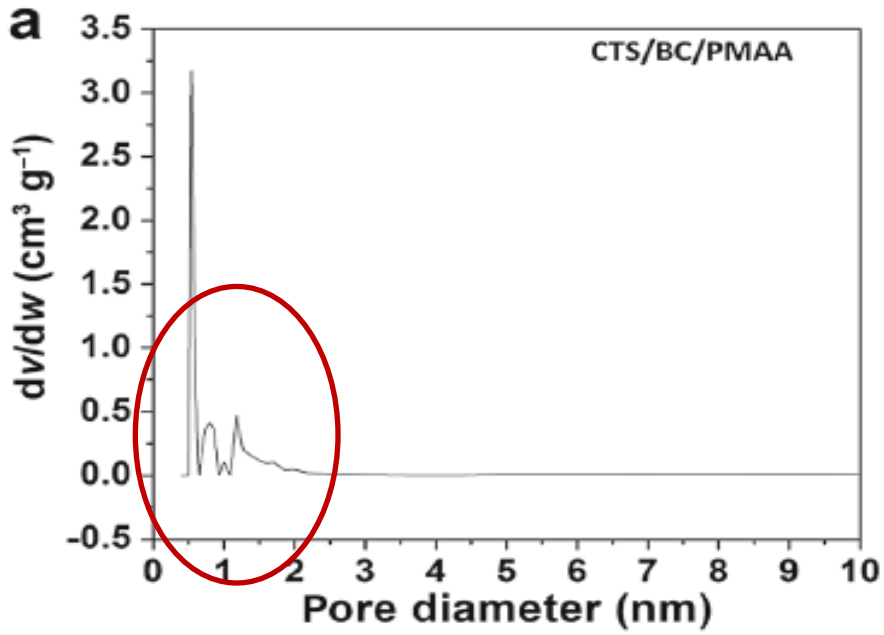
Uniform distribution of Chitosan and PMAA

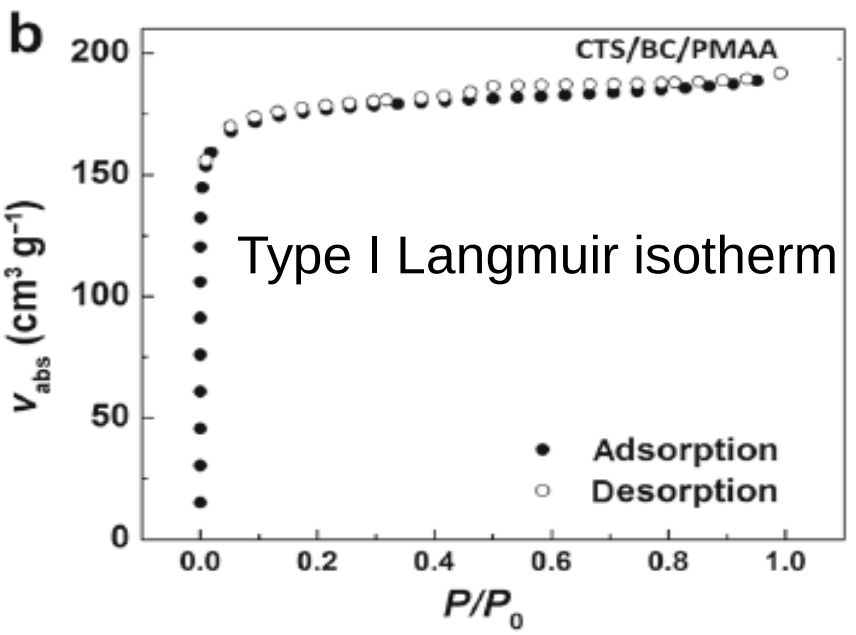
Homegeneity and Thermal Stability…



- ✓ Sharp endothermic melting temperature, *T*m peak at **74.6◦C (T_mPMAA)**
- ✓ **Uniform molecular weight, MW within the sample**
- ✓ **CTS/BC/PMAA (T_m66.77)**
- ✓ **T_{deg} CTS/BC/PMAA > CTS/BC**
- ✓ **higher degree of thermal stability**
- ✓ CTS/BC beads, CTS started to decomposed at temperature 100 - 382°C and burnt at 724° C
- ✓ CTS/BC/PMAA, CTS decomposed @ 306−465 °C and burnt at 746° C.

Surface Area & Pore size Size Distribution



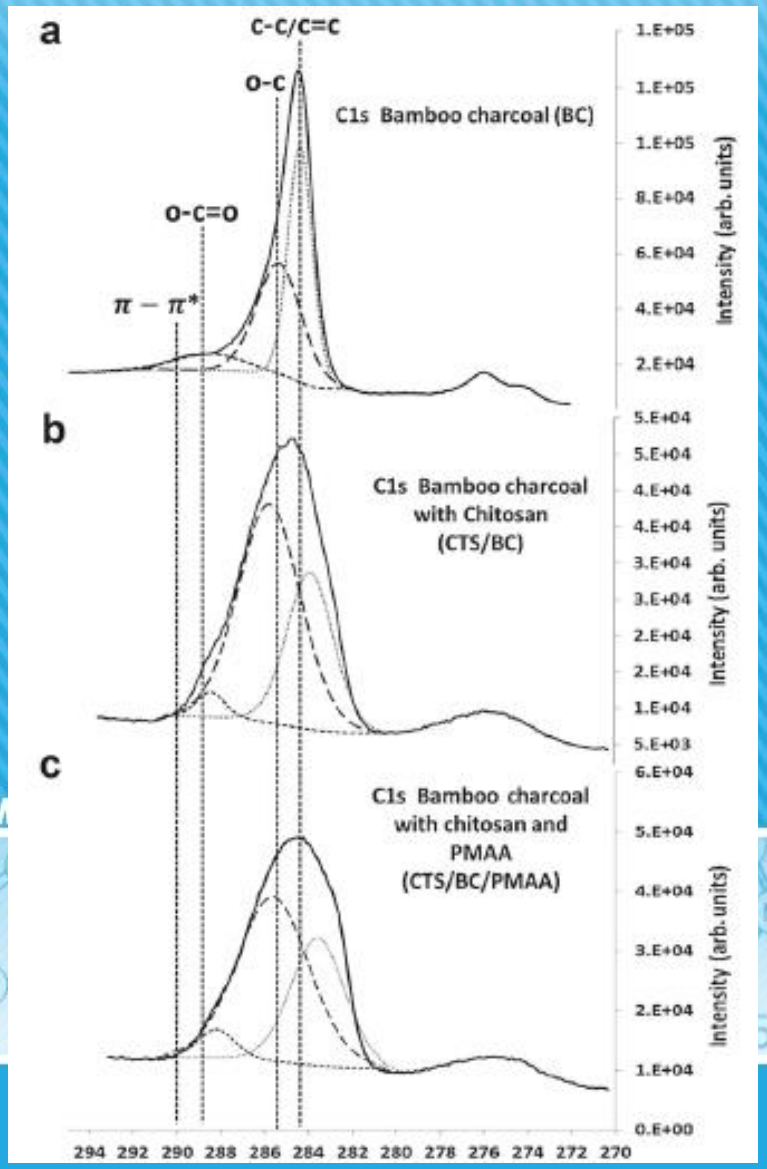


higher occurrence: micropores;
average pore width: less than 2 nm.

SSA: BET (681.15 m^2/g)
Langmuir (773.34 m^2/g)

(IUPAC definition :
micropores <2 nm,
mesopores 2-50 nm,
macropores >50 nm) [11].

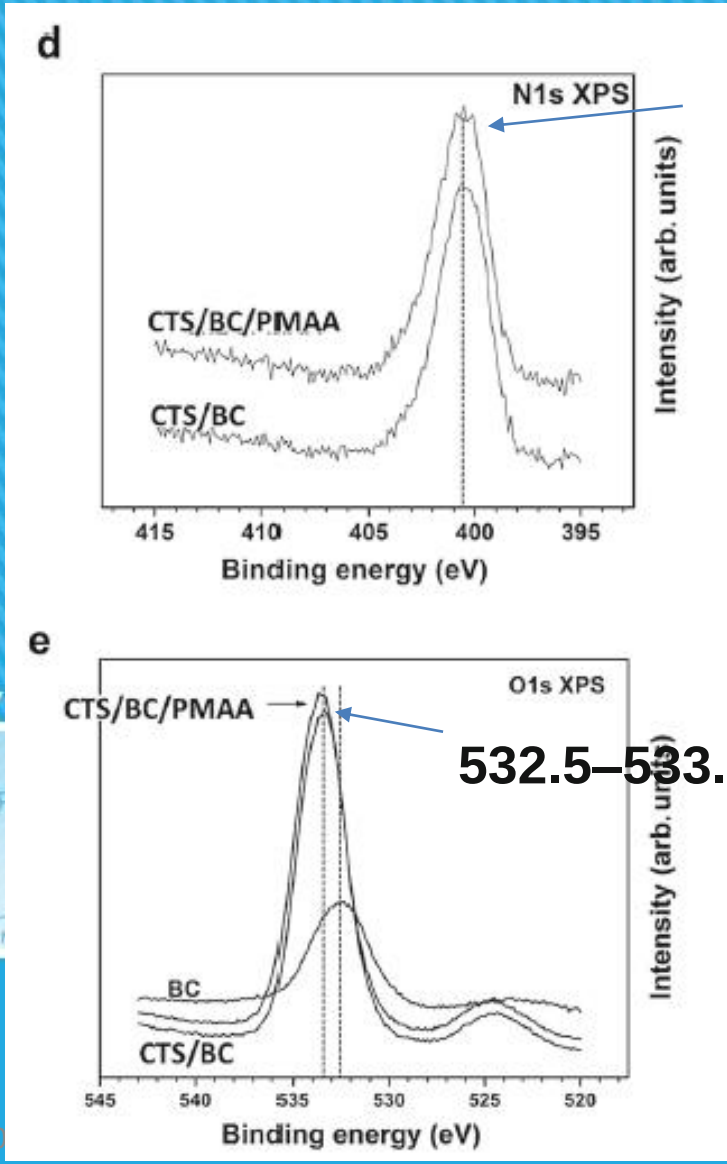
Surface Functional Groups : X-Ray Photoelectron Spectroscopy (XPS)



Adsorbent	Functional groups Electronic binding energy (eV)			
	pi-pi*	COO	C-O	C-C/C=C
BC	290.0	289.0	285.5	284.5
CTS/BC	-	288.5	285.6	284.0
CTS/BC/ PMAA	-	288.0	285.8 or (C=N)	283.6

- ✓ surface binding state of C1s shows graphitic carbons, COOH or COOC between 285 and 289 eV.
- ✓ **Acidic Properties**

Surface Functional Groups : XPS analysis



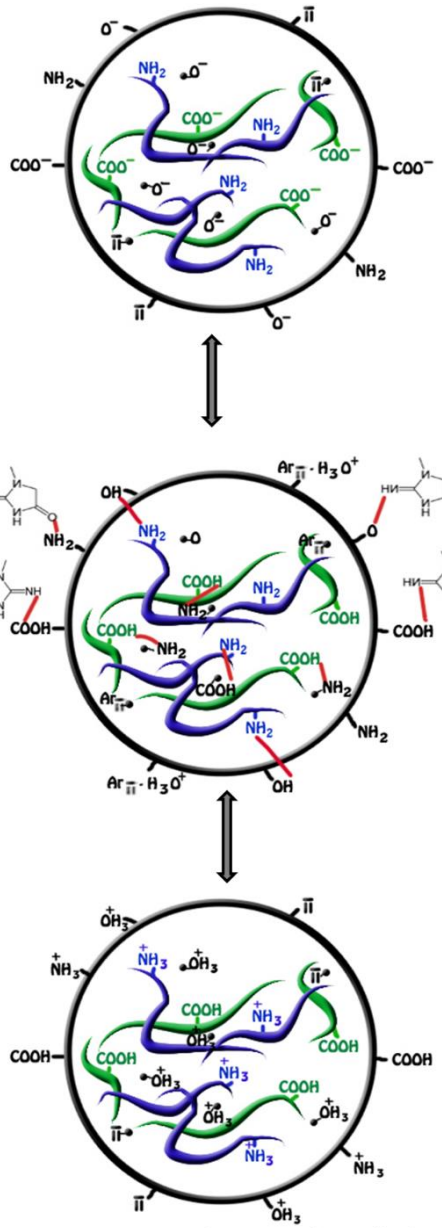
398–403 eV , sp^2 or sp^3 N

- 532.5 eV found in BC shifted to higher BE (533.5, 534.0 eV) for CTS/BC and CTS/BC/PMAA, respectively
  - ✓ increase in O content in the composite beads (C–O and O–C = O)

532.5–533.5 eV ,C-O

Results & Discussion

Surface Charge

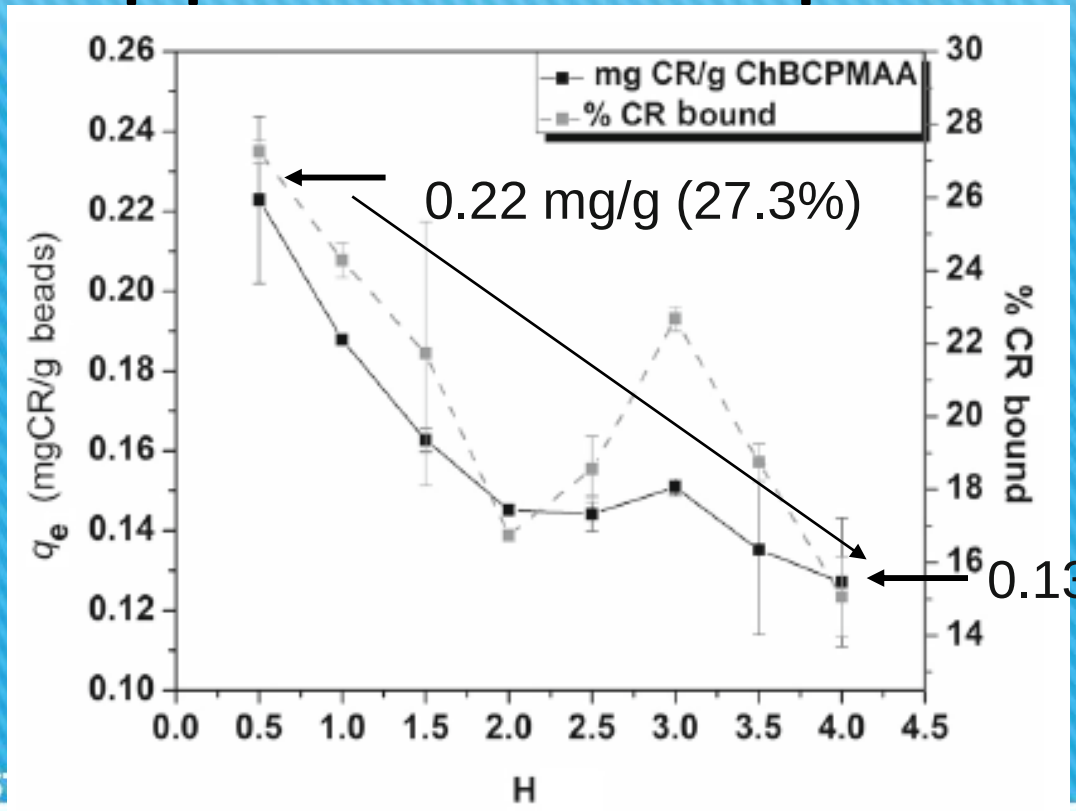


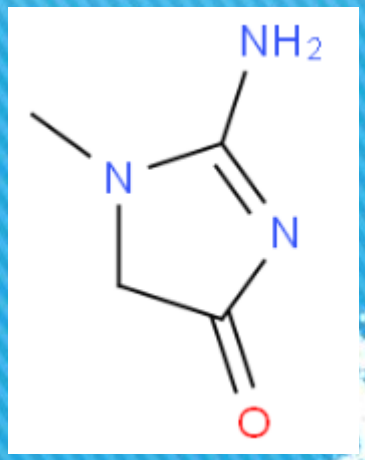
Negative

pHpzc = 6.70

Positive

Application : Capture of Creatinine





Dynamic adsorption of creatinine (CR) using 40 mg beads for 4 hrs continuous flow through of 100 mg/L creatinine (at pH 7 in phosphate buffer) using a peristaltic pump at a flow rate of 5 ml/min

- ✓ Decreased of only 12.2% from 0.5 H to 4 H of dialysis
- ✓ **Beads have not yet been exhausted**

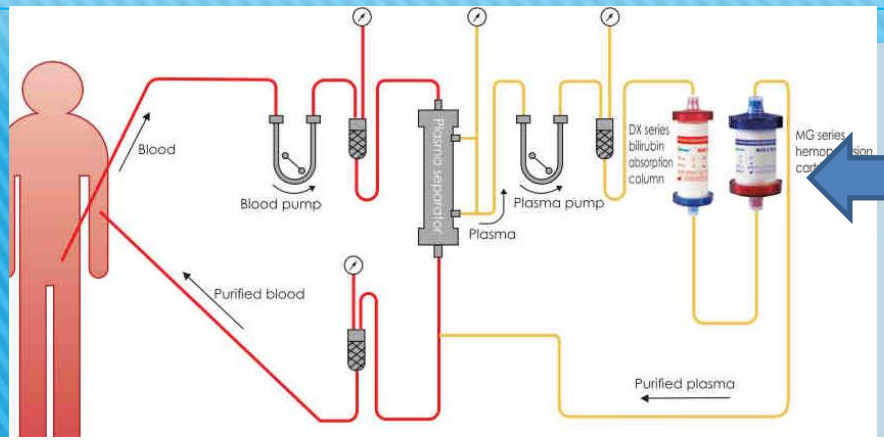
Conclusion

- ✓ Beads compose of CTS/BC/PMAA were prepared and characterized
- ✓ BET and Langmuir revealed high SA
- ✓ High occurrence of micropores with pore diameter of less than 2 nm.
- ✓ Boehm titration, XPS, solution pH (6.46) and pHzc (6.70) showed acidic surface of CTS/BC/PMAA beads

Conclusion

- ✓ The uniform distribution of CTS and PMAA in the composite beads was revealed by SEM.
- ✓ Successful coating of PMAA and CTS to neat BC was revealed by TGA, 5.1 wt.% of PMAA has been coated to CTS/BC/PMAA and 6.8 wt.% of CTS
- ✓ Coating of BC with PMAA and CTS rendered the composite beads with mechanical strength as indicated by low C particles released in the solution.
- ✓ Capture of aq.Creatinine showed a decrease of only 12% from 0.5 to 4 h of dialysis.

Recommendation and Future Direction

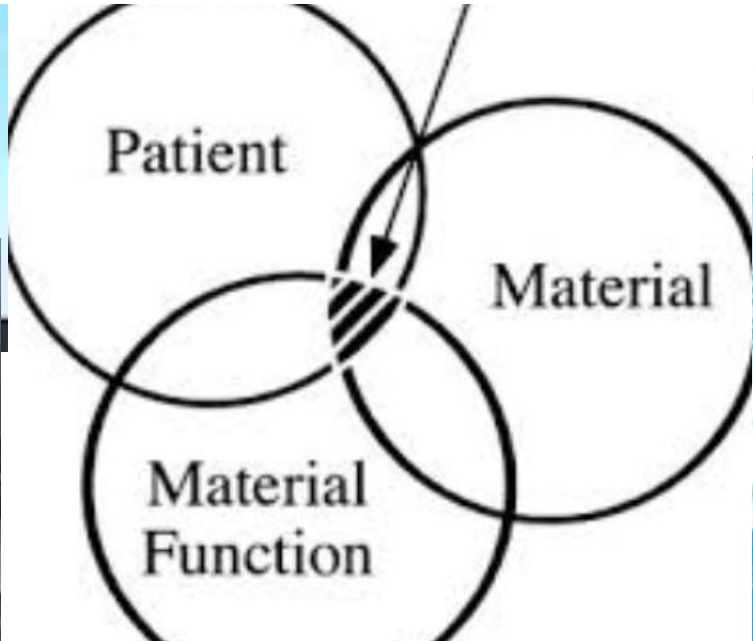


Column Adsorbent for hemodialysis and desorption studies

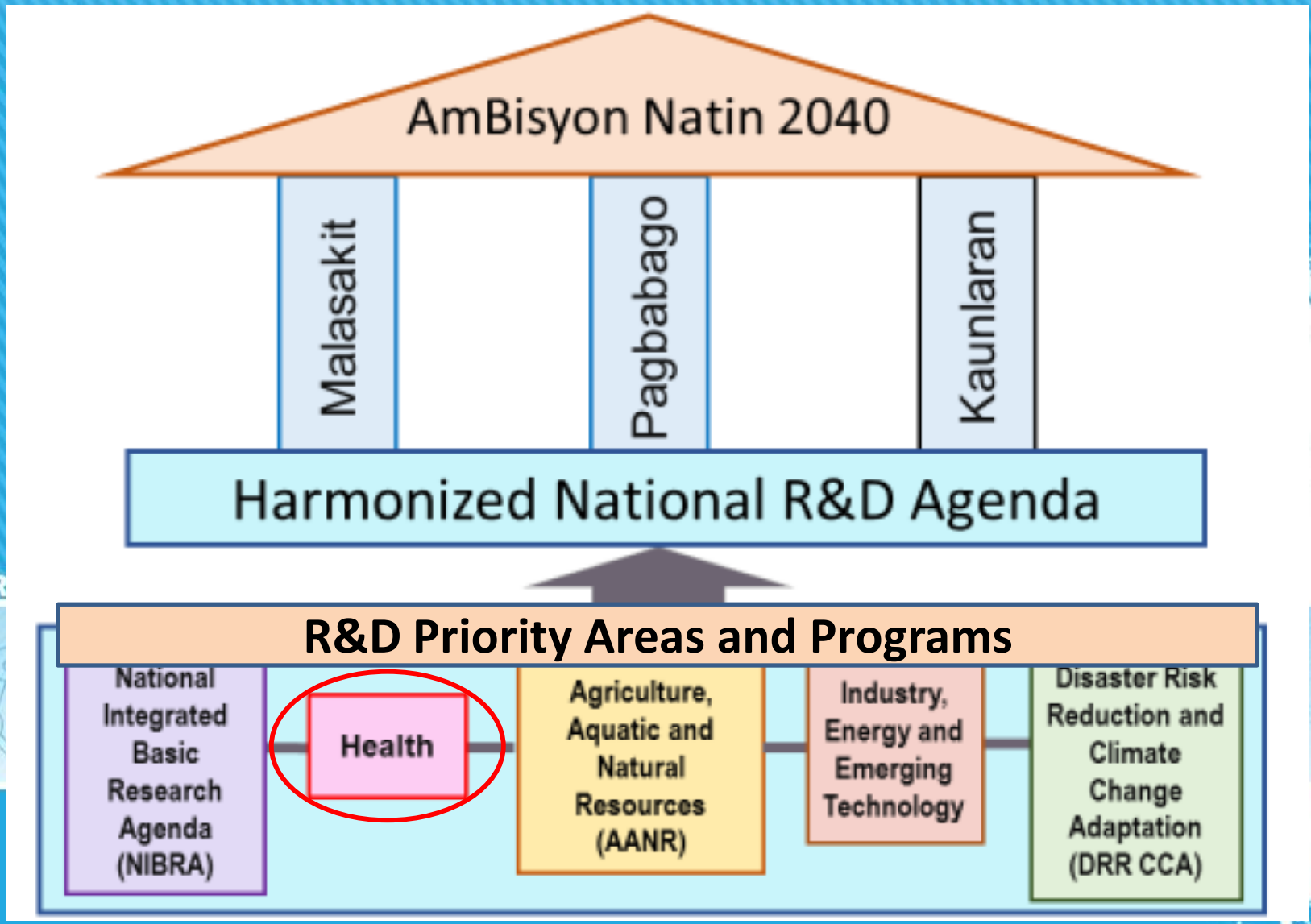
Oral application

Coat with Heparin

Biocompatibility



Biomedical Devices : HNRDA (2017-2020)



Publication…

Bull. Mater. Sci., Vol. 40, No. 6, October 2017, pp. 1179–1187
DOI 10.1007/s12034-017-1464-3

CrossMark

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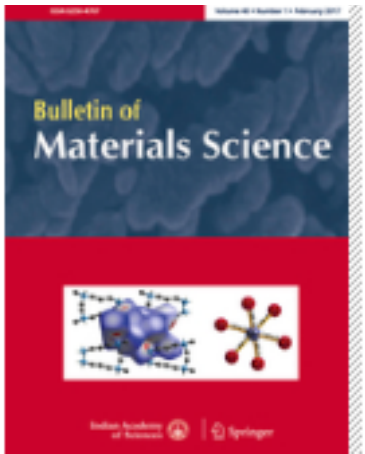
*Author for correspondence (dperuelo@xu.edu.ph)



Bulletin of Materials Science

Published by the Indian Academy of Sciences

Editor-in-Chief: G U. **Kulkarni**

ISSN: 0250-4707 (print version)
ISSN: 0973-7669 (electronic version)

Journal no. 12034

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[12] DOST, Harmonized Research & Development Agenda 2017-2022.

ACKNOWLEDGMENTS

- CHED, for the scholarship and sandwich program in Taiwan;
- My research mentors, Prof. Ronaldo M. Fabicon (AdMU)
- Ming-Fa Hsieh,PhD.,Chung Yuan Christian University (Taiwan)
- Regina C. So,Ph.D., Department of Chemistry (AdMU)
- Jose Mario A. Diaz, Ph.D., Department of Chemistry (AdMU)
- Prof. Clovia Holdsworth of the University of New Castle, Australia
- Prof.Rafael Luque & Manuel Rodriguez, Universidad de Cordoba, Spain
- Rick Arneil Arancon, Universidad de Cordoba, Spain
- Elvis T. Chua, University of Queensland, Australia
- Xavier - Ateneo, my home institution, study leave grant
- XU- Chemistry Department faculty and staff.
- Dr. Heide R. Rabanes, Dean of Research & Dr. Juliet Dalagan, AVP
- Dr. Ma.Cleofe N. Badang and Dr. Analyn C. Asok
- My husband, Roldan Peruelo; my kids, Ruthie, Rex and Francis

To Mama Mary

To GOD, be the GLORY & PRAISE.

"Barbecued dessert, anyone? The coals are PERFECT now!"

THANK YOU FOR YOUR ATTENTION!

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