ADSORPTION STUDIES OF FLUOROTELOMER OLEFIN ONTO GRANULAR ACTIVATED CARBON, NON-ION AND ION EXCHANGE RESINS

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Thesis submitted to the Centre for Graduate Studies, Universiti Pertahanan Nasional Malaysia, in fulfilment of the requirements for the Degree of Master of Science (Chemistry)

ABSTRACT

Introduction of fluorotelomer into the industry as a replacement for per- and polyfluoroalkyl substances (PFAS) has raised global concern due to its widespread dispersion and potential persistency, bioaccumulation, and toxicity. Adsorption has been recognized as an effective method for removing PFAS from water, however there are lack of studies focusing on fluorotelomer. This study aims to enhance the understanding of fluorotelomer olefin (FTO) adsorption onto different adsorbents, providing valuable insights into potential strategies to eliminate FTO from water systems. Various adsorbents including granular activated carbon (GAC), non-ion exchange resin (XAD-4), and ion exchange resin (IRA958) were examined for adsorption capacity, kinetics, and isotherms under different conditions: adsorbent dosages, contact times, and FTO initial concentrations. Scanning electron microscopy (SEM) images demonstrate that the roughness surface of GAC and smooth surface of XAD-4 remained the same after the adsorption. However, foreign substances were observed on the smooth and cracked surface of IRA958. Brunauer-Emmet-Teller (BET) measurement displayed large surface area of XAD-4 (860.8548 m²/g) followed by GAC (544.1870 m²/g) and IRA958 (2.0381 m²/g). FTO was detected and quantified using gas chromatography-mass spectrometry (GC-MS). The adsorption of FTO on all adsorbents reached equilibrium within 5 h and the data were best-fitted to pseudosecond-order kinetics model ($R^2 > 0.99$). Meanwhile, Freundlich isotherm model was found to be suitable for describing FTO adsorption, with GAC exhibiting the highest adsorption capacity ($K_f = 3.0853$), followed by IRA958 ($K_f = 3.0400$) and XAD-4 ($K_f = 0.0009$). The findings suggest that the adsorption mechanism of FTO onto adsorbents is characterized by multilayer and chemosorption behavior. Desorption study reveals that FTO was not able to desorb from the adsorbent's surface using either methanol, ethanol, or n-hexane. Removal of FTO remain high (>80%) with or without the presence of interfering compounds (FTI, FTOH and PFHxI), under optimum adsorption conditions: 1.25 mg adsorbent dosage and 5 h contact time with initial concentration of FTO 100 mg/L and above. The findings from this study have shown the potential of GAC, XAD-4 and IRA958 to be used as adsorbents in water treatment plants to remove FTO from water, hence improving the overall quality of water and environment.

ABSTRAK

Pengenalan fluorotelomer ke dalam industri sebagai pengganti sebatian perfluoroalkil (PFAS) telah menimbulkan kebimbangan global disebabkan penyebaran meluas dan potensi keberkekalan, bioakkumulasi, dan toksisiti. Penjerapan telah dikenal pasti sebagai kaedah yang berkesan untuk menyingkirkan PFAS dari air, namun terdapat kekurangan kajian yang memberi tumpuan terhadap sebatian fluorotelomer. Kajian ini bertujuan untuk meningkatkan pemahaman mengenai penjerapan fluorotelomer olefin (FTO) ke atas pelbagai penjerap yang berbeza, di mana kajian ini dapat membantu meberikan pemahaman yang berguna terhadap strategi yang bersesuaian dalam menyingkir FTO daripada sistem pengairan. Pelbagai penjerap termasuk butir karbon teraktif (GAC), resin pertukaran bukan ion (XAD-4), dan resin pertukaran ion (IRA958) telah dikaji bagi mengetahui kapasiti penjerapan, kinetik dan isoterma di bawah kondisi yang berbeza: dos penjerap, masa sentuhan, dan kepekatan awal FTO. Imej mikroskopi imbas elektron (SEM) menunjukkan bahawa permukaan kasar GAC dan permukaan licin XAD-4 kekal sama selepas penjerapan. Walau bagaimanapun, bahan asing telah diperhatikan muncul di permukaan licin dan retak IRA958. Pengukuran Brunauer-Emmet-Teller (BET) menunjukkan luas permukaan terbesar XAD-4 (860.8548 m2/g) diikuti oleh GAC (544.1870 m2/g) dan IRA958 (2.0381 m2/g). FTO telah dikesan dan dianalisis menggunakan spektrometri jisim gas kromatografi (GC-MS). Dari segi kinetik penjerapan, kebanyakan penjerapanan FTO pada penjerap mencapai keseimbangan dalam masa 5 jam di mana semua penjerap dideskripsikan dengan baik menggunakan model kinetik pseudo-kedua ($R^2 > 0.99$). Sementara itu, model isoterma Freundlich didapati sesuai untuk menerangkan penjerapan FTO, di mana GAC menunjukkan kapasiti penjerapan tertinggi ($K_f = 3.0853$), diikuti oleh IRA958 ($K_f = 3.0400$) dan XAD-4 ($K_f = 0.0009$). Kajian ini mencadangkan bahawa interaksi kuat antara molekul FTO dan permukaan penjerap menghasilkan pembentukan lapisan FTO yang berganda, melibatkan mekanisme penjerapan berlapis dan kimi-serapan. Hasil kajian penyahjerapan mendapati FTO tidak dapat disingkirkan dari permukaan penjerap menggunakan sama ada metanol, etanol atau n-heksana. Selain itu, penyingkiran FTO masih kekal tinggi (>80%) walaupun dengan kehadiran atau tanpa kehadiran sebatian mengganggu yang lain (FTI, FTOH dan PFHxI) di bawah kondisi penjerapan optimum: dos penjerap 1.25 mg dan masa sentuhan 5 jam pada kepekatan FTO 100 mg/L and ke atas. Penemuan dari kajian ini telah menunjukkan potensi GAC, XAD-4 dan IRA958 untuk digunakan sebagai penjerap dalam loji rawatan air untuk menyingkirkan FTO dari sistem perairan, dengan itu mampu meningkatkan kualiti air dan alam sekitar.

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APPROVAL

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LIST OF ABBREVIATIONS

AC	-	activated carbon
ACC	-	American Chemistry Council
ACFs	-	activated carbon felts
ACGIH	-	American Conference of Governmental Industrial Hygienists
AC-H ₃ PO ₄	-	modified activated carbons
AFFF	-	aqueous film-forming foam
AOP	-	advanced oxidation processes
BET	-	Brunauer-Emmet-Teller
CDP	-	cyclodextrin polymers
DFT	-	density functional theory
diPAP	-	disubstituted polyfluoroalkyl phosphates
DOM	-	dissolved organic matter
DWTP	-	drinking water treatment plant
ECHA	-	European Chemicals Agency
EfOM	-	effluent organic matter
EI	-	electron ionization
EI+	-	electron-impact positive ionization mode
EPA	-	Environmental Protection Agency
F-53B	-	polyfluoroalkyl ether sulfonic acid
FASAs	-	perfluoroalkane sulfonamides
Fe/H-GAC	-	FeCl ₃ modified GAC
FeRB	-	iron-reducing bacteria
FO	-	forward osmosis
FOSA	-	perfluorooctane sulfonamide
FTAB	-	fluorotelomer sulfonamidopropyl betaine
FTAC	-	fluorotelomer acrylate

FTAL	-	fluorotelomer aldehyde
FTCA	-	fluorotelomer carboxylic acid
FTI	-	fluorotelomer iodide
FTMAC	-	fluorotelomer methacrylate
FTO	-	fluorotelomer olefin
FTOH	-	fluorotelomer alcohol
FTP	-	fluorotelomer-based polymer
FTS	-	fluorotelomer sulfonate
FTUAL	-	fluorotelomer unsaturated aldehyde
FTUCA	-	fluorotelomer unsaturated carboxylic acid
GAC	-	granular activated carbon
GC-APPI- HRMS	-	gas chromatography-atmospheric pressure photoionization- high resolution mass spectrometry
GC-MS	-	gas chromatography-mass spectrometry
IPD	-	intra-particle diffusion
ITRC	-	Interstate Technology Regulatory Council
LC-MS	-	liquid chromatography-mass spectrometry
LC-MS/MS	-	liquid chromatography/tandem mass spectrometry
LOD	-	limit of detection
LOQ	-	limit of quantitation
MCN	-	multi-walled carbon nanotube
MD	-	molecular dynamics
МО	-	Moringa Oleifera
monoPAP	-	monosubstituted polyfluoroalkyl phosphate
NaOH/NaCl	-	sodium hydroxide/sodium chloride
NF	-	nanofiltration
NIST	-	National Institute of Standard and Technology
NOM	-	natural organic matter
nZVI	-	nanoscale zero-valent iron

OECD	-	Organisation for Economic Co-operation and Development
ОМ	-	organic material
PACFs	-	polyacrylonitrile fiber-derived activated carbon fibers
PAP	-	polyfluoroalkyl phosphoric acid ester
PBSF	-	perfluorobutane sulfonyl fluoride
PEPE	-	polymeric perfluoropolyether
PFAAs	-	perfluoroalkyl acids
PFAI	-	perfluoroalkyl iodide
PFAS	-	per- and polyfluoroalkyl substances
PFBA	-	perfluorobutanoic acid
PFBS	-	perfluorobutanesulfonic acid
PFCA	-	perfluoroalkyl carboxylic acid
PFDA	-	perfluorodecanoic acid
PFEI	-	perfluoroethyl iodide
PFHpA	-	perfluoroheptanoic acid
PFHpS	-	perfluoroheptane sulfonic acid
PFHxA	-	perfluorohexanoic acid
PFHxI	-	perfluorohexyl iodide
PFHxS	-	perfluorohexanesulfonic acid
PFO	-	pseudo-first-order
PFOA	-	perfluorooctanoate acid
PFOS	-	perfluorooctane sulfonate
PFPeA	-	perfluoropentanoic acid
PFPEs	-	perfluoropolyethers
PFSA	-	perfluoroalkyl sulfonic acid
POPs	-	persistent organic pollutants
PP	-	polypropylene
ppt	-	parts per trillion

PSO	-	pseudo-first-order
RMSE	-	root-mean-square error
RO	-	reverse osmosis
ROS	-	highly reactive oxidant species
$S_2O_8^{2-}$	-	persulfate ions
SAFF	-	surface-active foam fractionation
SCG	-	spent coffee grounds
SD	-	standard deviation
SEM	-	scanning electron microscopy
SIM	-	selected ion monitoring
TFE	-	tetrafluoroethylene
ТМ	-	telomerization
USEPA	-	United State Environmental Protection Agency
VUV	-	vacuum ultraviolet
XRD	-	x-ray diffraction
XRF	-	x-ray fluorescence
γ-AlOOH	-	aluminium oxide hydroxide

LIST OF SYMBOLS

b	-	free energy of adsorption
°C	-	degree Celcius
С	-	constant directly linked to the boundary layer thickness
C_0	-	initial concentration
C_e	-	equilibrium concentration of the FTO solution
Cl	-	chlorine
C_t	-	concentration of FTO in the solution at the time
F	-	fluorine
f_{oc}	-	organic carbon content
Н	-	hydrogen
<i>k</i> 1/ <i>k</i> 2	-	rate constant
K_{AW}	-	air-water partition coefficient
K_d	-	distribution coefficient
K_{f}	-	adsorption capacities (Freundlich)
<i>K</i> _{id}	-	interparticle diffusion constant
KOA	-	octanol-air partition coefficient
Koc	-	organic carbon-water coefficient
K_{OW}	-	octanol-water partition coefficient
m	-	dosage of adsorbents
т	-	slope
m/z,	-	mass to charge ratio
n	-	Freundlich exponent
n	-	length of the alkyl chain in carbon atoms
q_e	-	equilibrium concentration
q_{max}	-	maximum sorption capacity
q _t /qe	-	adsorption capacity
R	-	functional group linked to the chain
R^2	-	coefficient of determination
R_L	-	dimensionless separation factor

S_W	-	water solubility
t	-	time
V	-	volume of solution
х	-	number of non-fluorinated carbon atoms