THE OCCURRENCE, TRANSPORT MECHANISMS, AND REMOVAL OF SILVER NANOPARTICLES IN THE WATER ENVIRONMENT

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ABSTRACT

There is a heightened concern from communities on silver nanoparticles (AgNPs) disposal from industrial products into the water environment because of their toxicity to aquatic habitats. Aeration and coagulation methods to remove AgNPs have limitations such as complicated procedure involvement and the use of toxic materials. Hence, adsorption has the potential to be a simple and greener method to remove AgNPs. The aim of this study was to evaluate the occurrence, transport mechanisms, and removal of AgNPs in water environment. Water samples were collected from two sewage treatment plants (STPs), Melana River, and Skudai River. Effects of AgNPs on bacteria isolated from the rivers and STPs were also investigated. Transport mechanism of AgNPs in river was evaluated using constructed river model as a pilot study. AgNPs removal from the water via two different activated carbons derived from oil palm shell (ACfOPS) and coconut shell (ACfCS) were examined. In this respect, adsorption mechanism of AgNPs onto the proposed activated carbons was evaluated using physio-chemical approaches. In terms of kinetic behaviors, evaluation of nine kinetic adsorption models using six statistical indicators were scrutinized. Specific analytical methods namely UV-Vis spectrophotometer, FTIR, FESEM and SEM-EDX, and molecular analysis have been employed to gather the objectives. This study found that the concentration of AgNPs in the rivers and STPs ranges from 0.1 to 10.2 mg/L and 0.1 to 20.0 mg/L, respectively. AgNPs have antibacterial capability against all examined bacteria depending on their size and bacteria types. In the transport mechanism inspection, this study found that about 95% of the entering AgNPs into the river system was transmitted via the effluent. ACfOPS and ACfCS removed up to 99% of AgNPs from the water. The interaction mechanism between AgNPs and the activated carbon surface was mainly due to electrostatic force interaction via binding Ag^{+} with O^{-} presented in the activated carbon to form AgO. Experimental adsorption data can be best described using the mixed 1,2-order kinetic model. Furthermore, this study also found that the proposed model outperformed existing kinetic models that share the same number of parameters. Findings from this study are useful for the monitoring of AgNPs in water environment and providing a method to remove AgNPs.

ABSTRAK

Terdapat kebimbangan yang tinggi daripada komuniti tentang pembuangan nanopartikel perak (AgNP) daripada produk industri ke dalam persekitaran air kerana kesan toksik terhadap habitat akuatik. Kaedah pengudaraan dan penggumpalan untuk penyingkiran AgNP adalah terbatas kerana penglibatan prosedur yang rumit dan penggunaan bahan toksik. Oleh itu, kaedah penjerapan memiliki potensi untuk menjadi kaedah yang mudah dan mesra untuk penyingkiran AgNP. Tujuan kajian ini adalah untuk menilai kewujudan, mekanisme pergerakan dan penyingkiran AgNP dalam persekitaran air. Sampel air diambil daripada dua loji rawatan kumbahan (STP), Sungai Melana dan Sungai Skudai. Kesan AgNP pada bakteria yang diisolasi daripada sungai dan STP juga disiasat. Mekanisme pergerakan AgNP dalam sungai dinilai menggunakan model sungai yang telah dibina sebagai kajian rintis. Penyingkirannya AgNP daripada air menggunakan dua karbon teraktif yang diperolehi daripada tempurung kelapa sawit (ACfOPS) dan tempurung kelapa (ACfCS) telah dikaji. Dalam hal ini, mekanisme penjerapan AgNP oleh karbon teraktif yang dicadangkan telah dinilai menggunakan kaedah fizik-kimia. Dari segi sifat kinetik, penilaian terhadap sembilan model penjerapan kinetik dengan menggunakan enam petunjuk statistik telah diteliti. Kaedah analisis yang spesifik iaitu spektrofotometer UV-Vis, FTIR, FESEM, SEM-EDX dan analisis molekul telah digunakan untuk mencapai objektif. Kajian ini menemukan bahawa kepekatan AgNP di sungai dan STP adalah masing-masing antara 0.1 hingga 10.2 mg/L dan 0.1 hingga 20.0 mg/L. AgNP memiliki keupayaan antibakteria terhadap semua bakteria yang dikaji bergantung kepada saiz AgNP dan jenis bakteria. Di dalam penelitian mekanisme pergerakan, kajian ini mendapati bahawa lebih kurang 95% AgNP yang masuk ke dalam sistem sungai adalah melalui effluen. ACfOPS dan ACfCS masing-masing telah menyingkirkan sehingga 99% AgNP daripada air. Mekanisme interaksi antara AgNP dan permukaan karbon teraktif ialah utamanya disebabkan oleh interaksi kuasa elektrostatik melalui pengikatan Ag^+ dengan O^- yang berada di karbon teraktif untuk membentuk AgO. Data penjerapan eksperimen dapat diterangkan secara baik dengan menggunakan model kinetik campuran 1,2. Kajian ini juga telah mendapati bahawa model yang dicadangkan adalah lebih baik berbanding model kinetik sedia ada yang mempunyai bilangan parameter yang sama. Hasil daripada kajian ini berguna untuk pemantauan AgNP dalam persekitaran air dan menyediakan kaedah untuk penyingkiran AgNP.

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LIST OF ABBREVIATIONS

ACfCS	-	Activated Carbon Derived from Coconut Shell
ACfOPS	-	Activated Carbon Derived from Oil Palm Shell
$AgNO_3$	-	Silver Nitrate
AgNPs	-	Silver Nanoparticles
AP	-	*Aeromonas punctata*
BLAST	-	Basic Local Alignment Search Tool
GB	-	Glass Bead
LS	-	Large Pore Mesoporous Silica
MAD	-	Mean Absolute Deviation
MAPE	-	Mean Absolute Percent Error
NND	-	National Nanotechnology Directorate
OMP	-	Aged Iron Oxide Magnetic Particles
PCR	-	Polymerase Chain Reaction
PCSOMP	-	Fe_3O_4@ Polydopamine Core-Shell Microspheres
PEI	-	Nitrogen-Rich Core-Shell Magnetic Mesoporous Silica
RMSE	-	Root Mean Squared Error
River 1	-	Melana River
River 2	-	Skudai River
STPs	-	Sewage Treatment Plants
STP 1	-	STP at Taman Universiti
STP 2	-	STP at Desa Skudai
TR	-	Trypsin
USEPA	-	United States Environmental Protection Agency

LIST OF SYMBOLS

a_{RP}	-	Radke-Prausnitz equilibrium constant
C_i	-	Initial concentration
C_f	-	Final concentration
E_{max}	-	Percentage of error in maximum estimated value
E_{min}	-	Percentage of error in minimum estimated value
q	-	Adsorption capacity
k_1	-	First-order rate constant
k_2	-	Second-order rate constant
K_a	-	Affinity constant
K_{H}	-	Hill isotherm constant
k_{flfs}	-	Fractal-like mixed-1,2-order coefficient
n_{RP}	-	Radke-Prausnitz model exponent
V	-	Volume
W	-	Mass of the activated carbon

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Nanomaterial is becoming vitally important in many industrial fields. It has more superior properties compared to its larger material. The novel properties of nanomaterials have been exploited in various areas such as automotive, bio-medical, space, electronic, sensor, and water treatment applications. In addition, it is well established that the finer grain size can produce denser materials thus its mechanical properties can be improved three times better than the microstructure version (Corbett *et al.*, 2000).

Silver nanoparticles (AgNPs) are a class of materials with the sizes ranging from 1 nm to 100 nm. The interest to study AgNPs in various different behaviors has recently increased due to their unique and attractive physical, chemical, and biological properties. AgNPs are recognized as the highest level of commercialization which accounts for 55.4% of the total nanomaterial-based consumer products that are available in the market (Agnihotri *et al.*, 2014). In general, AgNPs are widely explored for medical devices, water treatments, sensors, cosmetics, textiles, catalyst, and food packaging.

Although it has been reported that AgNPs have toxicity properties that can be hazardous to zebrafish (Asharani *et al.*, 2008), hazardous to the human reproductive system as well as lethal to cell-based in-vitro systems (Skebo *et al.*, 2007; Braydich *et al.*, 2005; Hussain *et al.*, 2005), and can inhibit bacterial growth (Sondi and Sondi, 2004), the nanoparticles are still abundantly utilized in several commercial products. Due to such health concerns, a number of researchers have carried out measurements and reported that several consumer products released AgNPs into the environment in a large amount (Benn and Westerhoff, 2008). The aforementioned occurrence raise potential environmental and health alerts since the toxicity threat of AgNPs can be observed near the vicinity of consumers particularly in the freshwater ecosystem.

1.2 Problem Statement

The worldwide production of AgNPs was about 500 tons per year and was predicted to increase up to 800 tons per year by 2025 (Calderón-Jiménez *et al.*, 2017; Mueller and Nowack, 2008). Of central concern to the industry stakeholders, policymakers, communities, as well as researchers, however, is their disposal into the water environment such as rivers. Since the vast majority of the sources for drinking and household uses particularly in Malaysia are greatly dependent on the water from rivers, the current status of AgNPs in the water system should be well-understood. Also, the effects of AgNPs to the living organism such as bacteria isolated from these water bodies should be clearly investigated.

When AgNPs enter the water system, the nanoparticles are released into the water and are then transmitted through the water flow or the nanoparticles form sediment on the bottom of the system or on the surface of the land. Study on the transport mechanism of AgNPs through water bodies is still very rarely investigated so that some important questions remain unanswered. The most crucial question in this process is what is the main parameter that governs the process so that more exciting particles settle as sediment or are transported following the water flow. In general, it is a complex process, which depends on the particle size, flow speed, and other physio-chemical conditions. In order to understand the process, there is a need for a comprehensive study to verify the mechanism.

For AgNPs removal, recent scientific literature has invoked somewhat contradictory findings on the interaction mechanism between nanoparticles as the adsorbate and natural materials as the adsorbent. In the current work context, whether nanoparticles bind to the carbon, oxygen, or other constituents is still an inconclusively debated subject. In addition, although several mathematical models have been proposed and examined to describe the kinetic behaviors of AgNPs adsorption, no clear consensus has been achieved as to which model is more practically relevant. Therefore, a kinetic model with a simple expression having a manageably low set of parameters is still crucially needed for convenience and widespread use. Also, one major shortcoming found is the use of determination coefficient as the only statistical evaluating measure, which is deficient to gauge an overall competency.

1.3 Objectives of the Study

The present work aims to evaluate the behaviors and removal of AgNPs in the water environment. To achieve the purpose of the study, the following objectives were carried out:

(a) To verify the occurrence of AgNPs in sewage treatment plants (STPs) and rivers

(b) To evaluate the effects of AgNPs on bacteria growth isolated from STPs and rivers

(c) To evaluate the transport mechanisms and distribution behaviors of AgNPs in simulated water system

(d) To evaluate the performance of activated carbons derived from agricultural wastes as the potential natural adsorbent for removal of AgNPs and evaluate its adsorption mechanism

(e) To develop and evaluate a new simple kinetic model for describing AgNPs adsorption behaviors for different adsorbents

1.4 Scope of the Study

Scopes of the study are as follows:

(a) This study focuses on the estimation of AgNPs concentration in the water of STPs and rivers. Two STPs, located at Taman Universiti and Desa Skudai, were selected for this investigation. Also, two rivers, Melana River and Skudai River, were selected as sampling locations. These rivers were selected because the locations directly receive the effluent of the STPs. AgNPs concentration was estimated using the inductively coupled plasma optical electron spectrometry approach.

(b) The effect of AgNPs on bacteria isolated from the STPs and rivers was also examined. In this examination, AgNPs were synthesized using green approach from plant and weed extracts. Identification of bacteria was carried out using the polymerase chain reaction (PCR) method and their sequencing was identified using the basic local alignment search tool (BLAST) analysis. Simple and rapid analyses to evaluate the degradation bacteria such as the zone of inhibition were selected.

(c) Verification of the transport mechanism and the properties of AgNPs was evaluated using a lab-scale study. A river channel for transportation of AgNPs was constructed using a clay soil mounted on a PVC gutter. The presence of AgNPs in the water was investigated at three sampling points, which are at the near influent, middle, and effluent. After the experiment, the presence of AgNPs in sediment was also inspected. Properties of AgNPs during their transport were evaluated by inspecting their distribution behaviors.

(d) The present study proposed potential adsorbents using activated carbons derived from agricultural wastes. Physical and chemical properties of the proposed activated carbons were characterized. Several existing mathematical models were employed for the simulation approach. The model performance was judged using six statistical parameters. Moreover, the possible mechanism of the adsorption of AgNPs onto the employed adsorbents was proposed and verified.

(e) Lastly, the development of a new simple kinetic model to describe the kinetic behaviors of AgNPs onto activated carbon was carried out. The model was verified using experimental adsorption data carried out in this study and compared with other existing two parameter kinetic models. As an additional validation, the model was also tested using secondary AgNPs adsorption data obtained from various sources.

1.5 Limitation of the Study

Limitations of the study are as follows:

(a) Verification of the occurrence of AgNPs in STPs and rivers were only collected from two local STPs, located at Taman Universiti and Desa Skudai and two rivers, Melana River and Skudai River. A grab sampling procedure was selected for the water sampling at the STPs and rivers according to the method by the United States Environmental Protection Agency (USEPA). The water was sampled from raw water in the STPs and water in river after the STPs.

(b) Evaluation of the effects of AgNPs on bacteria growth isolated from STPs and rivers were evaluated using zone of inhibition study. In addition, AgNPs were synthesized using green approach without comparison with commercial AgNPs or other synthesis procedures.

(c) Evaluation of the transport mechanisms and distribution behaviors of AgNPs in simulated water system was evaluated using a lab-scale study. A river channel for the transportation of AgNPs was constructed based on scaling down approach without similarity analysis.

(d) Evaluation of the performance of activated carbons derived from agricultural wastes as the potential natural adsorbent for removal of AgNPs and its adsorption mechanism was carried out without any pH adjustment and comparison with commercial activated carbons as a control.

(e) Development of a new simple kinetic model for describing AgNPs adsorption behaviors for different adsorbents was based on mathematical perspective combined with empirical approach. Evaluation of the model was carried out using primary and secondary AgNPs adsorption data obtained from various sources. The performance evaluation of the proposed model was based on statistical analyses.

1.6 Significance of the Study

Although Malaysian government has launched National Nanotechnology Directorate (NND) for nanotechnology development, AgNPs are a new field in Malaysia. No official regulation was referred under the government to monitor and control AgNPs use in Malaysia. It is well established that AgNPs are widely used in household products, textiles, food containers, consumer products, and electronic devices. Also, there is supported by fact that several company focusing for producing AgNPs are established in Malaysia. This study can be an alternative suggestion for making regulation specifically to control and monitor the use of AgNPs.

Understanding on the transportation of AgNPs via water bodies is still limited because it needs a deep knowledge from the experimental and theoretical perspectives. Findings from the present study clarify several questions which are what extent do AgNPs pass the water flow, are AgNPs in the water sorbed to sedimentation or do AgNPs occur as freely dispersed AgNPs, and how does the speciation of the AgNPs change during their passage through the water flow. For the removal of AgNPs, findings from this study make several contributions to current literature. First, the use of natural adsorbents as proposed in this work provides an alternative material to remove AgNPs from water environment. Secondly, verification of AgNPs deposition onto the proposed natural adsorbents assists the understanding of the role of removal mechanism of AgNPs via the adsorbents. Lastly, development of a new kinetic model with a simple expression provides a basis for convenience and widespread use.

1.7 Thesis Outline

Content of this thesis is divided into six chapters. Each chapter is briefly presented as follows. The first chapter provides a general introduction by presenting background, statements of the problem, aim and objectives, scopes, limitations, and significance of this study. In addition, the outline of this thesis is also provided. In the second chapter, an overview on scientific literature of AgNPs from their global perspective, procedure to synthesis, properties, transport behaviors in the environment, removal, and summary is given. Chapter three contains a comprehensive methodology employed in this study. This includes description of investigation of the presence of AgNPs in the rivers and STPs. It is then followed by presenting the procedure to investigate the effects of AgNPs on a microorganism isolated from the water, AgNPs transport mechanism verification, and removal of AgNPs via adsorption. Chapter four demonstrates results and discussion focusing on the behaviors of AgNPs in the water systems. In general, this chapter offers results and discussions particularly devoted for objectives 1, 2, and 3. Chapter five gives results and discussion concentrating on the removal of AgNPs from the water via natural activated carbons derived from agricultural wastes. Performance of the proposed activated carbons via experimental and numerical perspectives is discussed. Next, evaluation of a new simple kinetic model is also presented. The chapter is devoted for objectives 4 and 5. At the end, chapter six recapitulates the main finding of the study and offers recommendations for future works.

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