Adsorption of cadmium using teak leaves powder (*Tectona grandis sp.*): embedding Mahaffy’s tetrahedral concept in undergraduate chemical engineering capstone lab project

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Abstract. Chemical engineering education can be aptly structured on four central components: macroscopic, particulate, symbolic, and human element domains; known as the Mahaffy’s tetrahedral concept. This paper discusses the application of the concept of undergraduate chemical engineering capstone projects. The capstone was carried out as an undergraduate final laboratory project to investigate the adsorption of heavy metal, namely cadmium (Cd), using powder of teak leaves (*Tectona grandis sp.*). The lab experiment was environmentally safe, completed at ambient temperatures and that the experimental data collection was easy. It was actively guided by lecturers in a way to encourage meaningful lab experience as proposed in the Mahaffy’s tetrahedral idea. Three experimental variables were investigated: initial pH solution, adsorbent dosage, and initial concentration of Cd. The results were analyzed to determine the diffusion mechanism and kinetics through Weber-and-Morris intra-particle diffusion model. The experimental results agreed with most published literature which gave satisfaction on both students and lecturers. To assess learning outcomes, pre- and post-evaluations in regard the Mahaffy’s tetrahedral idea was employed. The post-test scores were significantly higher than those of the pre-test indicating that the learning outcomes were achieved. The lab project was also found to increase the student’s writing and presentation skills.

1. Introduction

Wastewater may contain hazardous metal ions such as Cu$^{2+}$, Zn$^{2+}$, Pb$^{2+}$, Cd$^{2+}$, Cr$^{2+}$, et cetera [1]. Some of these metal ions are essential for human health but deleterious at higher concentrations due to a variety of diseases that is related to exposure of these ions [2]. Adsorption is one of the popular methods used in wastewater treatments especially for the removal of heavy metals. It has several advantages over other methods such as chemical precipitation, coagulation-flocculation, ion exchange, etc [3]. Moreover, due to environmental concerns, adsorption using agricultural wastes has been an extensive area of research to remove metal ions from wastewater [4,5]. In such a case, the adsorption method is seen as environmentally friendly.

Chemical engineering students may have a basic understanding of adsorption [6] since adsorption is taught within the core subjects, namely Process Operations or Equilibrium Staged Processes at chemical engineering undergraduate levels. Principles of adsorption are also prevalent in many chemical industries [7]. However, the students may not realize that adsorption involves basic concepts such as equilibrium, kinetics, and thermodynamics.
In this paper, a capstone laboratory experiment on adsorption of Cd\(^{2+}\) using powder of dried teak leaves (\textit{Tectona grandis sp.}) is discussed. The experiment was carried out individually by the student to enhance student understanding of the concepts underlying adsorption processes. The student conducted the experiment, analyzed the experimental data, plotted the graphs and so on under the supervision of lecturers. Emphasis was placed on analyzing the adsorption in terms of diffusion mechanism through Weber-Morris model \cite{8}.

2. Mahaffy’s tetrahedral metaphor
Metaphors are used widely in science and engineering education since many phenomena are too difficult to explain without resorting to illustrations or comparisons. In studies loaded with chemistry such as chemical engineering, numerous facts are related to things virtually too small for human perceptions: molecules, atoms, or ions. In relation to such concepts, one commonly used metaphor is the planar triangular metaphor developed by Johnstone \cite{9}. The three vertices of the Johnstone’s planar triangle (figure 1(a)) represent macroscopic, molecular, and symbolic thinking levels, respectively. Then, enriching the triangle representation, another vertex was proposed by Mahaffy \cite{10}, making it a 3D tetrahedral metaphor (figure 1(b)). The fourth facet encompasses the human context since it is human beings who create, use, and get affected by chemicals. Indeed it is human beings who associate with industrial processes, economic benefits, and environmental implications.

It can be argued that the human aspect is not inherently the domain of chemistry as a science. Instead, such an aspect is associated with the application and/or the consequences of chemistry. Such argumentation, however, is not considered in the present article. This article discusses an attempt to embed the 3D metaphor of Mahaffy into undergraduate chemical engineering capstone lab projects under the pretext that not only the scientific aspects but also the human issues are to be inculcated in students learning. Graphical representation of the two metaphors is compared in figure 1.

![Domains of chemistry knowledge: (a) Johnstone’s triangle; (b) Mahaffy’s tetrahedron.](image)

3. Student capstone lab project
The capstone lab project is mandatory as a partial requirement for the completion of an S1 (= bachelor) degree at the authors’ institution. It is carried out by the students individually and supervised by two lecturers. The student completes the capstone in three stages: preparing a capstone proposal, implementing the experiment, and writing up the capstone report. The time allotted to the capstone is typically one semester. Detailed description of a representative capstone lab project at our institution is presented in the literature \cite{11}.

In the current paper a capstone lab project on adsorption of cadmium ions (Cd\(^{2+}\)), using dry teak leaf powder (\textit{Tectona grandis sp.}) is reported and discussed in the light of Mahaffy’s tetrahedral concept.
4. Implementation of the capstone lab project
The student was tasked to carry out the capstone lab project individually under the supervision of two lecturers according to the procedure as follows.

4.1. Preparation
Dried fallen leaves of teak wood [Tectona grandis sp] were collected from our campus yard and gardens. The leaves were washed thoroughly with tap water to remove dust and other particulates, followed by rinsing with distilled water. Next, the cleaned leaves were oven-dried (60°C, 24 h) until a constant weight was achieved. Then, the leaves were pulverized using a household blender and sieved to obtain the powder of the sieve-cuts: 50–80 mesh. The powder was coded TLP (= teak leaf powder) and stored in air-tight containers until used as adsorbents.

Subsequently, a stock solution of 1000 mg L⁻¹ concentration of Cd²⁺ was prepared by dissolving 4.785 g of CdCl₂ in doubly distilled water to obtain a volume of 1000 ml. The experimental solutions of varying concentrations for the adsorption were obtained by successive dilution of the stock solution. The pH of the experimental solutions was adjusted to the desired values with 0.1 M HCl. All chemicals used were analytical grade and used without further purification.

4.2. Adsorption experiment
Batch adsorption experiments were carried out to investigate the effect of initial pH of the solution, adsorbent dose, and initial Cd²⁺ concentration on the adsorption of Cd²⁺ on TLP.

In all experiments, fixed volume (100 mL) of Cd²⁺ were placed in conical flasks, mixed with the TLP and thoroughly stirred at a constant speed of 250 rpm. The experiments were done at ambient temperatures for up to 90 min. Different conditions of the initial pH of the solution (4.0 and 6.0) were evaluated. At the end of the adsorption process run, the solutions were filtered through ash-less filter paper.

The concentrations of Cd²⁺ remaining in the filtrates were analyzed using flame atomic absorption spectrophotometer (AAS-Varian, AA240). The percentage adsorption of Cd²⁺ adsorbed from the solution by TLP was calculated using Eq. (1):

\[ \% \text{ adsorption} = \left(\frac{C_0 - C_e}{C_0}\right) \times 100, \]

where \( C_0 \) and \( C_e \) were the initial and equilibrium Cd²⁺ concentration (mg L⁻¹), respectively. The amount of Cd²⁺ adsorbed at time \( t \), i.e. \( q_t \) (mg g⁻¹) was calculated as Eq. (2):

\[ q_t = \frac{(C_0 - C_e)V}{m} \]

where

\( C_e \) = concentration of Cd²⁺ in solution at time \( t \), (mg.L⁻¹)
\( V \) = volume of solution, L
\( m \) = mass of TLP, g.

The adsorption at equilibrium, \( q_e \) (mg g⁻¹) was measured using Eq. (3) as follows:

\[ q_e = (C_0 - C_e) \frac{V}{m} \]

Finally, Weber-Morris model was investigated to elucidate the adsorption mechanism. Eq. (4) is utilized for the graph plotting of the model, as shown in the following.

\[ q_t = kt^{1/2} + 1, \]

where,

\( q_t \) = adsorbent uptake, m.g⁻¹
\( t^{1/2} \) = square root of adsorption time, min,
\( k \) = diffusion rate constant.

4.3. Characterization of TLP
The TLP used (see subsection 4.1) was characterized before and after the adsorption, to examine the change in morphology and surface characteristics, if any. The characterization was done through
scanning electron microscopy (SEM) analysis, with an accelerating voltage of 30 kV (FEI-Inspect-S-50). The SEM micrographs obtained were also useful for instilling the molecular concept (see figure 1) to the student.

5. Results and discussions

The relationship between contact time and amount of Cd adsorbed is depicted in the following figure (figure 2). As can be seen, the adsorption proceeded rapidly at the start of the experiment: 0.0 to 20.0 min. Next, the adsorption leveled off from 20 min onwards. In the beginning, large amounts of pores of the TLP are still available for adsorption. Hence, fast adsorption occurred. As contact time increases the pores were gradually saturated with adsorbents, resulting in less and finally no adsorption. Additionally, higher pH (4.0 to 6.0) resulting in less adsorption. This can be due to Cd$^{2+}$, being metallic, tends to form complexes/hydroxides as pH increases [12,13], rendering less cadmium ions available for adsorption onto TLP.

![Figure 2. Plot of percentage of Cd adsorbed as a function of contact time](image)

The current capstone lab project investigated the diffusion mechanism involved in the adsorption process, therefore Weber-Morris model was studied. As can be seen, the graph of the model as formulated by Eq. (4), shows two consecutive linear plots (figure 3). These two linear lines indicate that the adsorption proceeded in two steps: external mass transfer, followed by intra-particle diffusion [5]. The external mass transfer occurs rapidly from the solution onto the adsorbent surface through film diffusion. This is shown by the first segment of the lines with steeper slopes (figure 3). Then, the subsequent adsorption proceeds through internal pores of the adsorbent, termed intra-particle diffusion. As can be seen (figure 3), the slopes of the second segment are less steep.

It can be inferred from the graph (figure 3) that the adsorption of Cd$^{2+}$ using TLP, within the constraint of the present experimental parameters, took place in two consecutive steps as detailed previously. The diffusion rate constants, k, was 2.61 mg.L$^{-1}$.sec$^{-1/2}$ (for external mass transfer), and 0.357 mg.L$^{-1}$.sec$^{-1/2}$ (for intra-particle diffusion), respectively; which reasonably agreed with published values [8].
6. Pedagogical assessment
Mahaffy’s metaphor is depicted as a tetrahedron with each vertex denoting different thinking levels: macroscopic, sub-microscopic, symbolic, and human aspect. The current capstone lab project was analyzed by examining the applicability of Mahaffy’s metaphor as presented in the following.

6.1. Macroscopic mode
In order to investigate the student macroscopic level thinking, it is necessary to examine the ability of the student to think spontaneously about the facts that are observable, perceived or experienced. The evidence of such ability can be observed in the coherence of the student responses. The student should answer the following questions.

Q1 (= Question 1): What have you observed from the experiment?
- Answer: From the experiment what I observed was that the adsorption of Cd using powder of dried teak leaves was influenced by initial solution concentration, pH of the solution, and adsorbent dosage.
- Comment: The student could have misunderstood the question. He should describe what was physically observable. E.g. was there any change in the color of the solution during adsorption? Instead, the conclusion of the experimental results was given. Also, it could be possible that the question posed was neither clear nor well-defined.

Q2: Why did the powder was made as fine as possible?
- Answer: The leaves powder was made as fine as possible to increase the surface contact with the cadmium ions; so that the adsorption capacity increases. In addition, it was meant to homogenize the adsorbent (with finer mesh size, it was hoped that the powder will be more homogeneous so that the experiment can be observed easier at different variables.).
- Comment: “the adsorption capacity increases” – this statement is too vague. Need to be specific.

Q3: What was the effect of stirring the solution?
- Answer: Stirring served to accelerate and optimize the adsorption process. Considering that the leaf powder possesses a very light mass the adsorption would take longer without stirring.
- Comment: This answer is speculative. What is precisely meant by “to accelerate” and “to optimize”? The second statement (“Considering that …”) is acceptable. Without stirring the powder would stay afloat hence not readily adsorb the cadmium ions.
6.2. Sub-microscopic mode

In the current context, the sub-microscopic level thinking refers to the ability to think spontaneously of ions, atoms, or molecules and their behaviour. The student was tasked to illustrate the experiment in terms of atomic and molecular behaviour.

![before adsorption](before.png) ![after adsorption](after.png)

**Figure 4.** SEM micrographs of the teak leaf powder, before and after adsorption.

Q1: How can you elaborate the SEM micrographs of the TLP as presented in figure 4?

- **Answer:** As can be seen in the graph, before adsorption (left) the surface of the pores is smooth. On the other hand, after adsorption (right) the same surface looks grainy which may indicate the presence of the adsorbed metallic ions.

- **Comment:** The answer is reasonably correct. During the capstone project, a lot of journal references were provided by the lecturers. Some of the references contain SEM micrographs of agricultural-waste powder used for adsorption, before and after application. As an extra, and to enrich the student knowledge on the microscopic aspect, the TLP was characterized through a scanning electron microscope (SEM). Such characterization involves significant cost, therefore the analysis was personally the responsibility of the lecturers, and was not part of the capstone lab activities.

Q2: How are the Cd ions adsorbed onto the surface of the adsorbent?

- **Answer:** Metal ions of Cd were adsorbed onto the surface of the powder by diffusion through the film layer surrounding the powder, and this diffusion process occurred very rapidly. After that, the metal ions would enter the pores of the powder through the intra-particle diffusion process. This process was confirmed by the graphs which relate the amount of the metal ions adsorbed versus adsorption contact time.

- **Comments:** The oral presentation is required for the capstone lab results, and the student may have felt nervous elaborating the adsorption process. However, the student had more time when putting an explanation into writing. Hence, the description is better.

6.3. Symbolic mode

The student proficiency of symbolic level thinking can be manifested in how the student spontaneously able to explain the use of equations, formulas and so on.
Q1: The adsorption behaviour of the teak leaf powder can be explained by the intra-particle diffusion model known as the Weber-and-Morris equation. Please write the formula and explain.

- **Answer:** Adsorption processes from liquid onto a solid surface occur through a diffusion mechanism as shown by the Weber-Morris equation as follows:
  \[ q_t = k t^{1/2} + 1 \]

where,
- \( q_t \) = adsorbent uptake, mg g\(^{-1}\),
- \( t^{1/2} \) = square root of adsorption time, min,
- \( k \) = diffusion rate parameter.

Q2: Draw a graphical representation of the adsorption mechanism.

- **Answer:** Figure 5.
- **Comment:** The student copied a graphical abstract depicting a heterogeneous porous surface from an open-access source. Such should be acceptable. It was advised that copying from other sources must include acknowledgment, even if they are Open Access. The source reference was then given as shown in figure 5.

Q3: What can be inferred from the Weber-Morris plot obtained (see figure 3)?

- **Answer:** The Weber-Morris plot is not linear for the whole range of the experimental parameters tested. Thus, it could be possible that the adsorption took place in more than one stage, to be exact: two stages.
- **Comment:** Correct! You should have drawn the graph showing two distinct linear plots. Additionally, the linear equation for the two parts should have been formulated too.

6.4. Human element mode

To examine student understanding the application of adsorption in industry and its implications on society and human beings at large, the following questions were proposed for the student to complete.

Q1: What is the industrial application of adsorption process?

- **Answer:** For sugar refining (sugar whitening) in sugar industries using diatomaceous earth (food grade), and also bone ash. Utilization of activated carbon as adsorbent for gaseous substances, e.g. used in masks. Activated carbon pellets for diarrhea treatment. Alum \([\text{Al}_2(\text{SO}_4)_3]\) for water softening, and so on.
• Comment: Not all answers are correct. For example, the use of alum for water softening is not necessarily an adsorption, but a flocculation and coagulation process.

Q2: What are the advantages of adsorption for the removal of heavy metals from wastewater?
• Answer: Heavy metal removal using adsorption is relatively simple compared to other available methods. Additionally, for very low metal ion concentrations, the adsorption technique is highly efficient. Further, the adsorption process does not yield negative side effects such as toxic wastes.
• Comment: The answers are correct. All the answers can be found/traced back in journals used as references for the capstone project.

6.5. Overall comment:
As shown in the string of questions and answers previously, it seems that student understanding on adsorption processes has evolved and been enhanced. However, some limitations should also be acknowledged, i.e. those beyond lecturers’ control. These may include student’ inherence intellect, his interest in the capstone topic, his lab dexterity and self-discipline, and time allocation.

7. Writing and presentation skills
As reported earlier [11], the capstone lab project was found to increase student writing and presentation skills. The same result occurred for the current capstone. The student was tasked, as an extra, to write an article based on the results of the lab project after some discussions regarding journal paper writing with the supervising lecturers. The article handed in (not included in this article) shows consistency to scientific writing, although in some instances significant editing is required. As an example, the written text lacked of appropriate referencing styles. Instead of meticulously placing the citations right after the statements, all references were placed at the end of the text, as is common for bibliographic styles. As proposed previously, some extrinsic factors are beyond the control of the supervising lecturers.

8. Conclusions
Application of Mahaffy’s tetrahedral metaphor on adsorption experiment within the undergraduate capstone lab project in chemical engineering curriculum is presented. The results of the experiment was analyzed and discussed using the Weber-Morris model to examine the diffusion mechanism of the adsorption. It was found that the results of the experiment, as evidenced from the calculated rate constant, reasonably agreed with published values.

The embedding of the metaphor indicated that student understanding of the adsorption process was enhanced. This is substantiated by the ability of the student to answer the questions pertinent to the four thinking levels of the metaphor. Additionally, the enhancement was also seen in the writing and presentation skills of the student. Undoubtedly, embedding the Mahaffy metaphor into the capstone lab project requires greater faculty involvement, e.g. to contrive the questions that lead to the four thinking levels of the metaphor.

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