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ADSORPTION OF LEAD IONS (Pb^{2+}) FROM PECTIN-DERIVED ADSORBENTS: A SYSTEMATIC REVIEW AND META-ANALYSIS STUDY

Mark Jayson R. Digamon*, Aldous Matt Laurence M. Plata*, Engr. Ruth P. Barluado**, Dr. Jose Marie E. Ocdenaria**

Mark Jayson R. Digamon is currently pursuing Bachelor of Science in Chemical Engineering in Ateneo de Davao University, Philippines.
E-mail: marjrdigamon@addu.edu.ph
Aldous Matt Laurence M. Plata is currently pursuing Bachelor of Science in Chemical Engineering in Ateneo de Davao University, Philippines.
E-mail: amlmplata@addu.edu.ph
Engr. Ruth P. Barluado is currently a faculty member of the Chemical Engineering Department in Ateneo de Davao University, Philippines.
E-mail: rpbarluado@addu.edu.ph
Dr. Jose Marie E. Ocdenaria, LPT is currently a faculty member of the School of Arts and Sciences of the Ateneo de Davao University, Philippines.
E-mail: jmeocdenaria@addu.edu.ph

** Author*
*** Co-author*

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ABSTRACT
Water pollution is no longer new in this era as it deteriorates the quality of water resources through contamination which can cause various risks. To address the problem, a race to produce progressive solutions have been executed through different studies, and one of the promising methods proposed is using pectin and pectin-derived adsorbent to adsorb heavy metals. The purpose of this systematic review paper is to determine and analyze the effects of the different parameters affecting the adsorption capacity of pectin-derived adsorbents and determine which pectin-derived adsorbent had the highest adsorption capacity. Articles that mentioned adsorption of lead using pectin and pectin-derived adsorbent or any notations linked to the case were collected, and through meta-analysis, data from different literatures were integrated to determine the objectives of this systematic review paper. It was found that the parameters affecting the adsorption capacity of pectin-derived adsorbents were pH, contact time, initial concentration of lead solution, mass of the adsorbent, and temperature. Also, among the gathered literatures, Pectin Hydrogel Metal Organic Framework showed the highest adsorption capacity (913.88 mg/g). The results from different literatures showed a growing potential in the use of pectin and pectin-derived adsorbent in removing lead ions.

1. INTRODUCTION

In the present day, reports about pollution are no longer a surprise to the world. Pollution is rampant and despite the efforts done by countless organizations, people still fail to recognize that the problem is their lack of discipline and respect towards nature. Urbanization accelerated at the cost of the Earth's natural resources.

Water is an essential natural resource and vital for the survival of living beings. It is an important nutrient in our body as it aids our digestion, regulate body temperature, and helps to dispose waste in our body. The Earth is abundant with water which people, plants, and animals can use according to their needs. May it be for hygiene, for health, for recreation, or for commercial reasons, water is undoubtedly a necessary part of life. Living beings depend on water for their body to function and while inhabitants of the Earth were blessed to have it, humans have gone greedy and abused bodies of water.

Sources of water getting polluted with foreign substances like heavy metals that is caused by improper treatment of wastewater from industries that discharges directly to the bodies of water constitutes hazardous implication to health due to the presence of heavy metals contaminating the food cycle. While humans consume fishes that contains high level of protein around 17% - 20% with amino acid where heavy metals like lead can inhibit amino acid transport slowing down the ability to synthesize red blood cells of animals [1]. The presence of lead in food and water may cause poor health effects on humans when exposed, such as increase in blood pressure and result to the delay in physical and mental development of infants and children [2].

Having these kinds of problem, this research would perform a systematic review to synthesize and develop a literature from gathered references, literatures, and articles for aspiring research in characterizing different sources of pectin as a heavy metal adsorbent.

Pectin plays a significant role on the interaction mechanism of pectin molecules and heavy metals in which a binding region, upon contact, called the Egg-box is where heavy metals are being linked into the pectin chain service as binding sites, a mechanism that assumes several free carboxyl groups of galacturonic acid are present.

This review paper seeks to present the adsorption characteristics of pectin using meta-analysis by gathering data from different literature to establish a reference for future experimental studies and research that can lessen contaminated bodies of water with heavy metals causing natural resources and human lives at risk.

1.1 Lead

Lead is a natural element found, usually in small quantities, on the crust of the earth. It has been widely used for more than 50 centuries according to research by archaeologist. The researchers found traces of lead in the glazes of ceramics used during the prehistoric times. It was also found that the ancient Egyptian uses lead to darken their eyes to intimidate their enemies by looking stronger and more terrifying. In today's era, lead has been one of the most important heavy metal to be mined to be used in the industrializing sector [3].

Although lead is useful to different sectors, it can be harmful to humans, animals, and the environment if not properly disposed or used that is why there are standard concentrations that must be followed in an area especially in bodies of water.

In general, it is a well-known fact that majority of the global environmental pollution are brought about by man-made operations and harmful practices on the agricultural, domestic, industrial, and mining industry. But as a matter of fact, environmental pollution can also happen naturally. This occurs when: (1) metals are altered into hydroxide, oxide, and sulfide in which the process is called corrosion; (2) when pollution in the atmosphere is passed on to the earth's surface through deposition and vice versa through evaporation and; (3) accumulation of heavy metals as a result of degradation and siphoning [4].

At present, lead is being spread wide across the surface of the earth. This type of environmental pollution is predominantly caused by human activities such as mining and industrialization. Harmful practices such as blasting dynamite to dig up ores, burning of fossil fuels, manufacturing of lead-based paint for homes, and usage of lead-soldered water pipes can consequently leak lead particles into bodies of water. To some extent, the production of bullets, cosmetics, herbal/folk remedies, toys, and pottery can also be sources of lead [5].

Biological organisms are the most vulnerable when exposed to the consequences of natural alteration and man-made pollution. When an organism absorbs food or water that is contaminated with heavy metals (e.g., lead), the concentration of the toxic substance present in its body becomes higher. This eventually leads to an imbalance between the ingestion and digestion of an organism [6].

Similarly, untreated or improperly treated wastewater constitutes hazardous implications to health due to the presence of heavy metals contaminating the food cycle. Grave repercussions may include skin problems, impotence, cardiovascular diseases, cancer, and many others.

Moreover, contact to heavy metals, especially lead, may increase the risk of establishing an autoimmune disease. This refers to an abnormal response of the immune system wherein it is attacking healthy parts of the human body. Occurrence of this can eventually result to psoriasis, lupus, type 1 diabetes, arthritis, etc. [7]. Lead can also cause miscarriage to pregnant women, damaging of brain, kidney, and nervous system, seizure and even death [5].

Also, according to an environmental and health study in China by Zhang et al. in 2011, lead and cadmium, which are the main sources of pollution, are connected to the high concentration of lead in blood of the children, arthralgia (joint pains), osteomalacia (softening of bones), and lavish amount of cadmium present in urine [8].

1.2 Pectin

Pectin is an organic, innocuous, complex polysaccharide which originates copiously in cell walls of plants. It can be derived from different types of resources such as citrus peels and apple pomace. Innumerable research has exhibit that pectin and pectin derivatives have a high level of accord for metal ions which consequently makes it a propitious adsorbent [9].

Pectin contains three major building blocks. These are called Homogalacturonan (HG), Rhamnogalacturonan I (RG I) and Rhamnogalacturonan II (RG II). The nonmethoxylated galacturonic acid residues of HG, which are functional groups such as carboxylic acid, are known to have the capacity to ionize and enable the pectin to bind metal ions in aqueous solution; thus, making it essential for heavy metal binding activities.

However, it is important to note that the mixing of powdered pectin and water should be continuous because inconsistent or insufficient mixing would lead to the rapid hydration of pectin which results to build up of clumps. Once clumps are formed, it is no longer ideal to be used for the adsorption process as it will only constrain the pectin's access to the metal ions.

Pectin plays a significant role on the interaction mechanism of pectin molecules and heavy metals. Upon contact, a binding region called "egg box", as shown in Fig. 2.1 [10], is consequently formed. This is where heavy metals are being linked into the pectin chain service as binding sites – a mechanism that assumes several free carboxyl groups of galacturonic acid are present.

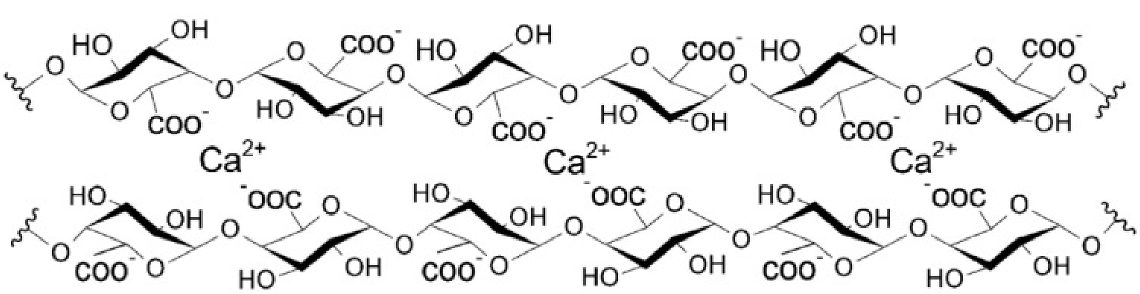

Figure 2.1. Egg Box Model, based on: [10]

To support this, many other studies have also proven that pectin can be an excellent bio sorbent as it exhibits a good capacity for adsorption of metal ions and through the facilitation of chemical modification and other treatments, pectin can be configured to perform with a higher level of efficiency [9].

1.3 Adsorption

Adsorption is operationally defined as an occurrence wherein a surface is subjected to component concentration. In any solid or liquid, atoms on the surface are exposed to unstable attraction forces which are normal to the surface plane. These are merely the amplifications of the forces that are acting within the material body and for the adsorption phenomenon's absolute responsibility [11].

The adsorption capacity can define as the quantity of the adsorbate adsorbed per weight of the adsorbent and it can be calculated using the formula below:

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

Equation 2.1 Adsorption Capacity

where q_e *(mg/g)* is the adsorbed quantity by the adsorbent, C_0 *(mg/L)* is the initial concentration of the solution, C_e *(mg/L)* is the concentration of the solution at equilibrium, *V (L)* is the volume of the solution, and *m (g)* is the dry mass of the adsorbent [12].

The percentage of a heavy metal that can be absorbed in a pectin from a solution can be presented as Ads_{HM} % and it can be calculated using the formula below:

$$Ads_{HM}\% = \frac{(C_0 - C_e)}{C_0}\ x\ 100$$

Equation 2.2 % Adsorption of Heavy Metal

1.4 Langmuir Adsorption Isotherm Model

Irving Langmuir presented his adsorption model in 1932 where it was applied to outline the gas monolayer adsorption on a solid plane [13]. The Langmuir adsorption isotherm model is an equation that is mainly acquired in perspective to kinetics and the monolayer assumption [14]. The following assumptions are: (1) adsorption occurs at specific binding sites that are localized on the surface of the adsorbent, (2) all adsorption sites on the surface of the adsorbent are identical, (3) the surface of the adsorbent is covered with a monolayer of adsorbed molecules and (4) there is no interaction between the adsorbed molecules on the adsorbent surface [15]. The Langmuir adsorption isotherm model equation is represented by Equation 2.1 [16].

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_m} + \frac{C_e}{Q_m}$$

Equation 2.3 Linear Equation of Langmuir Adsorption Isotherm

$$q_e = \frac{Q_m K_L\ C_e}{1 + K_L\ C_e}$$

Equation 2.4 Non – Linear Equation of Langmuir Adsorption Isotherm

q_e = the equilibrium concentration of the adsorbate in the adsorbent (mg/L)

K_L = Langmuir Constant (L/mg)

Q_m = Maximum adsorption capacity (mg/g)

C_e = the equilibrium concentration of the adsorbate solution (mg/L)

1.5 Freundlich Adsorption Isotherm Model

The Freundlich adsorption isotherm model describes the process (Ayawei et al. 2017) [17] that relates to multilayer and heterogeneous adsorption of adsorbate to the surface of the adsorbent (Kecili and Hussain 2018) [15] and it was predominantly developed to exhibit the variation of surfaces of the adsorbent and rapid allocation of active sites and energies (Chiou 2003) [18]. The Langmuir adsorption isotherm model equation is represented by Equation 2.2 [16].

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$

Equation 2.5 Linear Equation of Freundlich Adsorption Isotherm

$$q_e = K_F C_e^{\frac{1}{n}}$$

Equation 2.6 Non – Linear Equation of Freundlich Adsorption Isotherm

q_e = the equilibrium concentration of the adsorbate in the adsorbent (mg/L)

k_F = Freundlich constant (mg/g)

C_e = the equilibrium concentration of the adsorbate solution (mg/L)

$\frac{1}{n}$ = Adsorption intensity

2. METHODOLOGY

This chapter presents and discusses the research design and the methodology conducted in this review paper. Also, the technical design workflow is also presented in this chapter.

2.1 Research Design

The review paper utilizes a systematic review and a meta-analysis research design in which it was deemed the most appropriate in accomplishing this type of review paper. Systematic review includes the identification, evaluation, and the synthetization of all scientific and credible research related to this review paper. Meta-analysis in this review paper was used as a statistical tool to integrate all data and information gathered from the collected and examined research and experimental studies in which it summarizes and evaluates the different results from the systematic review in a justifiable and unprejudiced process [19].

2.2 Research Process

The review paper gathers literatures, experimental data, and articles related to adsorption of heavy metals using pectin in which keywords used in searching this information is tabulated in Table 2.1. Information that was gathered were from credible data-base and publishing websites where scientific research and data are bona fide. After gathering these literatures, experimental data, and articles, these were then segregated to be suitable and relevant for this review paper in which data segregation was based on the objectives that were laid out. Synthetization of data were executed and carried out after the gathering of data.

2.2.1 Collection of Articles

In the collection of articles, any items that mentioned adsorption of lead ions from pectin-derived adsorbents or any notations linked to the case were selected. All the gathered articles were not guaranteed to be part of the reference since an evaluation was done in the next process of this research.

Table 3.1

List of Keywords Used in Gathering Information

Base Material	**Process**	**Pollutant**	**Sample**	**Year**
Pectin	Adsorption	• Heavy Metal • Lead • Lead (II) Ions • Pb^{2+}	• Wastewater • Modified Wastewater	2011 – 2021

In this review paper, the included criteria are the experimental research and studies with data from the last ten years that utilized pectin-derived adsorbents for the adsorption of heavy metals specifically Pb^{2+} that contaminates wastewater. The excluded criteria are the articles that were irrelevant, without available full texts and incomplete data, abstract-only papers and experiments that did not use pectin as a source of adsorbent.

3.2.2 Segregation of Data and Selection of Themes

In determining the goal of this study, it was ideal for sorting out data according to its trends, themes, results, gaps, and possible studies. Table in Appendix A.1 showed the format in segregating the data and came up with the selection of themes.

3.2.2.1 Common Trends

The study of the subject became more complex; therefore, the factors in performing the research will change. The changes in the study might be the research's methodology or the equipment and raw materials of the experiments. This part will help locate the gaps of the study.

3.2.2.2 Common Themes

In research, it is expected to have limitations. These limitations that the author wish to exclude in their study can be the parameters or the variables. Some may vary in lacking the resources or instruments used to measure the other parameters. In every article all parameters were identified, collected, and written down by the authors to prioritize and assess its effects on the adsorption capacity.

3.2.2.3 Debates of Results

It is expected to have different results from one study to another. One study may have differences in terms of the outcome or impact and the methodology of the experiment or result of the experiment might be opposite. This area can help crucially in the objective part of this review.

3.2.2.4 Common Gaps

In getting the results above, it is obvious that there was a difference in every outcome, we can saw the gaps in those results by looking their difference in the theme. Significantly this part played importantly in the segregation process since the derived in its input to the concluding part of this review.

3.2.3 Analysis of the Articles Data

This section analyzed and explained the effects of pH, contact time, initial concentration of the adsorbate, and the mass of the adsorbents on the adsorption capacity of the pectin-derived adsorbents from the gathered literatures.

3.2.4 Synthesis

This section synthesized a detailed explanation of the effects of pH, contact time, initial concentration of the adsorbate, and the mass of the adsorbents on the adsorption process.

4. RESULT AND DISCUSSION

This chapter discusses the results of the gathered literatures, experimental data, and articles related to pectin-derived adsorbents. This chapter also analyzed and explained the effects of different parameters on the adsorption capacity of pectin-derived adsorbents

4.1 Analysis

This section analyzed and explained the effects of pH, contact time, initial concentration of the adsorbate, and the mass of the adsorbents to the adsorption capacity of the adsorbents from the gathered literature.

Table 4.3

Adsorption Capacity and its Adsorption Isotherm Model at Optimal pH and Contact Time

Pectin	pH	Contact Time	Mass of the Adsorbent	Adsorption Capacity	Adsorption Isotherm	Reference
Pectic Acid Microsphere	5	150 minutes	50 mg	325 mg/g	*did not indicate*	[20] (Li et al. 2018)
Pectin Microsphere				127 mg/g		
Pectin Hydrogel Metal Organic Framework	5	60 minutes	20 mg	913.88 mg/g	Langmuir Model	[21] Mahmoud and Mohamed 2020

HHP – assisted E Pectin	7	60 minutes	0.5 g	263.15 mg/g	Langmuir Model	[22] Arachchige et al. 2021
Sweet Potato Pectin				163.93 mg/g		
Commercial Citrus Pectin				142.85 mg/g		
Pectin/poly (m – phenylenediamine)$_{160\text{-}1}$	5	1440 minutes	30 mg	332.2 mg/g	Langmuir Model	[23] Wang et al. 2021
Pectin/poly (m – phenylenediamine)$_{80\text{-}1}$				350.9 mg/g		
Pectin/poly (m – phenylenediamine)$_{40\text{-}1}$				366.5 mg/g		
Pectin/poly (m – phnylenediamine)$_{20\text{-}1}$				390.9 mg/g		
Calcium Pectate: Low molecular pectate (Fraction A)	6	60 minutes	0.05 g	555.55 mg/g	Langmuir Model	[24] Khotimchenko et al. 2017
Calcium Pectate: Medium pectate (Fraction B)				526.32 mg/g		
Calcium Pectate: High molecular pectate (Fraction C)				476.19 mg/g		
Degraded pectin from Phyllospadix iwatensis	6	60 minutes	0.05 g	1.643 mmol/g (*340.43 mg/g)*	Langmuir Model	[25] Khozhaenko et al. 2016
Native pectin from Phyllospadix iwatensis				2.396 mmol/g *(496.451 mg/g)*		
Commercial citrus pectin				0.941 mmol/g *(194.975 mg/g)*		
Pec-g-poly (METAC-co-AMPS)-A5	7.1	120 minutes	20 mg	58.06 mg/g	Freundlich Model	[26] Zauro and Badalamoole 2018
Pec-gpoly (METAC co-AMPS)/MMT-C2				79.78 mg/g		
Pec-g-poly (AMPS-co-AAm)/Ag	7	120 minutes	20 mg	130 mg/g	Langmuir Model	[27] Kodoth and Badalamoole 2020

The adsorption capacity was acquired under its best conditions (pH and contact time) according to the adsorption studies during the experiments. The experimental studies by Wang et al. (2018), Mahmoud and Mohamed (2020), and Wang et al. (2021) occurred at pH 5, and for Khotimchenko et al. (2017) and Khozhaenko et al. (2016) occurred at pH 6 and the rest of the studies were around pH 7.

All of the adsorption studies had a duration of 60 minutes to 1440 minutes which assured that adsorption process reaches equilibrium.

Based on the results that was acquired, the adsorbent with the highest maximum adsorption capacity (913.88 mg/g) was the PHM composite by Mahmoud & Mohamed (2020) at pH 5 and a contact time of 60 minutes where the mass concentration of the adsorbent was at 20 mg. This is followed by fraction A and B of the low molecular pectate beads by Khotimchenko et al. (2017) with 555.55 mg/g and 526 mg/g where the conditions were at pH 6, contact time of 60 minutes, and the mass of the adsorbents was equal to 0.05 g.

Almost all of the study follows the Langmuir adsorption isotherm model in which it was assumed that adsorption occurs at specific binding sites that are localized on the surface of the adsorbent, all adsorption sites on the surface of the adsorbent were identical, the surface of the adsorbent was covered with a monolayer of adsorbed molecules and there was no interaction between the adsorbed molecules on the adsorbent surface. Only the study by Zauro and Vishalakshi (2018) showed that the adsorption process followed a Freundlich adsorption isotherm model in which it was related to a multilayer and heterogeneous adsorption of adsorbate to the surface of the adsorbent, and it was predominantly developed to exhibit the variation of surfaces of the adsorbent and rapid allocation of active sites and energies.

4.2 Synthesis

This section synthesized a detailed explanation of the effects of pH, contact time, initial concentration of the adsorbate, and the mass of the adsorbents on the adsorption process.

4.2.1 Effects of pH

The pH was used among these studies were ranging from 5 – 7 which resulted to a higher adsorption capacity.

At lower pH of the solution, the effect of the protonation of the functional groups, such as OH and –COOH, makes the surface of pectin-derived adsorbents positively charged, thus disrupting the adsorption of Pb^{2+} by electrostatic repulsion (Arachchige et al. 2021) [22], and this just shows the strong competitive behavior of H^{+} and Pb^{2+} at lower pH [23]. At higher pH of the solution, the degree of protonation decreases and the functional groups participating in the adsorption process increases [20]. The deprotonation of functional groups changes the zeta potential of pectin-derived adsorbents to negative in which the adsorbents could easily bind with the positively charged Pb^{2+}, thus increasing the adsorption capacity of the adsorbents [22].

In general, the increase of the pH of the solution also increases the adsorption of Pb^{2+}, but the increase in the adsorption varies for various types of adsorbents [28].

4.2.2 Effects of Contact Time

The contact time used among the studies ranges from 60 minutes onwards in which the adsorption process of pectin-derived adsorbents and the Pb^{2+} reaches equilibrium.

The increase in contact time between the pectin-derived adsorbents and Pb^{2+} increases the percentage removal of Pb^{2+} relatively, and upon reaching equilibrium stage of the adsorption process at *n* minutes depending on the various type of adsorbent, the percentage removal is at held constant. The adsorption process between the interaction of pectin-derived adsorbents and Pb^{2+} stabilized at equilibrium stage since all the binding sites in the surface of the adsorbent have been occupied [20].

In general, contact time between the pectin-derived adsorbents and Pb^{2+} is a significant factor and influences the adsorption process in which increasing the contact time between the adsorbent and Pb^{2+} increases the Pb^{2+} removal percentage and remain stable at equilibrium stage [21] [22].

4.3.3 Effects of Initial Concentration of Lead

The initial concentration of Pb^{2+} impacts the adsorption capacity of pectin-derived adsorbents towards Pb^{2+} because of the increase or decrease of the ratio of Pb^{2+} and the adsorbent. The increase of the adsorption capacity of the adsorbents is due to the fact that the active binding sites of the pectin-derived adsorbents interacts with the increase in concentration of Pb^{2+} in which it covers all binding sites of the surface of the adsorbent [23].

4.4.4 Effects of mass of the Adsorbent

The mass of the pectin-derived adsorbents can impact the efficiency of the removal of Pb^{2+}. Increasing the amount of the adsorbents also increases number of functional groups where it serves as the binding site between Pb^{2+} and the adsorbent [21]. The increase of interaction between the Pb^{2+} and binding sites of the adsorbent due to the increased mass of the adsorbents leads to an increase of amount of Pb^{2+} adsorbed [12] [22] [29].

4.4.5 Effects of Temperature

The effects of temperature can yield to higher adsorption capacity through acceleration of molecules via thermal motion in which interaction between Pb^{2+} and the adsorbents increases [22]. To iterate, increasing the temperature also increases the rate of diffusion of Pb^{2+} and also increase the pores of the binding sites of pectin-derived adsorbents [30].

However, decrease in the adsorption capacity of pectin-derived adsorbents can still occur at higher temperatures by damaging binding sites which can results to inefficiency of the adsorption process [31].

Conclusion

The objective of the review paper was to gather all the studies related to pectin-based adsorbents to treat wastewater that contains heavy metals specifically Pb2+, and identify, analyze, and synthesize the difference and similarities of the results of each study according to the presented data. Through systematic review and meta-analysis, the objectives of this review paper were attained. The involved articles, literatures, and review papers that were published were from websites that provided credible scientific research such as ScienceDirect, Research Gate, and Google Scholar.

The specific objectives of the study were accomplished in this review paper. (1) The parameters affecting adsorption capacity of pectin-derived adsorbents are pH, contact time, initial concentration of Pb2+, mass of the adsorbent, and temperature, and these are gathered from literatures about pectin-derived adsorbents to treat wastewater containing heavy metals specifically Pb2+. (2) The pectin-derived adsorbent with the highest adsorption capacity is the PHM composite by Mahmoud & Mohamed (2020) with an adsorption capacity of 913.88 mg/g. (3) Extensive set of data from 8 articles were analyzed and an analysis of the identified parameters (pH, contact time, initial concentration of lead, mass of the adsorbent, and temperature) affecting the adsorption capacity of pectin-derived adsorbents was generated.

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