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Archidendron jiringa seed peel extract in the removal of lead from synthetic residual water using coagulation-flocculation process

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ABSTRACT: *Archidendron jiringa* seed peel extract was used to aid the coagulation-flocculation process to ultimately remove lead from synthetic residual water. The effectiveness of this method was studied to obtain an alternative approach that is easy to be handled with low cost and energy in removing the lead from residual water. Optimum parameters were analyzed to determine the effectiveness of lead removal, including pH, alum dose, and *A. jiringa* seed peel extract dose. A study on the coagulation-flocculation process with and without the aid of *A. jiringa* was also conducted. The optimum pH, the alum dose, and the *A. jiringa* seed peel extract dose were 9.0, 2.44 g/l, and 60.2 mg/l, respectively. The percentage of lead removal with the aid of *A. jiringa* seed peel extract was 79%, and the percentage was dropped to only 47% without the extract. A significantly higher rate in the coagulation-flocculation process due to the presence of *A. jiringa* seed peel extract proved its effectiveness in removing lead from wastewater.

KEYWORDS: coagulation-flocculation, FTIR, *Archidendron jiringa*, lead, wastewater

INTRODUCTION

Industrial wastewater is a source of water pollution when it is not adequately treated and discharged at excessive rates directly to the environment [1]. Heavy metals can be detected in an environment, such as in the food chain. Therefore, the industrial water can pose a severe threat to human health. Lead is a heavy metal, soft, and malleable with a color of bluish-grey. It is of particular interest due to its toxicity and widespread presence in the environment. Lead can commonly be found in industrial wastewaters. The lead-ion concentrations in industrial wastewater are close to 200–500 mg/l, which is considered high compared with water quality standards [2]. The average lead concentration in domestic wastewater from various sources is 0.1 mg/l [3]. For storm-water wastewater, such as urban rainwater runoff, the annual concentration of heavy metals reported in the Poland/Częstochowa areas was within the range of 21.00–63.00 mg/kg [4].

Several methods for treating industrial wastewater include adsorption, precipitation [5, 6], coagulation [7], ion exchange, cementation, electrodialysis, electrowinning, electrocoagulation, and reverse osmosis [2]. However, the coagulation-flocculation process has been chosen as the primary alternative because it has more advantages than other methods [8], such as the low cost of a simpler water treatment process and the ability to reduce the repulsion force between colloidal particles for the formation of flocs, which can easily settle to the bottom. Another method that has been used is filtration. However, some suspended colloidal species resist to be settled because of their high stability in water, and some manage to pass through the filter due to their tiny size. As a result, the coagulation and flocculation process is considered the best alternative method to be used with the use of coagulants to induce charge neutralisation of colloidal particles and make the particles clump together and settle as flocs [9].

A. jiringa fruit can be found in local markets throughout the year [10]. It can ecologically be found in the primary and secondary rain forest, evergreen forest, and often saved when the forest is cut down. It is also cultivated in country-side villages. *A. jiringa* peel is considered an agricultural waste that can be found in large quantities in tropical nations. Local farmers use the peels and leaves as their primary sources for feeding farm animals; they are not utilised maximally [11]. These agricultural wastes can significantly act as an aid in heavy metal removal through flocculation-coagulation. Previous research has proven that coagulant aid materials usually increase the effectiveness of heavy metal removal from wastewater [12, 13]. The percentage of turbid removal using Fenugreek natural coagulant was 98%, which was more than the use of alum of 85% [14]. The application of a natural coagulant aid increased the effectiveness of arsenic removal from an aqueous solution [15]. The interaction of coagulants with a coagulant aid may result in elevated toxicity due to chemical reactions (especially those involving metal species). Thus, a biodegradable coagulant aid helps minimize the impact of toxicity to the environment especially water bodies where sludges are usually disposed [16]. In this study, *A. jiringa* seed peel was used as the coagulant aid to increase the effectiveness of lead removal from wastewater. The study aimed to investigate the removal of lead from synthetic wastewater using coagulation-flocculation process assisted by *A. jiringa* seed peel extract as a coagulant aid. The novelty of the research is that *A. jiringa* seed peel has not been used as a coagulant aid in the coagulation-flocculation process. Additionally, the optimum parameter test to identify the optimum dose of *A. jiringa* seed peel has been introduced in this study.

MATERIALS AND METHODS

Characterization of the chemical functional groups in *A. jiringa* extract

Fourier transform infrared (FTIR) spectrophotometer (Perkin Elmer, US) was used for the characterization and identification of chemical functional groups in the *A. jiringa* seed peel extract. The seed peel extract was treated with ethanol solvents, and the functional groups were observed in the infrared transmission region of 4000–650 cm^{-1}.

Coagulation-flocculation study

Lead (II) chloride, $PbCl_2$ (Merck, Germany), of more than 99% purity was used to prepare a 1 g/l lead stock solution. Synthetic wastewater containing 5 mg/l of lead was prepared by diluting the stock solution with deionized water. Industrial grade alum ($Al_2(SO_4)_3 \cdot 16\,H_2O$) was used to prepare a 1 kg/l coagulant stock solution. Then, the *A. jiringa* seed peel extract was prepared as a coagulant aid in this study. *A. jiringa* seed peels were cut into pieces of about 5 cm in diameter. The peel pieces were dried in an oven at 60 °C for 48 h and then crushed in a blending machine. *A. jiringa* seed peel extract was obtained by soaking the crushed peel in an alcohol solution for a few days, and the extract's concentration was determined.

Coagulation-flocculation experiments were performed using a standard jar test unit based on the procedures described in a previous study [17]. Firstly, for the optimum pH determination, lead solutions of pH 6–11 were prepared from the synthetic wastewater stock solution adjusted with sodium hydroxide and hydrochloric acid solutions. Secondly, in the optimum alum test, several doses of alum (i.e., aluminium sulphate) were prepared: 1.235, 1.478, 1.720, 1.961, 2.2, 2.439, 2.676, and 2.913 g/l. Six beakers were filled with 400 ml of similar initial concentration and pH lead solution. Then, 5 ml of *A. jiringa* seed peel extract was added into each beaker. The mixture was mixed at 80 rpm for 1 min, then at 20 rpm for another 30 min. Finally, the mixture was allowed for 30 min for the sediment to settle and then filtered; and the contents of particulate matters were analysed.

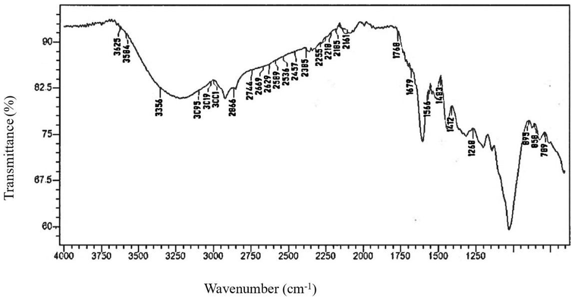

Fig. 1 FTIR transmission spectrum of *A. jiringa* seed peel extract in the frequency region of 650 cm^{-1}–4000 cm^{-1}.

Thirdly, different doses of *A. jiringa* seed peel extract: 15.1, 30.1, 45.1, 60.2, 75.2, and 90.1 mg/l were prepared in six separate beakers for the optimum test. An aliquot of 400 ml lead solution of similar initial concentration was added to each beaker. The mixtures were mixed at 80 rpm for 1 min and at 20 rpm for 30 min. The mixtures were allowed to settle for 30 min, while 50 ml of each mixture was filtered with a membrane filter and the filtrate obtained was analysed. The lead concentration was determined using Perkin-Elmer inductively coupled plasma-mass spectrometry (model ELAN 6000 ICP-MS) according to the Standard Methods for Examination of Water and Wastewater by the American Public Health Association [18].

Finally, the relationship between optimum parameters such as pH, alum dose and *A. jiringa* seed peel extract dose and the removal of lead using the coagulation-flocculation process with or without *A. jiringa* seed peel extract was analysed. The final test was conducted in 6 replicates under experimental conditions from previous optimum parameter tests, with optimum pH, alum dose, and *A. jiringa* seed peel extract dose of 9, 2.439 g/l, and 60.2 mg/l, respectively. The performance of coagulation-flocculation with or without *A. jiringa* seed peel extract was compared.

RESULTS AND DISCUSSION

Characterization of the phytochemical functional groups in *A. jiringa* extract

FTIR analysis had identified the presence of functional groups, such as phenols, amines, carbonyl, carboxylic, aliphatic compounds, alkene, and alcohol; in the *A. jiringa* seed peel extract extracted, and the transmission spectrum was shown in Fig. 1.

Table 1 FTIR functional groups and chemical bonds in *A. jiringa* seed peel extract.

Functional group	Wavenumber (cm^{-1})	Intensity
C—C vibrations	789, 858, 895	medium
O—H stretch/C—O stretch (carboxyl group)	1268	strong
=C—H stretch of aromatics	1483, 1412	medium
N—O/NO$_2$ asymmetric stretch of nitro compounds	1566	medium
C=C alkene	1679	weak
C=O stretch	1768	weak
Alkyne C≡C	2185, 2161	weak
Nitrile C≡N O—H stretch	2255, 2218	weak
Not identified	2457, 2385	–
Thiol group/S—H stretch	2669, 2629, 2589, 2536	weak
Alkyl C—H stretch	2866, 2744	weak
Alkene or aromatics	3019, 3001	weak
O—H stretch	3356, 3095	weak
N—H stretch	3625, 3584	weak

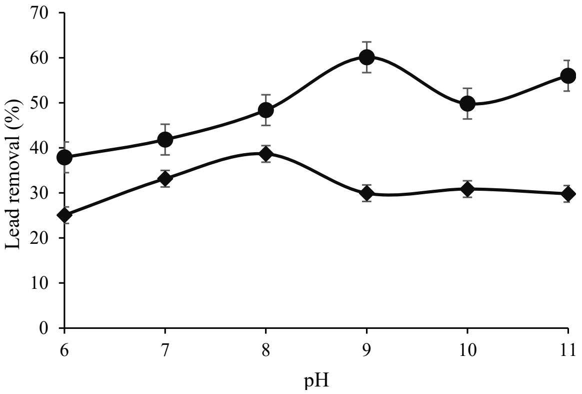

Fig. 2 Effects of pH on the percentage of lead removal by coagulation-flocculation process. ◆, lead removed without seed peel extract; ●, lead removed with seed peel extract.

The FTIR transmission spectrum showed weak bands at 3625 cm^{-1} and 3584 cm^{-1} from the N—H stretch, which indicated flavonoid compounds. The weak bands at 3356 cm^{-1} and 3095 cm^{-1} were from the O—H stretch, which indicated phenolic compound and saponins. The possible presence of alkene or aromatic could be indicated by the weak bands at 3019 cm^{-1} and 3001 cm^{-1}. Weak bands of Alkyl C–H stretch were observed at 2866 cm^{-1} and 2744 cm^{-1}. Thiol group or S—H stretch could be indicated by the 2669 cm^{-1}, 2629 cm^{-1}, 2589 cm^{-1}, and 2536 cm^{-1} bands, which might be originated from sulphur containing amino acid named Djenkolic acid commonly associated with *A. jiringa* seed flesh. Bands at 2457 cm^{-1} and 2385 cm^{-1} could not be identified. The weak bands at 2255 cm^{-1} and 2218 cm^{-1} could indicate the O—H group of carboxylic acid while those at 2185 cm^{-1} and 2161 cm^{-1} indicated alkyne C≡C. The weak bands at 1768 cm^{-1} and 1679 cm^{-1} could respectively indicate the carboxylic group and the C=C alkene (in saponin) or amide (in carbonyl compound), while the band at 1566 cm^{-1} was from N—O stretch. The medium bands at 1483 cm^{-1} and 1412 cm^{-1} showed the aromatic =C—H bond relating to the flavonoid. The medium band at 1268 cm^{-1} indicated the COOH group in flavonoid, while the bands at 789 cm^{-1}, 858 cm^{-1}, and 895 cm^{-1} indicated the C—C vibration (Table 1). The results of functional groups found in the *A. jiringa* seed peel extract were matched with the findings from previous studies on *A. jiringa* [19–21]. *A. jiringa* seed peels and leaves contain bioactive compounds such as saponin, total phenol, and flavonoid. Phenolic compounds are the main antioxidant part of *A. jiringa*'s seed shell which has powerful chain-breaking properties due to its ability to forage on the hydroxyl group and aid in the antioxidant activity [22]. A study done on *A. jiringa* shells reported the presence of proanthocyanidins from the polyphenolic compounds, specifically known as procyanidins B-3 and B-4 and prodelphinidin B-1 including flavan 3-ols [23].

Coagulation-flocculation study

The mean for the percentage of lead removal in the coagulation-flocculation process was determined. The *A. jiringa* seed peel extract aid achieved 79% lead removal, whereas 47% of lead removal was achieved in the absence of the seed peel extract. The percentage of removal might increase close to 100% by replacing alum with iron chloride or adding more alum dose. Three optimum parameters (i.e. pH, alum dose, and *A. jiringa* seed peel dose) were chose to determine the effectiveness of lead removal using the coagulation-flocculation process in this study [24]. These parameters affected the mechanism of coagulation-flocculation process [25, 26]. Furthermore, the mechanism also involved the neutralisation of negatively charged colloidal particles and the combination of foreign materials.

Fig. 2 shows that the optimum pH for the coagulation-flocculation process with and without *A. jiringa* seed peel extract were pH 9 and pH 8, respectively. The optimum pH assisted the neutralisation of negatively-charged colloidal particles and enhanced the floc formation between colloidal particles during coagulation [25]. pH was vital in removing heavy metals, and it involved the presence of in the coagulant's hydroxyl metal species [26].

Moreover, pH was crucial in coagulation-flocculation process because the process occurs in a certain pH range depending on the coagulant used [27]. Coagulants, such as aluminium salt, produce soluble hydroxyl metal species when added to a solution, resulting in different charged heavy metal species. depending on the pH of the solution. At low pH ($pH < 6$), the heavy metal species become positively charged and are negatively charged at high

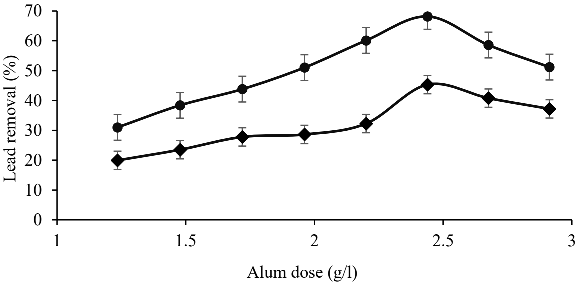

Fig. 3 Effects of alum dose on the percentage of lead removal by coagulation-flocculation process. ◆, lead removed without seed peel extract; ●, lead removed with seed peel extract.

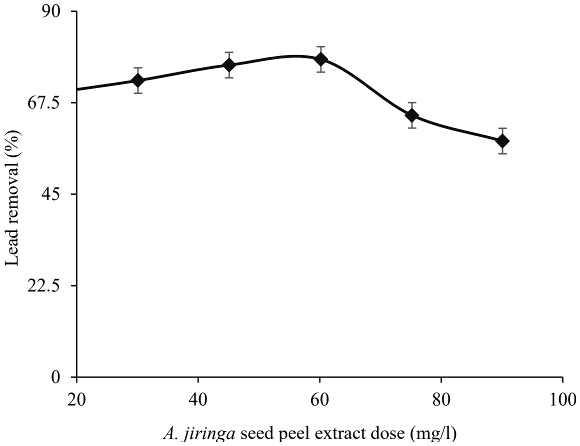

Fig. 4 Effects of *A. jiringa* seed peel extract dose on the percentage of lead removal by coagulation-flocculation process.

pH. In this study, the optimum pH values for removing lead were pH 8 and pH 9, indicating that the hydroxyl ions produced under alkaline conditions combined with heavy metal ions to form precipitates, and the heavy metals were removed [28]. The concentration of OH^- ion increased at a pH value above 9 (optimum pH), causing a decrease in the percentage of Pb(II) removal since $Pb(OH)_2$ were formed due to decreased electrostatic interaction [29].

An optimum dose is defined as the highest dose of coagulant that does not significantly affect the removal percentage of the process, even with more added dose [30]. Under the optimum pH 9, the results in this study showed that the optimum alum dose for the coagulation-flocculation process was 2.439 g/l with percentages of lead removal of 69% and 45% with and without the aid of *A. jiringa* seed peel extract, respectively (Fig. 3).

The presence of alum in the wastewater caused hydrolysis, and subsequently positively charged particles (such as Al_3^+ ions) were produced, reacted, and destabilized the pollutants (such as lead) present in the water; and they also neutralised the negative charges. Neutralisation of negative charges prevents the formation of electrostatic charges. The removal of lead occurred when negatively charged particles agglomerated and formed flocs. Higher coagulation efficiency could be obtained by adding sulphate materials to the solution. Addition of Al_3^+ ions to $Al(OH)_3$ could also accelerate precipitation. However, too much coagulant could destabilize the particles due to charge reversal [24, 31]. Hence, the amount of flocs removed would be reduced [32]. Aluminium salt is potentially toxic at a high dose. Therefore, it is essential to control the amount of dose to balance between the safety of a water treatment system and the effectiveness of heavy metal removal from wastewater.

The final studied parameter was *A. jiringa* seed peel extract dose as a coagulant aid. The coagulant aid was used when the addition of coagulant could not produce an adequate quantity of flocs. Thus, the use of coagulant aids was necessary to enhance the rate and the efficiency of floc formation, and subsequently increase the removal of pollutants from wastewater. With the optimum alum dose of 2.439 g/l and optimum pH 9, the coagulation-flocculation process with and without the aid of *A. jiringa* seed peel extract achieved 79% and 47% lead removal, respectively (Fig. 4). The result suggested that the introduction of *A. jiringa* seed peel extract into the solution produced large flocs which subsequently precipitated at the bottom. Hence, lead removal was quicker and more efficient than usual.

When alum was added to the solution, positively charged aluminium ions (Al_3^+) were formed, and the negatively charged particles were neutralised. Hence, electrostatic repulsion between these negative particles was removed; and, consequently, agglomeration occurred. The use of *A. jiringa* seed peel extract as a coagulant aid was vital to increase the rate of lead removal in this study. The mechanisms of lead removal in the coagulation process were adsorption and neutralisation. As identified by the FTIR analysis (Fig. 1), various functional groups were present in the *A. jiringa* seed peel, such as amino acids and Djenkolic acid. The amino acids derived from carboxyl group, ionized and produced carboxylate and H^+ ions. The carboxylate ions attracted positive ions from lead metal and colloidal particles in the medium. In the case of Djenkolic acid, negatively charged sulphur ions (sulfonate group) of *A. jiringa* seed peel (Table 1) played an important role in attracting positive ions from the lead metal. A high amount of phenol functional group in *A. jiringa* seed shells provided negative charges that caused destabilization and contributed to the charge neutralisation mechanism in the coagulation of metal colloid particles. As a result, these particles settled down as flocs. The utilisation of a coagulant aid in this study was necessary when the use of a coagulant did not reach a satisfactory level of lead removal.

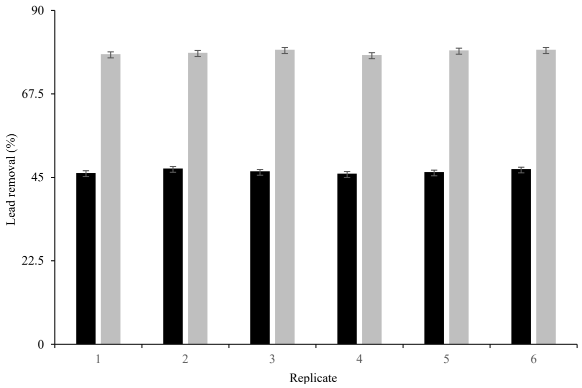

Fig. 5 Effects of *A. jiringa* seed peel extract on the percentage of lead removal by coagulation-flocculation process under optimum conditions: pH 9, alum dose 2.439 g/l and coagulant aid dose 60.2 mg/l. ■, without seed peel extract; ▨, with seed peel extract.

The coagulation-flocculation process without *A. jiringa* seed peel extract would still remove lead; however, the presence of *A. jiringa* seed peel extract notably increased the efficiency of lead removal.

By referring to the results in Fig. 5, the coagulation-flocculation process with the aid of *A. jiringa* seed peel extract achieved a higher mean percentage of lead removal (i.e., 79%) than that without the coagulant aid (47%). The huge difference of 32% of lead removal indicated that the removal of lead in wastewater was more effective with the aid of *A. jiringa* seed peel extract. Interestingly, it was reported in a previous study using different coagulants (e.g., ferric chloride, synthetic polymer polyethyleneimine, and biopolymers chitosan) that a 56% of lead removal was obtained with an optimized coagulant dosage of 0.10 mg/l. Hence, the result proved the effectiveness of *A. jiringa* seed peel extract in the removal of lead from synthetic wastewater [32].

Mechanism of *A. jiringa* seed peel as a natural coagulant

The coagulation capacity of *A. jiringa* seed peel as a natural coagulant originated from the functional groups and charges fixed in its structure. The importance of biomolecules in flocculation was discovered from the novel anionic bio flocculants coming from fermented fractions of sago mill effluent and palm oil mill effluent. As a result of charge neutralisation and intra-particle bridging, parts having xylose and glucose were observed to have a high flocculation performance [33]. The functional groups in the *A. jiringa* seed peel were capable of neutralising charges and bridging the contaminant particles together [34]. Lead pollutants are cationic by nature; thus, the anionic polyelectrolytes were used to remove the pollutants by electrostatic attraction between the two species, the cationic pollutant and the anionic polyelectrolyte [35].

In the present study, amino acids in the *A. jiringa* seed peel became ionized, and carboxylate and H^+ ions were produced. The carboxylate ions, then, attracted positive ions from lead metal and colloidal particles in the medium. A high amount of phenol functional group in *A. jiringa* seed shells provided negative charges that caused destabilisation and contributed to the charge neutralisation mechanism in the coagulation of metal-colloidal particles. As a result, the lead particles got close to each other forming flocs, settled down and removed [33]. The free Pb ions had a high affinity for the amino and phenol groups. Thus, complexation of Pb ions with these functional groups could also occur [32]. A report of a study revealed that metals were primarily bound to particulate and colloidal materials readily adsorbed to the surface of flocs resulting in the elimination of metal such as Pb [32]. The coagulation-flocculation process without *A. jiringa* seed peel extract could still remove lead; however, the presence of *A. jiringa* seed peel extract would notably increase the efficiency of lead removal.

CONCLUSION

The results revealed that at the optimum pH 9, 60% lead removal was achieved. The optimum alum dose was obtained at 2.439 g/l, which attained 69% lead removal. Meanwhile, the optimum *A. jiringa* seed peel extract dose was determined to be at 60.2 mg/l, with a percentage of lead removal of 79%. In addition, the coagulation-flocculation process using *A. jiringa* seed peel extract as a coagulant aid reached 79% lead removal, whereas only 47% lead removal was obtained in the absence of *A. jiringa* seed peel extract. Overall, a significant difference of 32% was achieved to prove the effectiveness of *A. jiringa* seed peel extract as a coagulant aid. For future research, it is suggested that different types of coagulant aid (such as iron (III) chloride) should be introduced instead of alum. Furthermore, the study can also vary the mixing time and speed to obtain a more effective coagulation-flocculation process in removing heavy metals from wastewater.

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