REMOVAL OF ASPIRIN FROM AQUEOUS SOLUTION USING ACTIVATED CARBON FROM KENAF FIBER AS ADSORBENT

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ABSTRACT

The discharge of aspirin into the wastewater as an emerging contaminant is considered very harmful especially in terms of its teratogenic and fatality effects towards biodiversity. Several methods had been introduced to eliminate this pollutant including the application of activated carbon as adsorbent through adsorption technique. Nevertheless, they are usually made from non-renewable sources derived from charcoal and less environmentally friendly. Thus, researches have explored the perspective of kenaf fiber as effective, renewable, and green adsorbent in aspirin removal process from wastewater. In the context of this research, the adsorbent which was the activated carbon synthesized from the kenaf fiber was prepared. Kenaf fiber was chemically activated with phosphoric acid at impregnation ratio of 1:1, carbonization temperature of 500^{o}C, and carbonization time of 3 hours. The surface properties of adsorbents were characterized by Fourier Transform Infrared Spectroscopy, Scanning Electron Microscope, Brunauer-Emmett-Teller. Thermogravimetric analysis point of zero charge and Energy Dispersive X-Ray spectroscopy. The adsorption process was done in batch mode with different parameters of contact time (0-180 minutes), initial aspirin concentrations (100-500 mg/L), adsorbent dosage (0.1-0.5 g), temperature (30 ^{o}C - 60 ^{o}C), and pH of aspirin (3-11). The best condition was attained with maximum removal efficiency (92.8%) after 120 minutes at an initial concentration of 100 mg/L, the adsorbent dosage of 0.3 g, pH of 3.45, and temperature of 30^{o}C. Desorption and regeneration studies were conducted to assess the reusability of the adsorbents which indicated that the adsorbents could be used up to three cycles. To evaluate the adsorption mechanism, equilibrium isotherm and kinetic data were determined by using Langmuir, Freundlich, and Dubinin-Radushkevich isotherm models as well as pseudo-first and second-order kinetic models. The adsorption mechanism thus fits well with the Langmuir model and the pseudo-second-order kinetic models, indicating the chemisorption mechanism. Thermodynamic parameters consisting of enthalpy change (ΔH = - 13.854 kJ/mol), Gibbs free energy change (ΔG = -1.867, -1.589, -1.239, and -0.662 kJ / mol), and entropy change (ΔS = -39.337 J / mol K) .Thus, this process had shown that the adsorption process was exothermic and occurred spontaneously. Hence, the high percentage of aspirin removal using Kenaf Fiber – Activated Carbon with a maximum adsorption capacity of 90.05 mg/g was revealed to be better than other adsorbents fabricated from other sources and could become the alternative for aspirin removal.

ABSTRAK

Pelepasan aspirin ke dalam air kumbahan sebagai bahan pencemar yang kian dikenali boleh dianggap memudaratkan terutamanya dalam memberi kesan kecacatan bentuk dan kematian kepada biodiversiti. Beberapa kaedah telah diperkenalkan untuk menyingkirkan bahan pencemar ini termasuklah penggunaan karbon teraktif sebagai penjerap dalam teknik penjerapan. Namun begitu, ia selalunya diperbuat daripada sumber yang tidak boleh diperbaharui dan kurang mesra alam. Oleh sebab itu, para penyelidik telah menyelidik perspektif serabut kenaf sebagai bahan penjerap yang efektif, boleh diperbaharui serta mesra alam dalam penyingkiran aspirin dari air kumbahan. Dalam konteks kajian ini, karbon teraktif telah disediakan daripada gentian kenaf. Gentian kenaf telah diaktifkan secara kimia dengan asid fosforik pada nisbah penyerapan sebanyak 1:1, suhu karbonisasi sebanyak 500^{o}C. dan tempoh karbonisasi selama 3 jam. Sifat-sifat permukaan bahan penjerap telah dicirikan dengan menggunakan teknik-teknik spektroskopi inframerah transformasi Fourier, mikroskop electron pengimbas pancaran, Brunauer-Emmett-Teller, teknik termogravimetrik, titik sifar caj dan Spektroskopi penyebaran tenaga X-Ray. Proses penjerapan telah dijalankan dalam mod berkumpulan dengan parameter yang berlainan iaitu masa tindakbalas (0-180 minit), kepekatan awal aspirin (100-500 mg/L), dos penjerap (0.1-1.0 g), suhu (30^{o}C – 60^{o}C), dan pH aspirin (3-11). Keadaan optimum telah dikenal pasti di mana kecekapan penyingkiran tertinggi (92.8%) selepas 120 minit pada kepekatan awal pada 100 mg/L, dos penjerap pada 0.3 g, pH 3.45, dan pada suhu 30^{o}C. Kajian nyahserapan dan penjanaan semula juga telah djalankan untuk mengkaji kebolehan guna pakai bahan penjerap yang mana menunjukkan bahawa penjerap boleh diguna semula sebanyak tiga kali kitaran. Untuk pentaksiran mekanisma proses penjerapan, data isoterma dan viinetic bagi ekuilibrium juga telah ditentukan dengan menggunakan model isoterma Langmuir, Freundlich, dan Dubinin-Radushkevich serta model viinetic pseudo-kadar-pertama dan kedua. Mekanisma penjerapan telah didapati menepati model Langmuir dan pseudo-kadar-kedua yang membuktikan bahawa penjerapan secara kimia telah berlaku. Parameter termodinamik yang terdiri daripada perubahan entalpi (ΔH = - 13.854 kJ/mol), perubahan tenaga bebas Gibbs (ΔG = -1.867, -1.589, -1.239, and -0.662 kJ / mol), dan perubahan entropi (ΔS = -39.337 J / moL K). Oleh itu, proses ini menujukkan bahawa proses penjerapan ini adalah eksotermik dan berlaku secara spontan. Peratus kecekapan peralihan aspirin yang tinggi sebanyak 90.05 mg/g bagi nilai maksimum kapasiti penjerapan menggunakan karbon teraktif daripada gentian kenaf menunjukkan bahawa ia adalah lebih baik dengan penjerap yang dicipta daripada sumber lain dan boleh menjadi alternatif dalam penyigkiran aspirin.

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LIST OF ABBREVIATIONS

Na_2SO_4	-	Glauber salt
NH_3	-	Ammonia
Pb	-	Lead
Na	-	Sodium
NaOH	-	Sodium hydroxide
S^2	-	Sulfide ion
$C_9H_8O_4$	-	Aspirin
Ir	-	Iridium
Fe_3O_4	-	Iron (II,III) oxide
SiO_2	-	Silicon dioxide
CeO_2	-	Cerium (IV) oxide
CNT	-	Carbon nanotube
C	-	Carbon
N	-	Nitrogen
O	-	Oxygen
H	-	Hydrogen
UTM	-	Universiti Teknologi Malaysia
UV-vis	-	Ultraviolet –visible spectroscopy
RMSE	-	Root Mean Square Error

LIST OF SYMBOLS

g	-	gram
min	-	Minute
L	-	Liter
J	-	Joule
K	-	Kelvin
^{o}C	-	Degree Celsius
%	-	Percent
ΔHo	-	Enthalpy
ΔSo	-	Entropy
ΔGo	-	Gibbs Free Energy
Δ	-	Delta
mg	-	Milligram
mg/L	-	Milligram per liter
mg/g	-	Milligram per gram
g/mg.min	-	gram / milligram.minute
g/L	-	gram per liter
M	-	Molar
rpm	-	Revolution per minute
mm	-	millimeter
w	-	Weight
v	-	Volume
% wt/v	-	Percentage weight per volume
Ml	-	Milliliter
cm	-	centimeter
cm^{-1}	-	Reciprocal centimeters
λ	-	Wavelength
m	-	meter
m^2/g	-	meter square per gram
cm^3/g	-	centimeter cubed per gram
Kv	-	kilovolt

nm	-	nanometer
R	-	Universal gas constant
R^2	-	Correlation coefficient
χ^2	-	Chi-square
R_L	-	Constant separation factor
kJ/mol	-	kilojoule per mole
J/mol.K	-	Joule / mole Kelvin
L/mg	-	Liter per milligram

CHAPTER 1

INTRODUCTION

1.1 Research Background

Countless development had arisen due to the rapid urbanization in the current technological realm. Various types of industries had been established including the pharmaceuticals, textiles, and agricultural. Pharmaceutical effluents were proven by Shi *et al*. (2017) to consist mainly of non-degradable compounds. Wong *et al*. (2018) proved that those discharges are extremely hard to be eliminated from wastewater due to their low concentrations (nanogram per liter).

Pharmaceutical wastes are regarded as one of incipient discharge contributor to water sources around the world (Wong *et al.,* 2018). In the United States of America, even though 80% of their streams had been polluted by those pharmaceutical wastes (Kyzas *et al.,* 2015), their trace concentrations in the water had made them difficult to be treated efficiently. Shi *et al.* (2017) deduced that chemical oxygen demand of pharmaceutical wastewater could even exceed 3000 mg/L, which is rather large, consisting mainly of pain killers, drug contained hormones, muscle relaxants, pain killers, and antibiotics (Rakić *et al.,* 2015) such as aspirin, carbamazepine, and estrone. They are considered as unfavorable substances to human health and aquatic species (Kyzas *et al.,* 2015), in an account of their enduring exposure effects towards nature and resilient towards bio-decomposition (Teo *et al*., 2016). Except for the small-scale concentrations of medicinal litters in the aquatic habitat, most of the wastewater treatments plants are still struggling in figuring out the best technique to eliminate those wastes proficiently.

Originating predominantly from medical amenities comprising of medical centers, hospitals, and health cares (Pereira *et al.,* 2019; Shi *et al.,* 2017), these effluents are promoting the discharge of detrimental medication outflow to the aquatic environment in deleterious ways. Besides, the non-biodegradability and presence of benzylic ring in those compounds (Ebele *et al.,* 2017) would cause harm to biological organisms upon their continuous exposure (Jiang *et al.,* 2015). It is substantiated by Delgado *et al.,* (2019) that at a very dejected concentration of wastewater, over 3000 biologically active pharmaceutical compounds are found due to their incomplete removal. Endocrine contained substance, for instance, could cause disruption in aquatic species if discarded inappropriately in terms of gender mutation in male fish, mainly (Chung and Brooks, 2019). Microorganisms' tendency towards drug resistance is also increased through antibiotics and pharmaceutical wastes even worsen the bad liver, kidney, and cancer diseases of people who suffer from them (Feier *et al.,* 2018). Thus, Kyzas *et al.,* (2015) contended that effectual treatment techniques must be applied either in physical or chemical means towards the pharmaceutical polluted wastewater to preserve biodiversity.

Various approaches are available in removing these pharmaceutical pollutants. That include biological membrane bioreactor (Chen *et al.*, 2019), photocatalysis (Singh *et al*., 2019), electrochemical methods (Feier *et al*., 2018), chemical coagulation, sonolysis (Patil *et al*., 2019), membrane filtration (Rosman *et al*., 2019), and adsorption (Wong *et al*., (2018). Pereira *et al*. (2019) even proposed an optimization technique by applying activated carbon as an efficient adsorbent in attaining high pharmaceutical removal efficiency. Patil *et al*., (2019) , Chowdury *et al.,* (2012), Silva *et al*., (2015) and Burakov *et al.,* (2018) deduced that adsorption is the best wastewater removal technique due to its high removal efficiency, low operability cost, resistant to toxic, and most importantly, it's reversible.

Kenaf fibers, acting as natural adsorbents, had become one of the promising alternatives in pharmaceutical elimination technology. According to Saba *et al*., (2015), kenaf originated from the African's and Asian's tropical and subtropical parts as well as deliberated as wild dicotyledons' species and currently grown in Kelantan, Malaysia. Belonging in natural fibers group, they have outstanding properties which are flimsy, high toughness, not dangerous to health, sustainable to the environment, not prone to damage caused by insects, and lastly, require less or no pesticides (Saba *et al*., 2015).

Besides, other biomass wastes are also comprehensively converted into activated carbon as the green adsorbents for a more efficient and economical process such as work done by Adebisi *et al.*, (2017) and Zhong *et al.*, (2012). Notwithstanding the vast amount of research done on the fabrication of activated carbon from lignocellulosic biomass materials, the potential of activated carbon from kenaf fiber in aspirin removal as the promising alternative has not yet to be discovered. Therefore, as per mentioned above, the kenaf fiber is selected to become a suitable adsorbent in this adsorption technology.

1.2 Problem Statement

Acetylsalicylic acid, commonly known as aspirin, is one of the most prescribed non-steroidal drug anti-inflammatory drug in the world and each year, tons of the compound is manufactured and released to the environment. Thus, the presence of carcinogenic, endocrine, and hormonal compounds in the substances make them more harmful and can cause even fatality to human and aquatic organisms. The risk severity of aspirin effect as effluent in wastewater has also brought the concern in solving this matter since it is able to cause fatality to microorganisms especially through its chronic effect from its low concentration in wastewater. It is also tremendously difficult to eliminate that substance efficiently through conventional treatments alone. In this study, the adsorption technique is mostly preferred in aspirin removal due to their simplicity in design, low operating cost, apart from its excellent removal capacity.

Current commercial activated carbon, that is commonly used as the adsorbent in the technique is also typically made of non-renewable sources and possesses higher environmental risk. Hence, in this study, kenaf fiber is chosen to be the agro-waste and renewable source of the adsorbent. Several researchers are also developing the application of this material in wastewater technology as adsorbents in terms of the fiber’s excellent mechanical and physical abilities such as non-abrasive, lightweight, and non-toxic. Besides, their low dense and great filling capacity properties would enable them to be further chemically modified as the adsorbent. The presence of lignocellulose, reactive hydroxyl groups, and high carbon content in the fibers also help to quicken the adsorption process with some chemical modifications. Hence, it is necessary to introduce the application of kenaf fiber as the new alternative for the precursor of new activated carbon.

Latterly, experimentations were done by Sajab *et al.*, (2011), Chowdhury *et al.*, (2012), and Rahman *et al.*, (2017) on kenaf fiber as adsorbent has been efficaciously conducted in sequestering methylene blue, copper (II) ions, and earth metals respectively. Nonetheless, limited work had been conducted on the kenaf fiber efficiency and ability in removing aspirin from wastewater. Therefore, phosphoric acid was selected as the activating agent in this study to enhance the lignocellulose and hydroxyl group reactivity for better adsorption capacity of the adsorbent. Thus, in this research, the performance of the kenaf fibers as activated carbon in adsorbing aspirin from aqueous solution is studied.

1.3 Objectives of Research

This research mainly aims to evaluate the performance of the synthesized adsorbent from the activated carbon derived from the kenaf fiber in removing the aspirin from the aqueous solution. To achieve the goals, the objectives are derived as follows:

(a) To synthesize and characterize the activated carbon from kenaf fiber in terms of its surface area properties, thermal and elemental composition.
(b) To study the performance of the newly synthesized adsorbent in the removal of aspirin.
(c) To determine the adsorption mechanism in terms of its isotherm, kinetic, and thermodynamic models which are associated with the adsorption process.

1.4 Scope of Research

The scope of the research is defined as follows:

(a) The synthesis of the activated carbon was done by chemical activation method with the best chemical activating agents (zinc chloride, potassium hydroxide, phosphoric acid), best impregnation ratios (1:1, 1:4, 1:6, 4:1, and 6:1), best carbonization temperature and time (400, 500, and 700oC) at (1, 3, and 5 hours) respectively.

(b) The surface morphology, textural properties, functional groups, thermal and elemental analysis of the raw kenaf fiber and synthesized adsorbent were characterized by Variable Pressure Scanning Electron Microscopy (VPSEM), Brunauer-Emmett-Teller (BET), Fourier Transform Infrared Spectroscopy (FTIR), Energy Dispersive X-Ray spectroscopy analysis, Thermogravimetric analysis, and point of zero charge.

(c) Determination of the performance of the newly synthesized adsorbent based on the adsorption parameters in batch experiments which are the effects of contact time (0-180 minutes), initial concentration of aspirin (100-500 mg/L), adsorbent dosage (0.1-1 g), pH (3-11), and temperature (30-60oC).

(d) Evaluation of the isotherm models (Langmuir, Freundlich, Dubinin-Raduskevich), kinetic models (pseudo-first order, pseudo-second order), and thermodynamic analysis (enthalpy, entropy, Gibbs free energy) correlated with the adsorption mechanism.

(e) Analysing the regeneration study of the activated carbon kenaf fiber by means of ethanol as desorption agent up to 50% percentage removal of aspirin.

1.5 Significance of Research

In this research, this new approach would reduce the environmental risk as it makes use of renewable agricultural sources from plants. The application of activated carbon from kenaf fiber as precursor also will yield high removal efficiency and adsorption capacity of the aspirin from wastewater as it is more feasible process if it is to be operated in the best circumstances. Thus, this study would provide an alternative for the existing commercial activated carbon.

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