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ORIGINAL PAPER

Exploring the use of cactus *Opuntia ficus indica* in the biocoagulation–flocculation of Pb(II) ions from wastewaters

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Abstract Coagulation–flocculation processes play an important role in the treatment of water using inorganic and synthetic materials. Presently, cactus *Opuntia ficus indica* is explored as a cheap, abundant and eco-friendly bioflocculant of Pb(II) ions from aqueous solutions. The cactus powder was characterized by Fourier transform infrared spectrometer (FTIR), and the coagulation–flocculation process was optimized for pH, initial concentration of Pb(II) ions, particle size, dosage, ionic strength, contact time and temperature before its application to Mukuvisi River water. FTIR confirmed the involvement of –C–O–C–, –OH, –C=O, –N–H and –C–H groups of the polysaccharides, carboxylic acids and amino acids in the removal of Pb(II) ions. The variation of percentage removal and zeta potential with pH and flocculant dosage suggested the involvement of adsorption–charge neutralization and adsorption–bridging mechanisms in the coagulation–flocculation process. The process was optimum at pH 5, natural ionic strength, dosage of 8 mg/L, corresponding to zero zeta potential, with particle size <75 µm, at 35 °C for a floc settling time of 180 min. The coagulation–flocculation process was spontaneous and endothermic with a positive change in entropy of the system. Total heavy metal removal of 100 % (Pb), 85.74 % (Zn), 84.16 % (Cd) and 93.02 % (Cu) were achieved, under optimum conditions, when *Opuntia ficus indica* powder was used to remove metals from water sampled from Mukuvisi River, Harare, Zimbabwe. The cactus *Opuntia ficus indica* powder was found to be an efficient, eco-friendly flocculant that can effectively remediate Pb(II) ions from wastewaters.

Keywords Adsorption–bridging · Adsorption–charge neutralization · Ionic strength · Thermodynamics · Zeta potential

Introduction

The growing population, increased economic activity and industrialization have created an increased demand for potable water. Water resources all over the world are threatened not only by over exploitation and poor management but also by ecological degradation (Mahmood et al. 2010). The cost of water treatment is increasing, and the quality of rivers is deteriorating due to land development (Varsha and Jay 2012). Palatable clean drinking water is becoming scarce, especially in developing countries due to indiscriminate disposal of industrial and domestic wastes. One widespread phenomenon which has drawn much attention is the contamination of the aquatic environment by toxic metals such as copper, mercury, lead, zinc, nickel, cadmium, cobalt, iron, aluminum and chromium.

Lead is discharged into the environment from industrial processes such as storage battery manufacture, printing, pigment manufacturing, petrochemicals and photographic materials. Pb(II) is not biodegradable, and it bioaccumulates in humans via the food chain. Assimilation in the human body of relatively small amounts of lead over a long period of time can cause decreased muscle growth and kidney damage, and high levels of lead above the optimum level of 0.05 mg/L can cause fertility problems in both men and women (Okoye et al. 2010). Hameed (2006) reported

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that lead is toxic to growing brains and nervous systems of fetuses and young children and hence must be reduced in the aquatic systems.

Conventional methods for water purification such as reverse osmosis, ultrafiltration, chemical precipitation and ion exchange are being employed in some industries such as pharmaceuticals and mining industries (Varsha and Jay 2012), and synthetic polymers and inorganic salts such as iron (III) chloride and aluminum sulfate are currently used in water treatment all over the world. However, these treatment strategies generate toxic sludge that present disposal challenges since they tend to accumulate in the environment (Ganji et al. 2005). They are also costly for developing countries that still seek economic growth and have less efficiency and sensitive operating conditions (Abia et al. 2003). Research in the wastewater treatment strategies in recent years has been focusing attention on the use of biomaterials to combat a variety of pollutants including heavy metal ions, dyes, phosphates, nitrates, chlorides, phenolic compounds, pesticides, detergents and particulates among others. This led to the discovery of natural flocculants that purify water through coagulation–flocculation processes (Li et al. 2009). Armenante (2004) defines coagulation as the process through which very fine solid suspensions are destabilized so that they can begin to agglomerate if conditions allow while flocculation is defined as the process by which destabilized particles actually conglomerate into larger aggregates that can be separated from wastewater. The inorganic, organic and biomaterials that promote aggregation and sedimentation of suspended particles in solution are called coagulants and flocculants.

Natural flocculants are a viable alternative for developing countries. Some of the bioflocculants that have been tested include okra, gaur and mesquite seed gum (Carpinteyro-Urban and Torres 2013), bacteria isolates (Buthelezi et al. 2012), *Moringa oleifera* and *Opuntia* spp. (Miller et al. 2008), kenaf, *Conicia indica* (Varsha and Jay 2012), and *Plantago psyllium*. The advantages of natural flocculants over conventional coagulants–flocculants include their harmlessness, biodegradability, low commercial cost, relative abundance, renewability and high efficiency and therefore of smaller environmental impact compared to inorganic and synthetic polymers (Sharma et al. 2006; de Souza et al. 2014). It is desirable, therefore, to explore for these cost-effective and more environmentally acceptable bioflocculants to meet legislative requirements for a clean environment.

Cactus *Opuntia ficus indica* has long been associated with its medicinal properties and as a dietary food source and can be propagated in most parts of the world. The cactus mucilage and cladode are receiving much attention for their coagulation–flocculation and biosorption abilities (Mane et al. 2011; Bouatay and Mhenni 2014; Miller et al. 2008; de Souza et al. 2014) coupled to their ability to remove bacteria. The results obtained show that the flocculant has the potential to remove heavy metals, dyes and organic materials from aqueous environments. The high coagulation capability of *Opuntia ficus indica* is attributed to the presence of complex carbohydrates stored in cactus inner and outer pads that have good water retention capacity (Vijayaragharan et al. 2011). Recent research by Bustillos et al. (2013) shows that most of the parts whether fresh or dry have some coagulation activity due to the presence of viscous natural polyelectrolytes bearing negative surface charges under acidic conditions. The plant can therefore serve as a natural, cheap and renewable material for water purification. The present work therefore focused on exploring the potential of *Opuntia ficus indica* powder (OFIP) as a natural flocculant for Pb(II) ion removal from aqueous environment. The research study was carried out at (removed for the purpose of blinding the manuscript) in the Department of Chemical Technology laboratory during the period October 2012 to June 2013.

Materials and methods

Sampling and preparation of *Opuntia ficus indica* powder

Cactus *Opuntia ficus indica* was harvested near Nehosho residential area in Gweru, Zimbabwe. The pads that were handpicked from various cactus plants were placed in polyethylene plastic containers. The *Opuntia ficus indica* pads were washed repeatedly with tap water to remove dust and extraneous material and then rinsed with deionized water. The pads were cut into small pieces, sun-dried for 4 weeks followed by oven-drying at 60 °C for 24 h (El-Sayed et al. 2011; Shilpa et al. 2012). The pads were then ground to a fine powder using a Christison Particle Technology commercial blender (HGB2WTG4, USA). The powder was stored in an air tight polythene bag awaiting use without any chemical pre-treatment.

FTIR analysis of *Opuntia ficus indica* powder before and after sorption

A mass of dry OFIP was mixed with dry finely powdered potassium bromide in a ratio of 1:100. The mixture was spread uniformly in a suitable disk and subjected to a pressure of 800 MPa. The disk was placed on a sample holder, and the spectrum was measured in the range 400–4000 cm^{-1} using a Nicolet 6700 Thermo Scientific Fourier transform infrared spectrometer.

Investigating factors that affect flocculation

Several factors can affect the coagulation–flocculation process: pH, contact time, temperature, flocculant dosage, ionic strength and concentration of the target pollutant. These factors individually and collectively have a great influence on the flocculant's optimum performance. The factors were investigated keeping the synthetic effluent volume constant at 100 in 250-mL Erlenmeyer flasks.

Effect of pH on the flocculation of Pb(II) ions from aqueous solution

The effect of pH on the flocculation of Pb(II) ions was investigated using 1 g of cactus powder in 100-mL portions of 6 mg/L of Pb(II) ions in triplicates. Each mixture was adjusted to pH ranging from 2 to 8 using 0.1 M HCl and 0.1 M NaOH solutions and a Thermo Scientific pH meter (Orion Star A211, Singapore). The mixtures were rapidly stirred for 2 min followed by gentle stirring for 3 min using a Labcon magnetic stirrer (MSH 10) before they were allowed to settle for 24 h. This procedure was repeated using different initial concentrations of Pb(II) ions ranging from 10 to 26 mg/L. After allowing the mixtures to stand for 24 h, they were vacuum-filtered and both the initial and residual metal ion concentrations were determined using a Shimadzu AA 6800 flame atomic absorption spectrometer (FAAS).

Effect of Pb(II) ion initial concentration and flocculant particle size

To evaluate the effect of initial Pb(II) ion concentration on the flocculation process, Pb(II) ion solutions of different concentrations in the range 5–40 mg/L at pH 5 were prepared. The 100-mL portions of each solution were transferred into seven Erlenmeyer flasks in triplicates, and 1 g of the OFIP of particle size less than 75 μm was added to each of the flasks. The procedure was repeated using OFIP of different particle sizes that were segregated as follows. The ground OFIP was first sieved through a 75-μm stainless steel mess (field master). The particles that passed through the mesh were designated <75 μm (<75 μm). The residue was then sieved using a 180-μm mess, and the product was labeled 75 ↔ 180 μm. The same procedure was followed using 250- and 350-μm stainless steel messes. The mixtures were rapidly stirred for 2 min followed by gentle stirring for 3 min using a Labcon magnetic stirrer (MSH 10) and were allowed to stand for 24 h before decanting and analysis by FAAS (Karthika et al. 2010; Mataka et al. 2010).

Effect of OFIP dosage and ionic strength on flocculation of Pb(II) ions

The influence of flocculant dosage on Pb(II) ion removal was investigated by transferring 100-mL portions of the synthetic water effluent containing 20.6528 mg/L of Pb(II) ion at optimum pH of 5 into Erlenmeyer flasks. Cactus powder (75 μm) of mass ranging from 0.1 to 2.0 g was added to the flasks in triplicates, and the mixtures were rapidly stirred for 2 min followed by gentle stirring for 3 min. The solutions were maintained at 30 °C for 24 h. The procedure was repeated with varying amount of NaCl added to the synthetic water effluent to give final NaCl concentrations ranging from 0.2 to 0.8 M (different ionic strengths). The solutions were filtered, and both the initial and residual metal ion concentrations were determined.

Effect of contact time and temperature on the flocculation of Pb(II) ions

Synthetic water effluent containing 23.0068 mg/L of Pb(II) at optimum pH 5 was transferred in 100-mL portions into eight Erlenmeyer flasks in triplicates. Cactus powder in approximately 0.8 g (75 μm) of portions was added to the flasks, and the mixtures were rapidly stirred for 2 min followed by gentle stirring for 3 min using a magnetic stirrer. The Erlenmeyer flasks were covered and allowed to stand for a period ranging from 30 to 360 min at 20 °C. The procedure was repeated at different temperatures ranging from 25 to 45 °C. The solutions were filtered, and the initial and residual Pb(II) ion concentrations were determined using FAAS.

Effect of pH and OFIP dosage on zeta potential of the system

The effect of pH on the zeta potential of the coagulation–flocculation system was explored using 1 g of cactus powder in 100-mL portions of 20 mg/L of Pb(II) ions in triplicates. Each mixture was adjusted to pH ranging from 2 to 8 using 0.1 M HCl and 0.1 M NaOH solutions. Cactus powder (75 μm) of mass ranging from 0.1 to 2.0 g was added to the flasks containing 100-mL portions of 20.2242 mg/L of Pb(II) ion at optimum pH of 5. The suspensions were stirred for 10 min on a magnetic stirrer and centrifuged before 2 mL of solution was transferred to a closed measurement cell for zeta potential (ζ) measurement using a Zetasizer Nano-ZS (ZEN3500) meter (Kurama and Karaguzel 2013).

Description of the sampling area for the river water sample

River water samples were collected at a small pool close to a bridge near Mbudzi traffic cycle, in Mukuvisi River—a tributary to Manyame River that feeds into Lake Chivero. Lake Chivero supplies both domestic and industrial water to the capital city of Zimbabwe, Harare. The industrial wastewaters from Harare's heavy industrial areas (Msasa, Mbare, Graniteside, Highfield and Waterfalls) are deposited into this river. Occasional bursting of the sewage pipes and indiscriminate deposition of domestic wastes also lead to increase in pollutant levels in this river. The river is well known for its high pollution levels that have resulted in the death of some aquatic life including fish (Masere et al. 2012). Due to incessant water cuts in the city, most urban dwellers resort to the use of Mukuvisi River water for various purposes, leading to inevitable human exposure to the pollutants.

Collection of the river water samples for total heavy metal analysis

Grab samples were collected along Mukuvisi River using three 500-mL Teflon plastic containers at each point. The containers were pre-rinsed three times with the sample water. The containers were filled to the brim, and a few drops of 10 % HNO_3 were added until the pH of the sample was 1.6 (<2 mL was added). The samples were refrigerated at a temperature below 4 °C awaiting analysis by FAAS. The samples, 100 mL, were transferred each into a 250-mL beaker, and 5 mL of 2 M HNO_3 was added. The beakers were placed over hot plates, and the contents were evaporated to 20 mL. The beakers were allowed to cool, and 5 mL of 2 M HNO_3 was further added. The beakers were covered with watch glasses and were heated again. Portions of 2 M HNO_3 were added to the solution until the solution turned clear (Akan et al. 2008). The digested solutions, together with the beaker walls and watch glass washings, were filtered, and the volume of the solution was adjusted to 100 mL with distilled water prior to analysis of Pb, Zn, Cd and Cu by FAAS.

Coagulation–flocculation of Mukuvisi River water

The pH-adjusted river water samples in 100-mL portions were transferred into 250-mL Erlenmeyer flasks in triplicate. Optimum dosage of 0.8 g (75 µm) of OFIP was added to each of the flasks. The coagulation–flocculation was performed under optimized conditions. The filtrate was digested and analyzed for Pb, Zn, Cd and Cu.

Results and discussion

Characterization of *Opuntia ficus indica* by FTIR

The Fourier transform infrared (FTIR) spectra of OFIP before and after flocculation of Pb(II) ions were used to determine the vibrational frequency changes of the surface functional groups on the flocculant. This helped to identify the functional groups that were active in the coagulation–flocculation processes. The analysis was carried out using the KBr disk, and the spectrum was collected from 4000 to 400 cm^{-1}. Table 1 shows the band assignments associated with OFIP spectrum before and after flocculation of Pb(II) ions.

The shift in the absorption peaks signified a change in the chemical environment of the functional groups and indicated the existence of a metal-binding process taking place between the flocculant and the Pb(II) ions (Awwad and Salem 2014). The nonsuperimposable nature of the two spectra, as a result of shifting to higher frequency of all the absorption peaks and the disappearance of peaks at 1078.04 and 2920.37 cm^{-1} on OFIP after the flocculation process, signified the involvement of carboxylic acids, amino acids and polysaccharides in the removal of Pb(II) ions from aqueous solution. The result is in line with findings of de Souza et al. (2014) who established the chemical composition of *Opuntia ficus indica* to be: 3.52 ± 0.21 % crude protein and 0.28 ± 0.015 % lipids among carbohydrates, L-arabinose, D-galactose, L-rhamnose, D-xylose and galacturonic acid, with the later existing in its polymerized form, polygalacturonic acid, and is reported as the active ingredient that affords the coagulation capability of *Opuntia ficus indica* mucilage (Jadhav and Mahajan 2014).

Optimization of factors that affect the coagulation–flocculation process

To optimize the flocculation process, the effect of pH, initial Pb(II) ion concentration, flocculant particle size,

Table 1 FTIR structural elucidation of *OFIP* before and after flocculation of Pb(II) ions

Before flocculation	After flocculation	Inference	Compound responsible	References
1078.04	–	–C–O–C and –OH	Polysaccharides	Bouatay and Mhenni (2014)
1438.06	1458.60	O–H and C=O	Phenols and carboxylates	Bouatay and Mhenni (2014)
1618.26–1654.80	1654.93	C=O stretch, C=C ring	Carboxylic acid, alkenes, amide, amine	Singha et al. (2011)
2920.37	–	C–H stretch, O–H stretch	Alkanes, carboxylic acid	Sari and Tuzen (2009)
3422.54–3691.34	3448.50–3855.11	Bounded –OH –NH	Carboxylic acid, amino acids	Sari and Tuzen (2009)

contact time, temperature, dosage and ionic strength was investigated. The parameter value that gave the highest magnitude of percentage removal was identified as the optimum. The percentage removal of Pb(II) ions by the OFIP was calculated using Eq. (1):

$$\%\ \text{Removal} = \frac{(C_{\text{o}} - C_{\text{F}})}{C_{\text{o}}}\frac{\delta y}{\delta x} \times 100\% \qquad (1)$$

where C_{o} is the initial metal ion concentration in solution and C_{F} is the metal ion concentration (mg/L) in solution after coagulation–flocculation.

Effect of pH and initial concentration

The effect of pH was monitored in the range 2–8, keeping all other factors constant, that is: temperature of 30 °C, OFIP dosage of 10 g/L, 24-h contact time and Pb(II) ion initial concentration of 6 mg/L. The investigation of pH was repeated with 10, 15, 20 and 26 mg/L of Pb(II) ion concentrations, and the results are presented in Fig. 1.

Figure 1 shows that the percentage removal of Pb(II) ions depends greatly on pH. The percentage removal increased gradually with increase in pH up to pH 5 after which it decreased to pH 8 at all initial concentrations of Pb(II) ions investigated. pH > 8 was avoided in this investigation due to precipitation of $Pb(OH)_2$ that would be mistaken as coagulation–flocculation due to OFIP.

In acidic environments, there is competition between hydrogen ions and the metal ions for the negatively charged sites on the anionic flocculant (El-Sayed et al. 2011) of which the H^+ ions win. The protonated flocculant repelled the positive Pb(II) ions; hence, minimum percentage removal was observed. The diminishing levels of H^+ ions in solution as pH was increased gave rise to Pb(II) ions being the dominant cationic species in solution; hence, its percentage removal increased with pH. Vijayaragharan et al. (2011) cited the high electrostatic attraction between the positive metal ions and the negatively charged sites of the flocculant, resulting in an enhancement of metal flocculation as pH is increased. Similar trend of results was obtained by Mataka et al. (2006) in the remediation of lead (II) ions from wastewaters using *Moringa stenopetala* and *oleifera*. The decrease in the percentage removal after pH 5 might also have been caused by the limited ionization of the –COOH groups on the OFIP that limits the bridging actions responsible for the efficient growth of flocs (Mounir et al. 2014).

Mane et al. (2011) indicated that *Opuntia ficus indica* comprises of anionic polyelectrolytes on which positive metal ions serve to form bridges that result in particle agglomeration and eventual coagulation and flocculation. pH affects the degree of ionization and consequently the electric potential of the particles. At some pH, colloidal particles possess the zero point of charge (ZPC; where zeta-potential approaches zero), which implies no net charge. Figure 2 shows the variation of zeta potential with pH in a system comprising 10 mg/L of OFIP dosage in Pb(II) ion solution.

The zeta potential was always negative across all pH values investigated. However, it was close to zero (minimum) at pH 5–6 where maximum removal of Pb(II) ions was achieved. At this pH, there was efficient aggregation of the colloid and flocculation was high (Lee 2014). Variation of pH above or below the pH of zero-point charge resulted in surface charges that stabilized the colloid, and hence minimum flocculation occurred. For the Pb(II) ions and OFIP system, this pH was observed to be five and was maintained constant in further investigations.

The percentage removal of Pb(II) ions decreased with increase in initial concentration at all the pH values investigated (Fig. 1). This was attributed to the exhaustion of the flocculant that it became the limiting factor in the flocculation process.

Effect of Pb(II) initial concentration and OFIP particle size

The effect of Pb(II) ion concentration on the flocculation behavior of the OFIP was studied at pH 5. Figure 3 shows

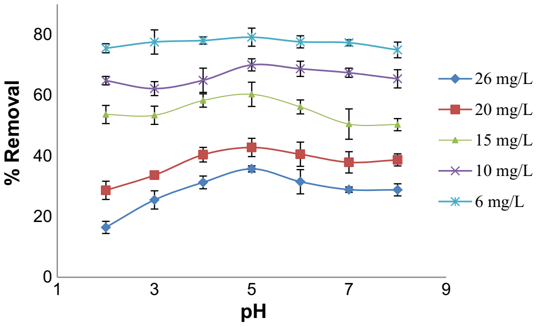

Fig. 1 Effect of pH and initial concentration of Pb(II) ions on the flocculation of Pb(II) ions by OFIP

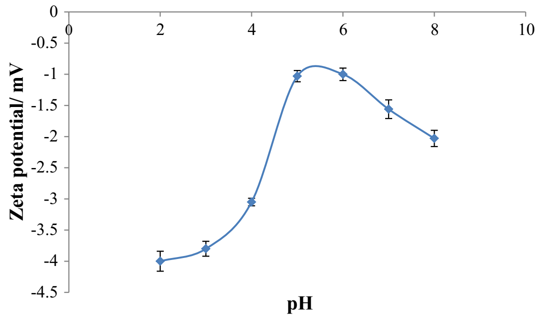

Fig. 2 Effect of pH on zeta potential at constant OFIP dosage

the relationship between initial Pb(II) ion concentration, particle size and the percentage removal of Pb(II) ion at fixed dosage of 10 g/L at constant temperature (30 °C) and contact time of 24 h.

The trend shows a decrease in percentage removal of Pb(II) ions with increase in both initial concentration of Pb(II) ions and OFIP particle size. At low concentrations, most of the metal ions present in the solution would interact with the active sites of the flocculant, resulting in high percentage removal of Pb(II) ions. At higher concentration of Pb(II) ions, some remained in solution due to the saturation of the negatively charged sites, hence causing electrostatic repulsions between the flocculated Pb(II) ions and those in solution. This conclusion agrees with that reported by Mataka et al. (2010) during the flocculation of Cd (II) ions by *Moringa oleifera*.

The increase in percentage removal with decrease in OFIP particle size was due to the greater surface area exposed to the Pb(II) ions by the flocculant that resulted in the efficient scavenging of the metal ions by the flocculant. Cactus *Opuntia ficus indica* mucilage contains carbohydrates, L-arabinose, D-galactose, L-rhamnose, D-xylose and galacturonic acid, with the polymerized form of galacturonic acid (polygalacturonic acid), being largely responsible for the coagulation activity of the plant (Jadhav and Mahajan 2014; Miller et al. 2008). The high surface area of the flocculant implied the greater expose of polygalacturonic acid in the solution that is eventually adsorbed to colloidal particles bridging them up forming large flocs. The low anionic charge of the organic molecule (polygalacturonic acid) and its size decrease the potential around particles (through adsorption), thereby cascading the coagulation–flocculation of the pollutant (Armenante 2004). The OFIP size <75 µm emerged the optimum particle size for subsequent investigations.

Effect of OFIP dosage and ionic strength on the flocculation of Pb(II) ions

The experiment to determine the optimum dosage and ionic strength on Pb(II) removal was carried out at optimum pH of 5, contact time of 24 h, at a temperature of 30 °C and OFIP of particle size <75 µm. The OFIP dosage was varied in the range from 1 to 20 g/L. Sodium chloride was used to investigate the effect of ionic strength. Its concentration was varied from 0.2 to 0.8 M. The relationship between the percentage removal of Pb(II) with flocculant dosage and concentration of NaCl is shown in Fig. 4.

The graph shows that the percentage removal of Pb(II) ions increased with increase in OFIP dosages up to the optimum dosage of 8 g/L after which its gradual decrease was observed. The increase in dosage resulted in increase in negatively charged sites available for metal ion aggregation. However, after the optimum dosage, further increase in dosage caused the aggregated particles to re-spread and would also disturb particle settling (Bouatay and Mhenni 2014). These conclusions agree with that reported by Mishra and Malvika (2005) on the flocculation of model textile wastewater with a food-grade polysaccharide. Above the optimum dosage of 8 g/L, the trends showed a decrease in percentage removal attributed to particle stabilization due to some steric repulsion (Tripathy and De 2006). Aygun and Yilmaz (2010) presented that the flocculant forms an envelope on the suspended particles and causes them to remain in suspension; thus, removal efficiency decreases with increasing dosage beyond the optimum.

The zeta potential of the coagulation system was explored at increasing OFIP dosages in the range 1–20 mg/L in approximately 20 mg/L of Pb(II) ions at pH 5.

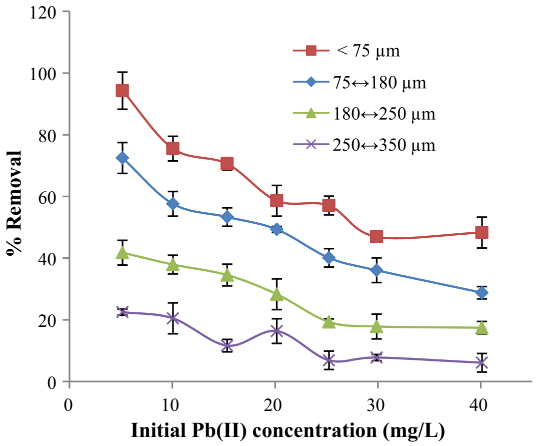

Fig. 3 Effect of Pb(II) initial concentration and OFIP particle size on the flocculation of Pb(II) ions by OFIP

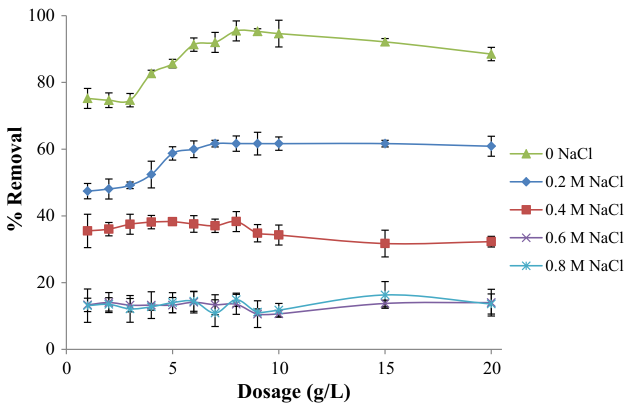

Fig. 4 Effect of OFIP dosage and ionic strength on the flocculation of Pb(II) ions from wastewaters

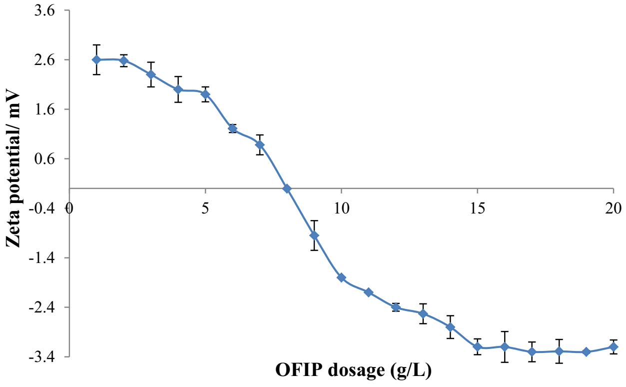

Fig. 5 Effect of OFIP dosage on the zeta potential at constant pH

Figure 5 shows that the zeta potential decreased with increase in OFIP dosage up to an OFIP dosage of 15 mg/L where it became constant at approximately −3.2 mV. The point of zero charge occurred at a dosage of around 8 mg/L (−0.005 mV) where the graph cuts the *x*-axes. The decrease in zeta potential revealed the anionic nature of the flocculant. The trend is opposite to that obtained when *Moringa oleifera* and chitosan were used due to their cationic nature (Renault et al. 2009; Miller et al. 2008). The *Opuntia Ficus indica* dosage of 8 g/L brought about complete charge neutralization of the system and was therefore identified as the optimum dosage for the removal of Pb(II) ions from wastewaters.

Different concentrations of NaCl were used to vary the ionic strength of the Pb(II) synthetic effluent. Figure 4 shows that the flocculation of Pb(II) ions decreased significantly with increase in concentration of NaCl in solution. This effect of the ionic strength implied the noninvolvement of double-layer compression mechanism in the removal of Pb(II) ions by OFIP (Miller et al. 2008). Taher et al. (2012) highlighted that the electrostatic interactions between the metal ions and the flocculant's negative sites can be reduced by increasing ionic strength of the solution due to screening effect of the surface charge produced by the added salt. Earlier investigations ruled out the existence of ions in Opuntia spp. that would affect the ionic strength of its solutions (Fedala et al. 2015). Decreasing the ionic strength expands the flocculant in solution due to mutual charge repulsion and enhances bridging by increasing the effective particle radius; hence, more pollutant is removed (Tripathy and De 2006). The flocculation of Pb(II) ions from wastewaters was efficient in low ionic strength solutions with no NaCl ions. The Pb(II) ion percentage removal was always greater than 70 % at all OFIP dosages investigated in the absence of NaCl. The subsequent investigations were therefore done in the absence of NaCl.

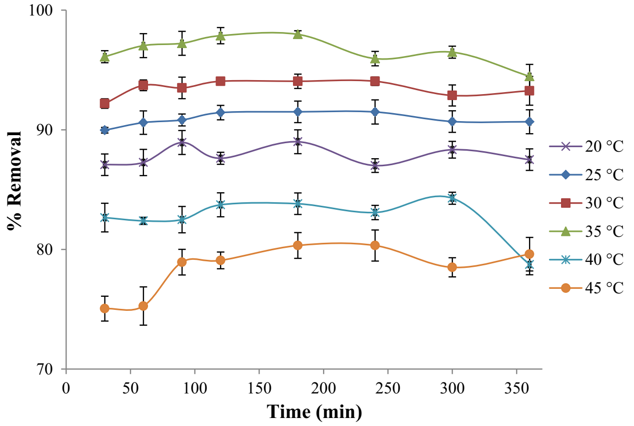

Fig. 6 Effect of contact time and temperature on the flocculation of Pb(II) ions by OFIP

Effect of contact time and temperature

The effect of contact time on the coagulation–flocculation of Pb(II) ions was investigated using 8 g/L of OFIP of particle size <75 µm at optimum pH 5. The investigation was repeated at different temperatures ranging from 20 to 45 °C. The relationship between the percentage removal, contact time and temperature is shown in Fig. 6.

As shown in Fig. 6, the rate of Pb(II) removal increased from 30 to 180 min, and thereafter, the percentage removal remained constant or decreased slightly at all temperatures. Between 30 and 180 min, most of the metal ions present in solution would interact with the negatively charged sites of the flocculant, facilitating high percentage removal. Progressive occupation of these negatively charged sites after 180 min, at fixed OFIP dosage, resulted in total available binding sites being limited; hence, the coagulation–flocculation process attained equilibrium (Karthika et al. 2010). A similar trend was observed for the removal of dyes from model textile wastewater using *Plantago psyllium* mucilage (Mishra and Malvika 2005).

Figure 6 shows that as temperature increased, the percentage removal of Pb(II) also increased and peaked at 35 °C, producing a characteristic curve for the effect of temperature on enzyme activity. The flocculation of particles in a liquid depends on collision among particles, caused by their relative motion (Moss and Dymond 2015). The slight increase in percentage removal of Pb(II) ions between 20 and 35 °C was due to the increase in the diffusion rate of the flocculant throughout the solution. Vik et al. (1989) in Duan and Gregory (2003) present that as the viscosity of a fluid increases with decreasing temperature, the poor rapid-mixing conditions caused by low water temperature might lead to inhomogeneous distribution of polyelectrolytes in the water which results in poor coagulation. Temperatures greater than the optimum result in the rapid movement of particles that disturbs their agglomeration; hence, minimum coagulation–flocculation occurs. The adsorption step in coagulation systems is exothermic and therefore is unfavorably affected by higher temperatures (Moss and Dymond 2015). Maximum percentage removal was observed at 35 °C and was chosen as the optimum temperature for the flocculation of Mukuvisi River water.

Coagulation–flocculation mechanisms applied to the OFIP–Pb(II) ion system

Coagulation–flocculation can be brought about by one and/or any of the following mechanisms: double-layer compression (salting out), adsorption and charge neutralization, adsorption inter-particle bridging, enmeshment in a precipitate (sweep flocculation) and electrostatic patch mechanism (Duan and Gregory 2003; Renault et al. 2009). These mechanisms can occur simultaneously or sequentially in one coagulation–flocculation system. Double-layer compression is the destabilization of a colloid that involves reduction in the double layer around the colloidal particle as a result of change in ionic strength that is brought about by the addition of an indifferent electrolyte, e.g., NaCl (Chukwudi and Uche 2008). The reduction in the double layer brings colloidal particles close together for the attractive van der Waals forces to dominate over the electrostatic repulsions; hence, coagulation occurs. With this mechanism, there is no overdosing of the coagulant, and the same amount of electrolyte is required for both high and low colloidal concentrations.

Adsorption and charge neutralization are brought about, in finely divided suspensions, by chemical species that are capable of being adsorbed onto the surface of the colloidal particles. Opposite charges (to those on particles) on these chemical species reduce the surface potential of the particles and eventually lead to colloidal particle destabilization (Chukwudi and Uche 2008). It is observed that:

- The sorbable species destabilize colloids at lower dosages than nonsorbable–indifferent ions.
- As the concentration of colloids increases, the dosage of the coagulant increases (stoichiometric relationship).
- Re-stabilization of the colloidal particles occurs by overdosing the coagulant due to charge reversal.
- It can be enhanced by addition of a bridging flocculant.
- Flocs can be broken down under high shear conditions.

These are significant for differentiating the adsorption and charge neutralization mechanism from the other coagulation–flocculation mechanisms.

When long-chain polymers (polyelectrolytes) are added in small dosages to colloidal particles, an individual chain can become attached to two or more particles, thus bridging them together. The stages involved in this adsorption and bridging mechanism include: dispersion of the polymer in the suspension, adsorption at the solid–liquid interface followed by settling down and finally colliding of adjacent polymer-coated particles to form bridges resulting in larger flocs (Bratby 1980). The dispersion of the polymer should not be violent nor be too long to prevent desorption and/or rearrangement of looped chains on the surface of the colloid. An optimum polymer dosage is identified before which the polymer is insufficient to form enough bridging links among particles and beyond which the polymer saturates the colloidal surfaces leaving no sites for the formation of polymer bridges (Tripathy and De 2006).

In coagulation–flocculation systems where strong electrostatic attraction between polymers (polyelectrolytes) and colloidal surfaces exists, the electrostatic patch mechanism applies as opposed to the polymer bridging mechanism. According to Sharma et al. (2006), the mechanism involves uneven distribution of charges resulting from the adsorption of discrete patches of polymer on the surface. Tripathy and De (2006) report that the mechanism predominates in systems characterized by high cationic charge on the flocculant and anionic colloidal solids at low particle concentrations. The polymer is completely adsorbed, and its chains form a charge mosaic (patch) with alternating regions of positive and negative charges. The charge mosaics of adjacent particles can align to give strong attractive electrostatic interactions, resulting in colloidal destabilization (Renault et al. 2009).

Enmeshment in a precipitate (sweep flocculation) involves the addition of relatively large dosages (to exceed the solubility product of the metal hydroxide, oxide or carbonate) of coagulants [$Al_2(SO_4)_3$, $FeCl_3$] that precipitate as hydrous metal oxides or carbonates. Colloidal particles are then enmeshed in the precipitate and settled down. With this mechanism, there is inverse relationship between optimum coagulant dosage and colloid concentration (Rattanakawin 2005). At low colloidal concentrations, large excess of coagulant is required for production of excess precipitate to enmesh the particles. At high colloidal concentrations, a lower coagulant dosage is required because the colloids provide nuclei to enhance precipitate formation.

A close examination of the experimental results versus the various coagulation–flocculation mechanisms presented above helps identify the mechanism/s involved in the removal of Pb(II) ions by *Opuntia ficus indica* pad powder. The inverse relationship between the percentage removal of Pb(II) ions and the concentration of the indifferent electrolyte (NaCl), together with the observed overdosing of OFIP dosage, points to the noninvolvement of the double-layer compression mechanism, while the positive correlation between optimal OFIP dosage and percentage Pb(II) ion removal casts doubt on the involvement of sweep flocculation mechanism (Fedala et al. 2015; Miller et al. 2008).

The stoichiometric relationship between colloidal concentration and OFIP dosage together with variation of zeta potential with coagulant dosage is consistent with adsorption and charge neutralization as well as adsorption and bridging mechanisms (Miller et al. 2008). Both mechanisms involve the increase in coagulation up to an optimum dosage after which coagulation diminishes due to steric stabilization. The zeta potential of the OFIP and Pb(II) ions decreases with OFIP dosage, and the optimal dosage (8 mg/L) corresponds to the zero zeta potential (−0.005 mV). The result is closely related to what Ndabigengesere et al. (1995) got using *Moringa oleifera* in remediation of turbid waters. They attributed the result to the involvement of adsorption and charge neutralization mechanism, while Miller et al. (2008) argue that the reduction in coagulation activity when Opuntia spp. dose is too low or too high is consistent with bridging mechanism as a stoichiometric relationship is expected. Renault et al. (2009) concluded that charge neutralization mechanism was involved in chitosan's removal of dyes. Increasing chitosan dosage increased dye removal up to a concentration corresponding to complete neutralization of anionic charges after which the excess of cationic charges added led to suspension re-stabilization. Vishali and Karthikeyan (2014) suggested the involvement of adsorption and charge neutralization mechanism when cactus *Opuntia ficus indica* was used for the removal of color, chemical oxygen demand and turbidity from paint effluent. It is therefore concluded that cactus *Opuntia ficus indica* is involved in a dual mechanism involving coagulation by charge neutralization and flocculation by bridging mechanisms as was reported for chitosan (Renault et al. 2009).

In this coagulation–flocculation system, Pb(II) ions are electrostatically attracted and neutralize the anionic charges on the deprotonated carboxyl groups (–COOH) and hydroxyl groups (–OH) of the macromolecule polygalacturonic acid. Due to its polymeric nature, it becomes attached to several Pb(II) ions, thus bridging them. Adding OFIP dosage beyond the optimum provides excess anionic charges in the system that leads to re-stabilization of the system with a negative zeta potential as depicted in Fig. 5.

Coagulation–flocculation thermodynamics

The thermodynamic parameters of the coagulation–flocculation process between OFIP and Pb(II) ions were estimated. These included the distribution coefficient (K_F) and changes in free energy (ΔG) at different temperatures, enthalpy (ΔH) and entropy (ΔS) of the system.

Change in free energy was calculated using Eq. (2);

$$\Delta G = -RT \ln K_{\mathrm{F}} \tag{2}$$

where R is the universal gas constant of magnitude 8.314 J mol^{-1} K^{-1} and T is the absolute temperature (K). The distribution coefficient, K_{F}, was estimated from Eq. (3);

$$K_{\mathrm{F}} = \frac{q_{\mathrm{F}}}{C_{\mathrm{F}}} \tag{3}$$

where q_F is the amount of metal ion in the floc per gram of flocculant added (mg/g) and was calculated as shown in Eq. (4);

$$q_{\mathrm{F}} = \frac{C_{\mathrm{o}} - C_{\mathrm{F}}}{F_{\mathrm{m}}} \tag{4}$$

F_{m} is the mass of OFIP added as a flocculant (g).

The free energy of coagulation–flocculation was found to be positive at 20, 40 and 45 °C, indicating the non-spontaneity of the flocculation process at these temperatures. This may be attributed to low Brownian movement of the suspended particles at 20 °C that minimized particle mobility. At temperatures above 35 °C, the decrease in coagulation–flocculation can be attributed to the disintegration of flocs and the reduction in the adsorption–bridging mechanism at elevated temperatures. However, the magnitude of ΔG was negative and increased in magnitude from 25 to 35 °C. The coagulation–flocculation process was spontaneous, and the degree of spontaneity increased with temperature, indicating the favorability and endothermic nature of the removal of Pb(II) ions by OFIP (also confirmed by the positive change in enthalpy of the system). The system's optimum temperature was identified to be 35 °C (Table 2).

The change in enthalpy and entropy of the coagulation–flocculation process was estimated from Eq. (5) (Mohammad et al. 2014);

$$\Delta G = \Delta H - T\Delta S \tag{5}$$

Combination, followed by rearrangement, of Eqs. (2) and (5) gives Eq. (6);

$$\ln K_{\mathrm{F}} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \tag{6}$$

A plot of lnK_{F} against $1/T$ generates the gradient and intercept from which ΔH and ΔS can be calculated. The linear relationship between lnK_{F} and $1/T$, in the temperature range 20–35 °C, was determined to be:

$$\ln K_{\mathrm{F}} = -\frac{11491}{T} + 39.07 \tag{7}$$

with correlation of determination (R^2) of 0.9972.

The positive values of the ΔH and ΔS pointed to the endothermicity and increase in disorder of the system as

Table 2 Thermodynamic parameters for Pb(II) and OFIP coagulation–flocculation system

Temperature/°C	K_F	ΔG/kJmol^{-1}	ΔH/kJmol^{-1}	ΔS/kJmol^{-1}K^{-1}
20	0.8912	0.9574	95.5362	0.3248
25	1.6122	−3.9709		
30	3.0072	−9.1537		
35	6.0548	−14.9722		
40	0.6475	3.6135		
45	0.5105	5.5897		

the coagulation–flocculation process occurred. This might be attributed to the dissociation of the polyelectrolyte/sugars in OFIP that requires energy and would generate slightly greater number of ions in solution (increase in entropy).

Total heavy metal analysis and flocculation in Mukuvisi River water

To test for the applicability of the OFIP in the coagulation–flocculation of the real water sample, the technique was applied to the Mukuvisi River water sample. The results are presented in Fig. 7. Based on the maximum concentration levels of these heavy metal in wastewaters ($Pb < 0.006$ mg/L; $Zn < 0.8$ mg/L; $Cd < 0.01$ mg/L; and $Cu < 0.25$ mg/L), the three toxic heavy metals were found to be above the maximum concentration levels (Barakat 2011), confirming the pollution of the Mukuvisi River water calling for the implementation of better wastewater treatment strategies by the responsible industries and city authorities.

The biocoagulation–flocculation of the river water sample was performed in triplicate under optimized conditions, and the results are presented in Fig. 7.

The concentration of lead in the filtrate after the biocoagulation–flocculation process was undetectable by FAAS, suggesting the effectiveness of OFIP in its removal even in the presence of other metal ions. Calculation of the percentage removal using Eq. (1) gave the following results: Pb (100 %), Zn (85.74 %), Cd (84.16 %) and Cu (93.02 %). The difference in the percentage removal was attributed to flocculation conditions and the physicochemical properties of the heavy metal ions such as ionic radius, atomic masses, hard or soft acid, coordination numbers, magnetic properties, electronegativity and reduction potential among others.

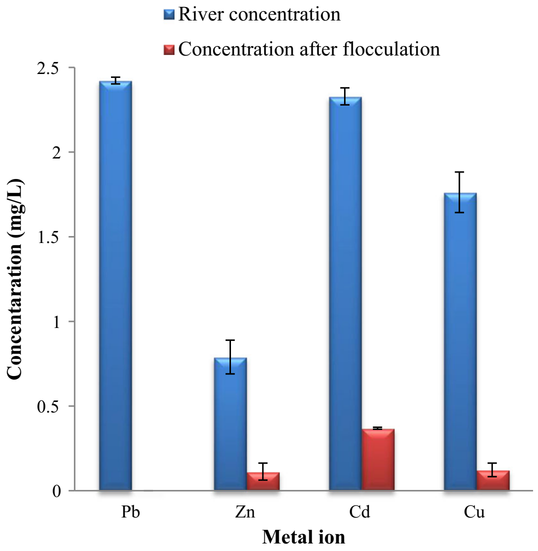

Fig. 7 Total heavy metal concentration in river water before (Co) and after (Cf) bioflocculation using OFIP

Conclusion

Cactus *Opuntia ficus indica* was investigated on its potential to remove Pb(II) ions from wastewaters. FTIR characterization confirmed the involvement of –C–O–C, –OH, –C=O, N–H and –CH groups of the carboxylic acids, amino acids and polysaccharides in the removal of Pb(II) ions. In the investigation of factors affecting coagulation–flocculation process, the percentage removal of Pb(II) ions increased with pH and OFIP dosage up to the optimum and then decreased gradually thereafter which is consistent with adsorption–charge neutralization and adsorption–bridging mechanisms in coagulation–flocculation processes. The optimum pH and OFIP dosage were found to be 5 and 8 mg/L, respectively. The zeta potential of the OFIP–Pb(II) ion system was found to decrease with increase in OFIP dosage, and the point of zero charge occurred at a dosage of 8 mg/L (optimum). The coagulation–flocculation process was most efficient using OFIP particle size <75 µm, at a temperature of 35 °C for a settling period of 180 min. A thermodynamic study revealed the flocculation process to be spontaneous and endothermic in nature and was associated with a slight increase in entropy. Extremely high percentage removal of Pb, Zn, Cd and Cu was observed when the flocculant was tested on Mukuvisi River water that was found to contain total heavy metal concentrations above acceptable limits. The results show OFIP to be an effective bioflocculant of Pb(II) ions that needs to be explored at commercial and industrial scales.

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