Synthesis and characterization of amine functionalized cellulose-silica composites for heavy metal adsorption in contaminated water

M.T Mazibuko[1], Dr. T.H Mokhothu[2*], Prof. P.S Mdluli[3], Dr. Vimla Paul[4]

[1,2,3,4]Department of chemistry, Durban University of Technology, Durban, South Africa

Introduction

- Heavy metal ion contamination in fresh water had an serious Impact of on human health.
- The Agency for Toxic Substances and Disease Registry (ATSDR) of the United State Environmental Protection Agency (US, EPA) has prioritized Pb, Cd, and Cr at the 2nd, 7th and 17th positions in its Substances Priority List (SPL), respectively (ATSDR, 2019).
- The National Toxicology Program (NTP) of the United State department of health and human services categorized three heavy metals (Pb, Cd and Cr) as known human carcinogenic (NTP, 2016)
- The World Health Organization (WHO) has established the allowable limit for Pb, Cd, and Cr in drinking water at 0.01 mg/L, 0.03 mg/L, and 0.05 mg/L, respectively (WHO, 2017).
- Various techniques have been employed to address the removal of heavy metals, amongst those techniques adsorption has been chosen due to it advantages.
- Agricultural residue and unmodified cellulose have used as adsorbent for removal of heavy metals in wastewater However, these nanopapers were too dense to allow for high water permeability, resulting materials inefficient in heavy metal removal.
- This study is focused on fabricating a novel adsorbent derived from renewable resource, cellulose,.

Methodology

Extraction and Characterization nof cellulose from stem banana fibre

- The cellulose, lignin and the hemicellulose contents of the extracted cellulose will be characterized by using a standard method recommended in TAPPI-T222 om-88 content will be obtained as described in TAPPI T19m-54 (Motaung, and Mokhothu, 2016).
- The morphology of extracted cellulose will be characterized by scanning electron microscope; crystal structure by X-Ray Diffraction (XRD), functional groups by Fourier transform infrared spectroscopy (FTIR) and thermal stability using Thermogravimetric analyser (TGA).

Fabrication of cellulose /silica silane functionalized composites.

- *In situ* sol-gel method described by Mokhothu *et al.*, 2015 will be used to fabricate amine functionalized cellulose-silica composites from tetraethylortosilicate (TEOS) as a silica precursor in the presence of amine-based silane coupling agent.
- Silica nanoparticles will be synthesized from the addition of TEOS, H_2O, ethanol and sodium hydroxide catalyst (1:4:4:3.52 mol ratio) respectively, followed by varying concentrations of amine-based silane coupling agent (2, 4, and 6%).

Results and discussion

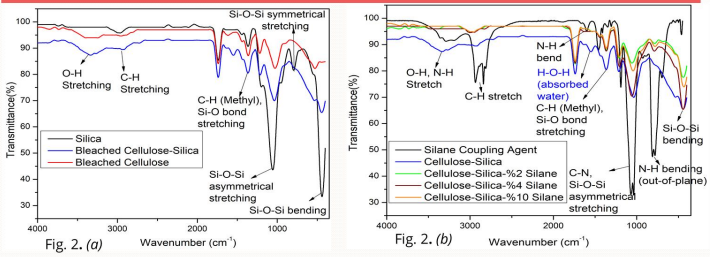

Figure 2. *(a)* FTIR spectra of Cellulose-Silica, *(b)* FTIR spectra of amine functionalized Cellulose-Silica

Amine functionalized Cellulose-Silica

- FT-IR analysis verified the modification of bleached cellulose-silica nanoparticles with an amine coupling agent (Figure 2). The peak at 441 cm^{-1} in the FT-IR spectrum of amine functionalized cellulose-silica (Figure 2*(a)*) are related to Si–O–Si bending (Khan *et al.*, 2016).
- The Si-O-cellulose asymmetric vibration is related to the overlap with the strong band at 1042 cm^{-1}, verifying the covalent bonding of silica and cellulose (Azarshin *et al.*, 2017).
- The FT-IR spectra of unfunctionalized cellulose-silica with amine base silane coupling agent shows peaks at 3338 cm^{-1} and 1550 cm^{-1}, which are related to the O–H stretching band and the residual adsorbed water molecules, respectively (Auta *et al.*, 2017).
- However, after functionalizing cellulose-silica using an amine base silane coupling agent, the FTIR spectrum shows that the O-H and adsorbed water bands are no longer present (Figure 2.(b)).
- The C–H stretching vibration is responsible for the peak at 2959 cm1 (Fig. 2.(b)). (Yousif *et al.*, 2019).
- N-H out-of-plane bending is responsible for a tiny band observed in the 850–750 cm^{-1} region. The N–H bending vibration of primary and secondary amine groups is responsible for the new band (796 cm^{-1}) (Khan *et al.*, 2016). All of these findings suggest that amine functionalized cellulose-silica has been prepared successfully.
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Research Problem and Aims

Aim

- The aim of this is study is to extract cellulose from banana stem to fabricate an absorbent reinforced with silica nanoparticles and functionalized with amine-based silane coupling agent for heavy metal ions adsorption.

Objectives

- To synthesize and characterize cellulose/silica composites functionalized with amine base silane coupling agent by *in-situ* sol-gel process.
- To characterize cellulose/ silica composite using Zeta potential; Scanning Electron Microscopy (SEM); transmission electron microscopy (TEM); X-Ray Diffraction (XRD), Fourier transform infrared spectroscopy (FTIR); Brunauer–Emmett–Teller (BET) and Thermo gravimetric analyser (TGA).
- To determine the metal ion concentration using ICP-OES and Graphite Furnace Atomic Absorption Spectroscopy for trace levels adsorbed by cellulose/silica composites.
- To simulate the interaction between amine functionalized cellulose-silica and metals.

Methodology

Characterization of amine functionalized cellulose-silica silane composites.

- The surface charge of amine functionalized cellulose-silica composites will be characterized by Zeta potential, morphology by TEM and SEM, crystal structure using XRD, identification of functional using FTIR, and surface area analysis will be done using BET.

The adsorption technique for the removal of heavy metals.

- The adsorption study of the selected metal ions which are Lead (Pb), Cadmium (Cd) and Chromium (Cr) will be conducted using UV-Visible spectrophotometer and ICP-EOS.

Molecular modelling studies

- The molecular modelling will be performed using the Materials Studio software.

Results and discussion

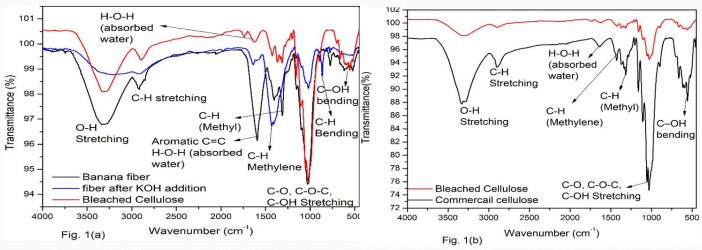

Figure 1. *(a)* FTIR spectra of cellulose extraction step-by-step, *(b)* FTIR spectra of bleached and commercial cellulose

Conclusion

- In this study, Cellulose was extracted successfully from banana stem fibre and FTIR spectra confirmed the removal of hemicelluloses and lignin; these results were compared to FTIR spectrum of commercial Cellulose.
- The synthesis of cellulose/silica composites functionalized with amine base silane coupling agent via *in-situ* sol-gel process was perform successfully
- FT-IR analysis verified the modification of bleached cellulose-silica nanoparticles with an amine coupling agent.
- N-H out-of-plane bending is responsible for a tiny band observed in the 850–750 cm^{-1} region. The N–H bending vibration of primary and secondary amine groups is responsible for the new band (796 cm^{-1}).
- All of these findings suggest that amine functionalized cellulose-silica has been prepared successfully.

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