Data Article

Descriptive data on trichloroethylene and Congo red dye adsorption from wastewater using bio nanosponge phosphorylated-carbon nanotube/nanoparticles polyurethane composite

Anny Leudjo Taka [a,1], Elvis Fosso-Kankeu [b], Kriveshni Pillay [a], Xavier Yangkou Mbianda [a,*]

[a] *Department of Chemical Sciences, University of Johannesburg, Doornfontein, Johannesburg 2028, South Africa*
[b] *School of Chemical and Minerals Engineering, Faculty of Engineering, North West University, Potchefstroom Campus, Potchefstroom, South Africa*

A B S T R A C T

The availability and quality of water resources is currently the primary concern in Southern Africa. The challenge is to improve or develop water treatment materials or methods to solve this problem of potable water scarcity. Hence, this article presents the analyzed data, which are supplementary data information on the study of bio nanosponge phosphorylated-carbon nanotube/nanoparticles polyurethane composite (pMWCNT/β-CD/TiO$_2$-Ag) as polymeric nanobiosorbent, for water treatment. The developed polymeric nanobiosorbent (pMWCNT/β-CD/TiO$_2$-Ag) was synthesized using combined methods of amidation reaction, cross polymerization, and sol-gel process. The removal of water pollutants (trichloroethylene (TCE) and Congo red (CR) dye) was conducted by the batch adsorption method. The conditions used during the adsorption experiments and methods (applied to quantify the water samples after

adsorption studies) are described. Additional data obtained on the effect of pH, isotherm, kinetic, and thermodynamic studies are also illustrated.

Specifications Table

Subject area	Chemistry, Chemical Engineering and Physics
Specific subject area	Wastewater treatment and adsorption
Type of data	Figures and images
How data was acquired	The thermal stability of pMWCNT/β-CD/TiO$_2$-Ag nanobiosorbent was evaluated using differential scanning calorimetry (DSC) Mettler Toledo DSC 822 and thermal gravimetric analysis (TGA) Pyris Series-TGA 4000 analyzer.
	The surface charge (zeta potential) on the adsorbent was determined using Malvern Zeta-Sizer Nanoseries.
	After adsorption experiment the TCE filtrates were analyzed by Leco Pegasus 4D GCxGC TOF MS while UV–Vis spectrophotometer (UV–Vis spectrometer Shimadzu UV-2450) was used to quantify the remaining amount of CR dye in water samples.
	The determination of total organic carbon (TOC) concentration in the real water sample was done using TELEDYNE TEKMAR TOC FUSION.
Data format	Raw data
Parameters for data collection	GC Methods: Column length 29.577 m, internal diameter 250 µm, film thickness of 0.25 µm with a maximum temperature of 260 °C. Carrier gas=Helium; front inlet type = split/splitless. Flow rate=1.00 mL/min, front inlet temperature = 250 °C. Initial oven temperature of 50 °C and ramped at 30 °C/min to 250 °C/min, the transfer line temperature was 300 °C.
	UV–Vis spectrophotometer was set on the photometric mode with 495 nm as the maximum absorption wavelength for CR dye.
Description of data collection	The data were collected as .asc and .txt files, then analysed using Microsoft Excel and Origin 8 Lab software programs to obtain the presented figures. The images were captured using a camera.
Data source location	University of Johannesburg, Gauteng, South Africa
Data accessibility	Data are with this article
Related research article	A. Leudjo Taka, E. Fosso-Kankeu, K. Pillay, X. Yangkou Mbianda, Metal nanoparticles decorated phosphorylated carbon nanotube/cyclodextrin nanosponge for trichloroethylene and Congo red dye adsorption from wastewater, J. Environ. Chem. Eng. Volume 8, Issue 3, June 2020, 103,602. doi:10.1016/j.jece.2019.103602.

Value of the Data

- The data obtained are essential because the developed polymeric nanobiosorbent (pMWCNT/β-CD/TiO$_2$-Ag) has a good capacity to be used as a filter or adsorbent material for wastewater purification.
- The data acquired can help other researchers have detailed information on the mechanism of adsorption of TCE and CR dye.
- These data might be useful for further experiments regarding column adsorption design for the removal of TCE from wastewater samples using the developed pMWCNT/β-CD/TiO$_2$-Ag as adsorbent material.

1. Data Description

DSC (Fig. A.1) and TGA (Fig. A.2) analyses were used to evaluate the thermal stability of pMWCNT/β-CD/TiO$_2$-Ag nanobiosorbent [1]. From Fig A.1, it can also be depicted that the
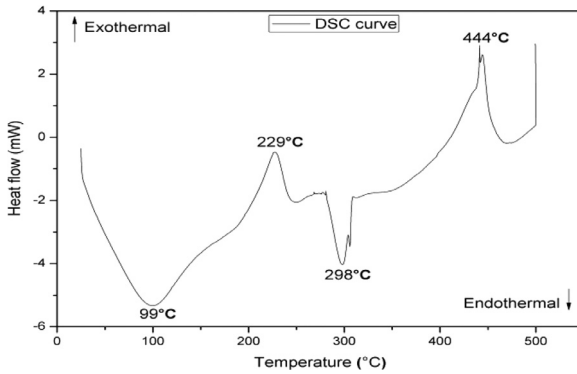
Fig. A.1. DSC curve of pMWCNT-βCD/TiO$_2$-Ag nanobiosorbent [1].

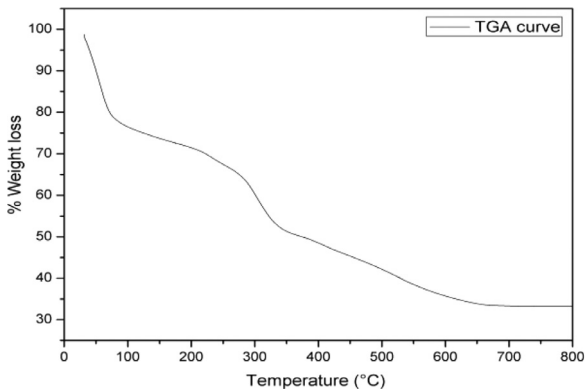**Fig. A.2.** TGA curve of pMWCNT-βCD/TiO$_2$-Ag nanobiosorbent [1].

synthesized pMWCNT/β-CD/TiO$_2$-Ag has a DSC curve comparable to that of a semicrystalline polymer and polymorph material.

1.1. Solution pH and adsorbent dosage studies

The surface charge on the nanobiosorbent (pMWCNT/β-CD/TiO$_2$-Ag) was determined by zeta potential analysis. The surface charge of the adsorbent material plays a huge role during the adsorption and desorption processes. From Fig. B.1, it can be clearly observed that the developed polymeric nanobiosorbent is charged positively at pH < pH$_{pzc}$ (pH point of zero charge) whereas the nanobiosorbent is negatively charged at pH > pH$_{pzc}$. At the point of zero charge (pH = 4.058), the developed adsorbent is neutral; it has no charge (pH = pH$_{pzc}$).

1.2. Isotherm models

The color changes of the model pollutant solution at different concentrations were observed after adsorption of CR dye unto the polymeric nanobiosorbent (Fig. C.1(B) and (D)). The color
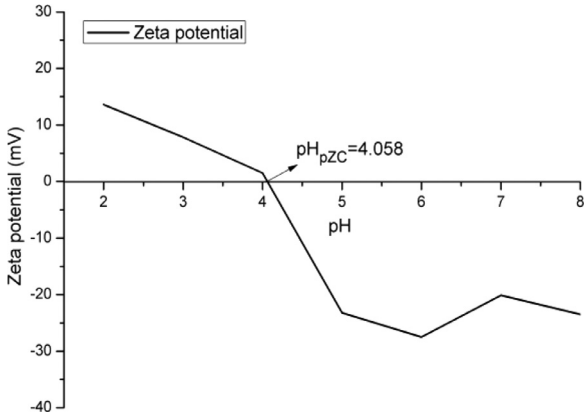
Fig. B.1. Zeta potential analysis of pMWCNT/β-CD/TiO$_2$-Ag nanobiosorbent [2].
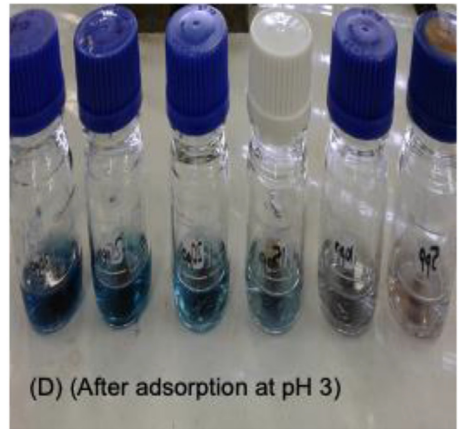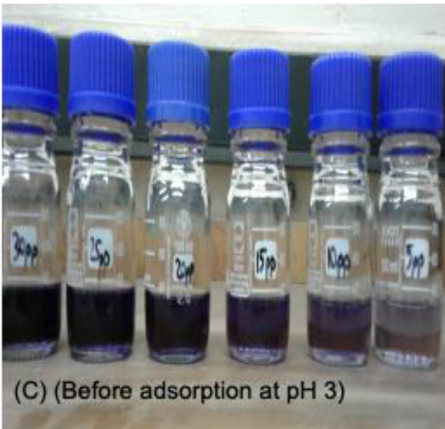

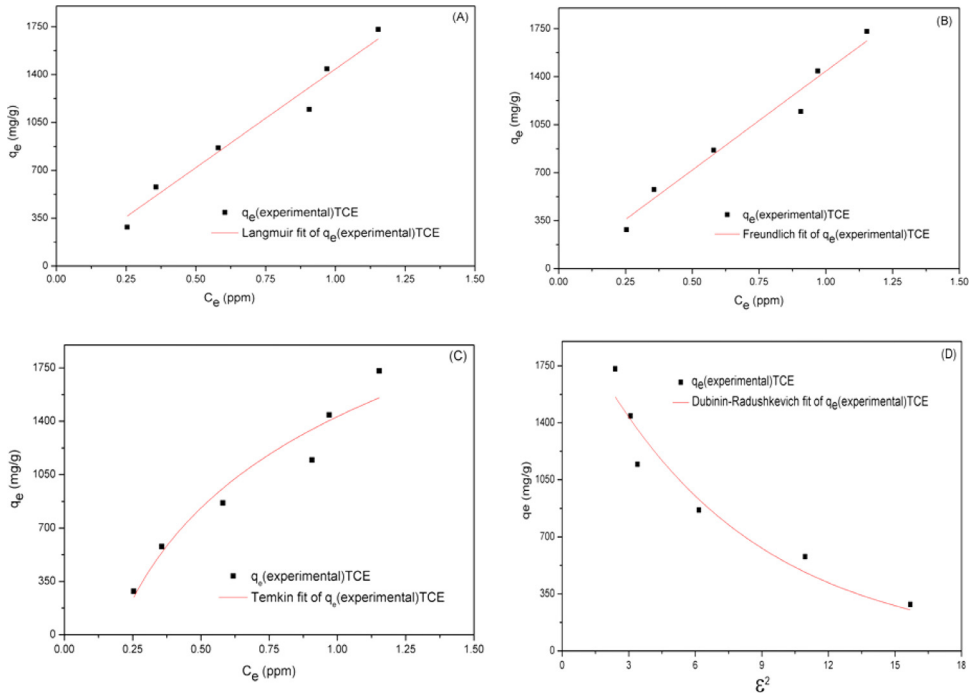**Fig. C.1.** Color changes observed on model pollutant solutions of CR dye before and after adsorption unto pMWCNT/β-CD/TiO$_2$-Ag.

Fig. C.2. Isotherm model plots for the adsorption of TCE unto pMWCNT-βCD/TiO$_2$-Ag.

change of the polymeric nanobiosorbent (from gray to red) was also noticed after adsorption of CR dye.

Figs. C.2(A) and C.3(A) present Langmuir model plots for the sorption of TCE and CR dye respectively onto the developed polymeric nanobiosorbent. According to the Langmuir model, the adsorptions of TCE and CR dye are favorable at low and high concentrations because the R_L factor values calculated were less than 1 [3]. The maximum sorption capacity (q_{max}) of the developed nanobiosorbent for TCE adsorption was higher than the q_{max} for CR dye. Therefore, the developed polymeric nanobiosorbent (pMWCNT/β-CD/TiO$_2$-Ag) has demonstrated a higher adsorption capacity for TCE than for CR dye.

Figs. C.2(B) and C.3(B) represent the Freundlich isotherm plot for the sorption of TCE and CR dye, respectively. The calculated "n" values were found to be less than 1, which is an indication that the adsorption was favourable [3] for both TCE and CR dye. The R^2 of the Freundlich isotherm was higher for the sorption of TCE than for CR dye sorption.

The Temkin plots for TCE and CR dye adsorption, respectively, are presented in Figs. C.2(C) and C.3(C). The constant related to the heat of sorption (b_T) was higher than −40 kJ/mol for the adsorption of both TCE and CR dye. This means that the chemisorption process [4] was involved in the mechanism of sorption of both TCE and CR dye onto pMWCNT/β-CD/TiO$_2$-Ag according to Temkin model.

Figs. C.2(D) and C.3(D) illustrate the Dubinin-Radushkevish (D-R) model plot for the adsorption of TCE and CR dye, respectively. The respective mean free energy (also called activation energy, E) for the sorption of TCE and CR dye was calculated (Table 1 of the main article); the E values obtained were less than 8 kJ/mol, meaning the adsorption of TCE and CR dye onto pMWCNT/β-CD/TiO$_2$-Ag was taking place through a physisorption process according to (D-R) model [4].
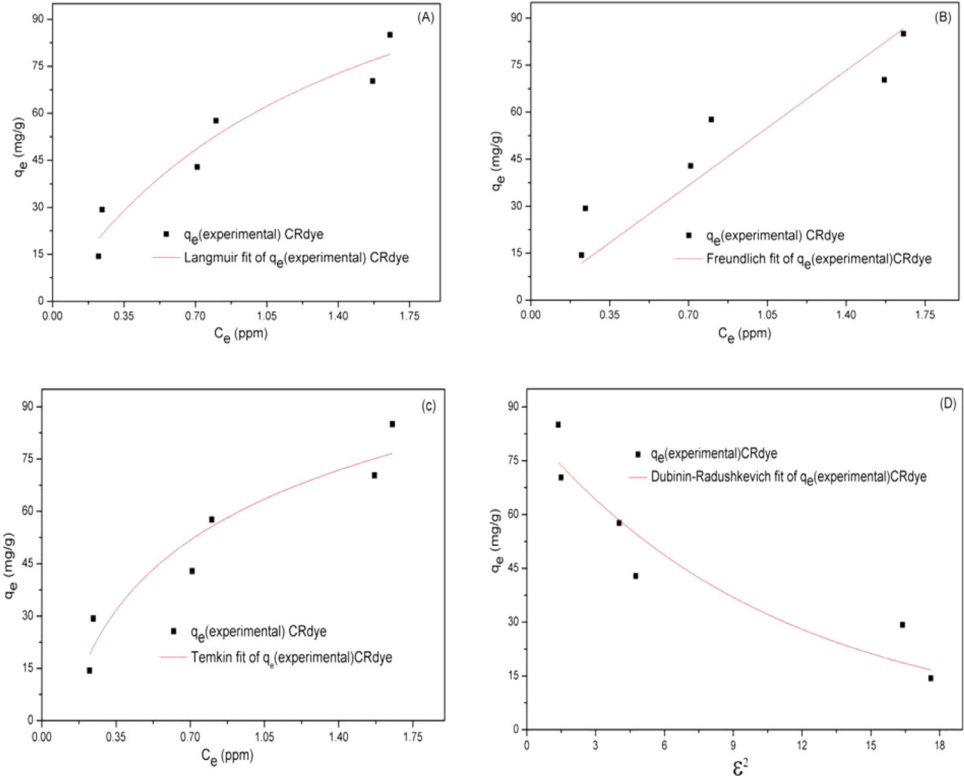
Fig. C.3. Isotherm model plots for the adsorption of CR dye unto pMWCNT-βCD/TiO$_2$-Ag.

1.3. Evaluation of temperature's effect and thermodynamic study

The parameters of thermodynamic were calculated using the Van't Hoff Eq. (1) [3,5]:

$$\Delta G^\circ = -RT \, lnk_c \tag{1}$$

According to the thermodynamic law, the Van't Hoff equation (Eq. (1)) can also be illustrated as [6–8]:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{2}$$

Eq. (2) above can be further transformed as follows [3]:

$$lnk_c = -\Delta G^\circ/RT = -\Delta H^\circ/RT + \Delta S^\circ/R \tag{3}$$

where k_c = equilibrium constant; T = absolute temperature (Kelvin (K)); and R = universal gas constant.

The equilibrium constant (k_c) is dimensionless and has no unit. It is essential to note that its values can be derived in different ways: from the Langmuir or Freundlich constant obtained at different temperatures; from the partition coefficient (k_p) or the distribution coefficient (k_d) [3]. The equilibrium constant (k_c) values were derived from the distribution coefficient (k_d). Firstly, k_d values were determined at different temperatures (288 K, 298 K, and 310 K) from $ln(q_e \, / \, C_e)$ vs C_e graph and extrapolating to zero C_e. Then the intercept value when $C_e = 0$, gives the value of K_d. Since k_d has a unit (L/g), its values should be multiplied by a factor of 1000 to finally obtain the dimensionless k_c values used to design the Van't Hoff plots [3].
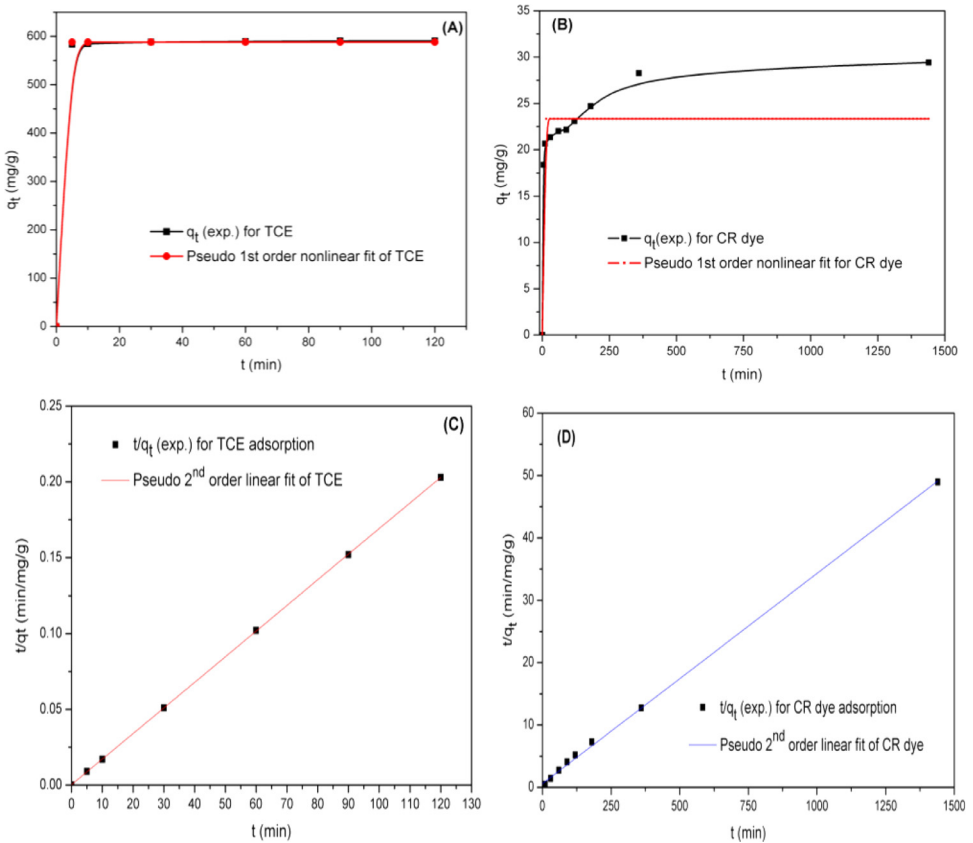
Fig. D.1. The plots of Kinetic models for the adsorption of TCE and CR dye on pMWCNT-βCD/TiO$_2$-Ag.

1.4. Kinetic studies

Effect of contact time: Fig. D.1(A) and (B) illustrates the effect of contact time on the adsorption of TCE and CR dye, respectively. It is seen that the sorption of TCE unto pMWCNT/β-CD/TiO$_2$-Ag occurs very fast, while the uptakes of CR dye is very slow.

The nonlinear method, using the Origin program (for nonlinear curve fit) [3], was used to evaluate the pseudo-first-order (PFO) model parameters as well as the values of R^2 and SSE. Fig. D.1(A) and (B) shows a nonlinear relationship representing the PFO model for sorption of TCE and CR dye, respectively.

Fig. D.1(C) and (D) presents a linear relationship representing, respectively the pseudo-second-order (PSO) model for TCE and CR dye sorption unto pMWCNT/β-CD/TiO$_2$-Ag. The values of q$_e$ (cal.) and K$_2$ were calculated from the slope and intercept of the linear trendline t/q$_t$ vs t.

Besides, since the PSO is the suitable kinetic model, therefore the chemisorption process might be considered as the rate-limiting step [9,10]. Similarly, it was also shown in previous research studies (reporting on the use of organic-inorganic composite adsorbent) that the PSO was the good fit kinetic model and the intra-particle diffusion was not the only rate-limiting step due to multi-linearity and the trendline not passing through origin [11–14].

The supplementary files have also been provided, and these supplementary files represent the raw data for the analyses conducted: TGA, DSC, zeta-potential, TOC, UV–Vis analysis for Congo red dye, GCMS analysis for TCE with method development.
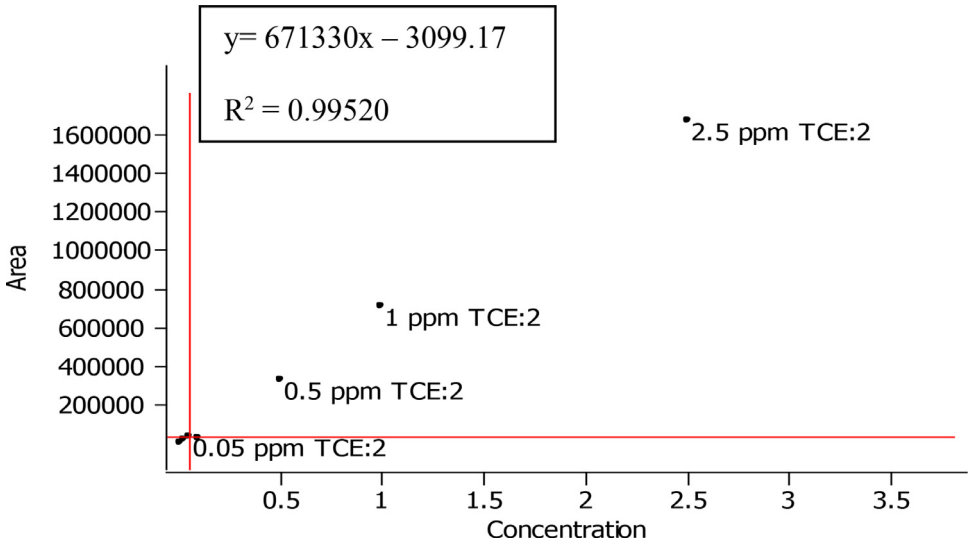
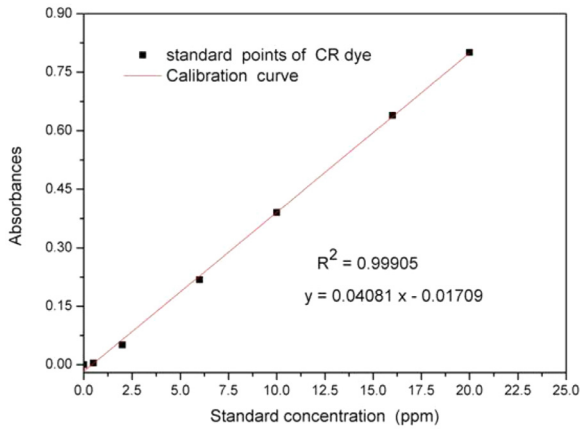**Fig. E.1.** Standard calibration curve for TCE.

Fig. F.1. Standard calibration curve for CR dye.

Fig. E.1 presents the Standard curve employed to calculate the unknown concentration of water samples.

Fig. F.1 presents the standard calibration curve used to calculate the unknown concentration of water samples.

1.4.1. Determination of total organic carbon (TOC) in the real wastewater samples

Fig. G.1 shows the standard calibration curve for the determination of unknown concentrations of total organic (TOC) before and after adsorption using TELEDYNE TEKMAR TOC FUSION.
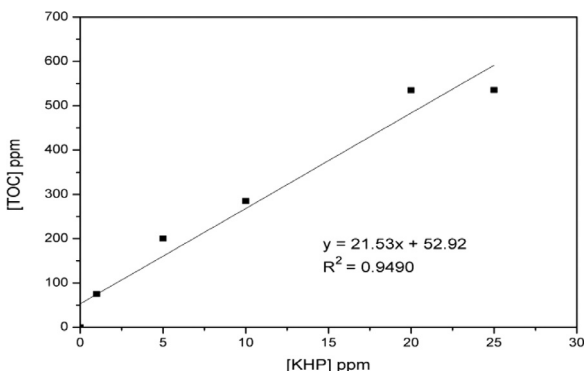
Fig. G.1. Standard calibration Curve for the determination of unknown concentration of TOC in wastewater samples.

2. Experimental Design, Materials and Methods

2.1. Materials

MWCNTS ($>$90%), oxalyl chloride, β-cyclodextrin (β-CD) (97%), hexamethylene diisocyanate (98.0%), TiO$_2$ precursor (Titanium tetraisopropoxide, 97%), TCE (99.5%), acetonitrile ($>$99.5%), and sodium sulfate (Na$_2$SO$_4$, anhydrous salt) ($>$99.0%) were obtained from Sigma Aldrich. CR dye (98%) was obtained from Associated Chemical Entreprises while and the precursor for Ag (silver nitrate, 99.8%) was from Rochelle Chemicals South Africa.

2.2. Preparation and characterization of the polymeric nanobiosorbent (pMWCNT/β-CD/TiO$_2$-Ag)

In short, pristine MWCNTs were first purified then oxidized and chlorinated using oxalyl chloride followed by the amidation reaction in order to obtain phosphorylated carbon nanotubes (pMWCNTs). pMWCNTs obtained were then co-polymerized with β-CD using hexamethylene diisocyanate as a linker agent. Then using the sol-gel method, the resulting polymer (pMWCNT/β-CD) was decorated with TiO$_2$ and Ag nanoparticles to yield the bio nanosponge polyurethane composite, pMWCNT/β-CD/TiO$_2$-Ag [1]. The new material developed (pMWCNT/β-CD/TiO$_2$-Ag) was characterized using differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA), electron microscopy, and combined spectroscopy techniques [1].

2.3. Batch adsorption experiments and conditions of the adsorption parameters investigated

The adsorption experiments were conducted under specific conditions as described below. For these experiments, 100 mL glass bottles were used. These glass bottles, containing a specific amount of the prepared nanobiosorbent and 30 mL model pollutant solution, were agitated by a thermostatic platform shaker at 200 rpm.

(a) Solution pH [conditions: C_o = 10 ppm, adsorbent dosage = 0.0025 mg, temperature = 298 K, time = 24 h, V = 30 mL and speed = 200 rpm]
(b) Adsorbent dosage [conditions: C_o = 10 ppm, temperature = 298 K, time = 24 h, optimum pH (TCE) = 2, pH (CR dye) = 3, V = 30 mL and speed = 200 rpm]
(c) Contact time [conditions: C_o = 10 ppm, temperature = 298 K, pH (TCE) = 2, optimum pH (CR dye) = 3; optimum adsorbent dosage = 0.5 g (for TCE) and 0.01 g (for CR dye), V = 30 mL and speed = 200 rpm]

(d) Initial concentrations and temperatures [conditions: C_o range = [5–30 ppm], optimum pH (TCE) = 2, pH (CR dye) = 3, optimum adsorbent dosage = 0.5 mg (for TCE) and 0.01 g (for CR dye), V = 30 mL, time = 24 h, speed = 200 rpm, temperatures = 288 K, 298 K & 310 K].

2.4. Preparation of stock, model pollutants and standard solutions for TCE

The main stock solution (1000 ppm) was prepared in acetonitrile, and then a standard solution of 100 ppm was prepared by dilution method of a volume of 1000 ppm stock solution using acetonitrile. The model pollutants (ranging from 5 to 30 ppm) and standard solutions for the calibration curve (ranging from 0 to 5 ppm) were prepared by dilution method of a volume of 100 ppm solution using ultrapure deionized water (milli-Q-water). After adsorption experiments, the TCE filtrates were analyzed by Leco Pegasus 4D GCxGC TOF MS using the static headspace method [15].

2.5. GCxGC-TOF MS Leco method development

The analysis of TCE filtrate water samples was performed on a Leco GCxGC-TOF Low-resolution mass spectrophotometer.

a) Sample Preparation: 3.5 g of sodium sulfate (Na_2SO_4, anhydrous salt) was added in 5 mL of each water sample, and then well mixed. (The addition of Na_2SO_4 anhydrous salt helps to saturate the sample solution in order to enhance the sensitivity of the static headspace method to obtain peaks with high intensity). The sample mixtures were then used for GC–MS analysis.

b) Sample Identification: Headspace GCxGC / GC Tof MS

c) Pre-injection Methods: Using the agitator the sample was incubated at 70 °C for 10 min. The agitator speed was 500 rpm, with agitator speed of 100 rpm when extracting the sample.

d) Injection Methods: 1 mL sample was injected in the injection port using a 2.5 mL-HS syringe with an injection temperature of 120 °C. The injection fill-up and injection speeds were 200 μL/sec and 100 μL/sec, respectively. The injection port had a Topaz liner, split single taper gooseneck w/Wool = 4 mm x 6.5 × 78.5) using a Gerstel Multi-Purpose Autosampler.

e) GC Methods: Column type (Stabilwax), length 29.577 m of (30 m), the internal diameter 250 μm, and film thickness of 0.25 μm with a maximum temperature of 260 °C was used. Carrier gas=Helium; front inlet type = Split/splitless with a splitless mode activated. Flow rate=1.00 mL/min at constant flow while front inlet temperature = 250 °C. Initial oven temperature of 50 °C which was maintained for 1 min and it ramped at 8 °C /min to 180 °C/min. It was further ramped at 30 °C/min to 250 °C/min, where it was held for 0.17 min. The transfer line temperature was 300 °C.

f) MS Methods: acquisition delay = 0 min, a mass range of 45 to 450 *m/z* was analyzed with the acquisition rate of 50 spectra/second. The acquisition voltage of 1452 eV was used and electron energy (ionization energy) = −70 eV; ion source temperature = 200 °C.

g) Data Processing Methods: The baseline offset of 1 (just above the noise) was used, with a peak width of 4 secs; the signal to noise (S/N) ratio =10. The library search mode was normal and forward. The number of libraries hit = 10, minimum molecular weight allowed = 45 and maximum molecular weight = 450. Mass threshold = 2% and minimum similarity before the name were assigned = 50%. The standard reference database was NIST and contents two electron ionization (EI) libraries, replib and mainlib (which were used).

2.6. Preparation of stock and model pollutants and standard solutions for CR dye

The Main stock solution (1000 ppm, $v = 1$ L) was prepared using 1 g of CR dye powder made up with ultrapure deionized water (milli-Q-water). The model pollutants (ranging from 5 to 30 ppm) and standard solutions (ranging from 0 to 20 ppm) were made up by diluting a specific volume of 1000 ppm solution using ultrapure deionized water (milli-Q-water). After adsorption experiments, the CR dye filtrates were analyzed by UV–Vis spectrophotometer (UV–Vis spectrometer Shimadzu UV-2450) using the photometric mode (the maximum absorption wavelength used for CR dye was 495 nm).

2.7. Regeneration and desorption studies

The regeneration of the developed pMWCNT/β-CD/TiO$_2$-Ag nanobiosorbent was predicted by conducting three cycles of adsorption-desorption. Desorption study for TCE was performed at pH = 4 while for CR dye it was done at pH 11 (pH > pH$_{pzc}$) using diluted ethanol solution and deionised water as desorbing solvents under the following conditions: $C_o = 10$ ppm, temperature = 298 K, optimum pH (TCE) = 2, pH (CR dye) = 3; optimum adsorbent dosage = 0.5 mg (for TCE) and 0.01 g (for CR dye), $V = 30$ mL, speed = 200 rpm and contact time = 24 h.

All the raw data related to this article are available in Mendeley data repository [16].

Ethics Statement

The work reported in this article has no ethical issue.

CRediT Author Statement

Anny Leudjo Taka: Methodology, Investigation, data analysis and Writing-Original draft preparation; **Elvis Fosso-Kankeu:** Supervision, Reviewing and Editing; **Kriveshni Pillay:** Supervision, Reviewing and Editing; **Xavier Yangkou Mbianda:** Conceptualization, Supervision, Reviewing and Editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have or could be perceived to have influenced the work reported in this article.

Acknowledgments

References

[1] A. Leudjo Taka, K. Pillay, X.Y. Mbianda, Synthesis and characterization of a novel bio nanosponge filter (pMWCNT-CD/TiO2-Ag) as potential adsorbent for water purification, in: P. Ramasami, M.G. Bhowon, S.J. Laulloo, H. Li, K. Wah

(Eds.), Emerging Trends in Chemical Sciences, © Springer International Publishing AG, 2018, pp. 313–343, doi:10.1007/978-3-319-60408-4_18.

[2] A. Leudjo Taka, E. Fosso-kankeu, K. Pillay, X.Y. Mbianda, Removal of cobalt and lead ions from wastewater samples using an insoluble nanosponge biopolymer composite: adsorption isotherm, kinetic, thermodynamic, and regeneration studies, Environ. Sci. Pollut. Res. 25 (2018) 21752–21767, doi:10.1007/s11356-018-2055-6.

[3] N.H. Tran, S. You, A. Hosseini-bandegharaei, H.-P. Chao, Mistakes and inconsistencies regarding adsorption of contaminants from aqueous solutions: a critical review, Water Res. 120 (2017) 88–116, doi:10.1016/j.watres.2017.04.014.

[4] P. Thilagavathy, T. Santhi, Studies on the removal of Cu(II) from aqueous solutions using modified Acacia nilotica leaf, Bioresources 9 (2014) 3805–3824.

[5] R. Prabakaran, S. Arivoli, Removal of cobalt (II) from aqueous solutions by adsorption on low cost activated carbon, Int. J. Sci. Eng. Technol. Res. 2 (2013) 271–283.

[6] P. Thilagavathy, T. Santhi, Adsorption and desorption performance of acacia nilotica for the removal of cobalt (II) by column and batch mode study from aqueous phase and its suitability in real waste water, Int. J. Sci. Res. 3 (2014) 393–399.

[7] E. Fosso-kankeu, H. Mittal, F. Waanders, S. Sinha, Thermodynamic properties and adsorption behaviour of hydrogel nanocomposites for cadmium removal from mine effluents, J. Ind. Eng. Chem. 48 (2017) 151–161, doi:10.1016/j.jiec.2016.12.033.

[8] K. Parashar, N. Ballav, S. Debnath, K. Pillay, A. Maity, Rapid and efficient removal of fluoride ions from aqueous solution using a polypyrrole coated hydrous tin oxide nanocomposite, J. Colloid Interface Sci. 476 (2016) 103–118, doi:10.1016/j.jcis.2016.05.013.

[9] Y.S. Ho, G. McKay, A comparison of chemisorption kinetic models applied to pollutant removal on various sorbents, Process Saf. Environ. Prot. 76 (1998) 332–340, doi:10.1205/095758298529696.

[10] H. Qiu, L. LU, B. Pan, Q. Zhang, W. Zhang, Q. Zhang, Critical review in adsorption kinetic models, J. Zhejiang Univ. Sci. A 10 (2009) 716–724, doi:10.1631/jzus.A0820524.

[11] M. Bhaumik, R. Mccrindle, A. Maity, Efficient removal of Congo red from aqueous solutions by adsorption onto interconnected polypyrrole – polyaniline nanofibres, Chem. Eng. J. 228 (2013) 506–515, doi:10.1016/j.cej.2013.05.026.

[12] J. Liang, J. Liu, X. Yuan, H. Dong, G. Zeng, H. Wu, H. Wang, J. Liu, S. Hua, S. Zhang, Z. Yu, X. He, Y. He, Facile synthesis of alumina-decorated multi-walled carbon nanotubes for simultaneous adsorption of cadmium ion and trichloroethylene, Chem. Eng. J. 273 (2015) 101–110, doi:10.1016/j.cej.2015.03.069.

[13] S. Ghorai, A. Kumar, A.B. Panda, S. Pal, Effective removal of Congo red dye from aqueous solution using modified xanthan gum/silica hybrid nanocomposite as adsorbent, Bioresour. Technol. 144 (2013) 485–491, doi:10.1016/j.biortech.2013.06.108.

[14] S. Pal, A. Shankar, S. Ghorai, A. Kumar, V. Mahato, Efficient and rapid adsorption characteristics of templating modified guar gum and silica nanocomposite toward removal of toxic reactive blue and Congo red dyes, Bioresour. Technol. 191 (2015) 291–299, doi:10.1016/j.biortech.2015.04.099.

[15] K. Jacq, F. David, P. Sandra, M.S. Klee, Analysis of Volatile Organic Compounds in Water Using Static Headspace-GC/MS. Application Note Environmental, 2008. www.agilent.com/chem%0AAgilent.

[16] Y.M. Xavier, L.T. Anny, Descriptive data on trichloroethylene and Congo red dye adsorption from wastewater using bio nanosponge phosphorylated-carbon nanotube/nanoparticles polyurethane composite, Mendeley Data V1 (2021), doi:10.17632/d2pr838xv5.1.