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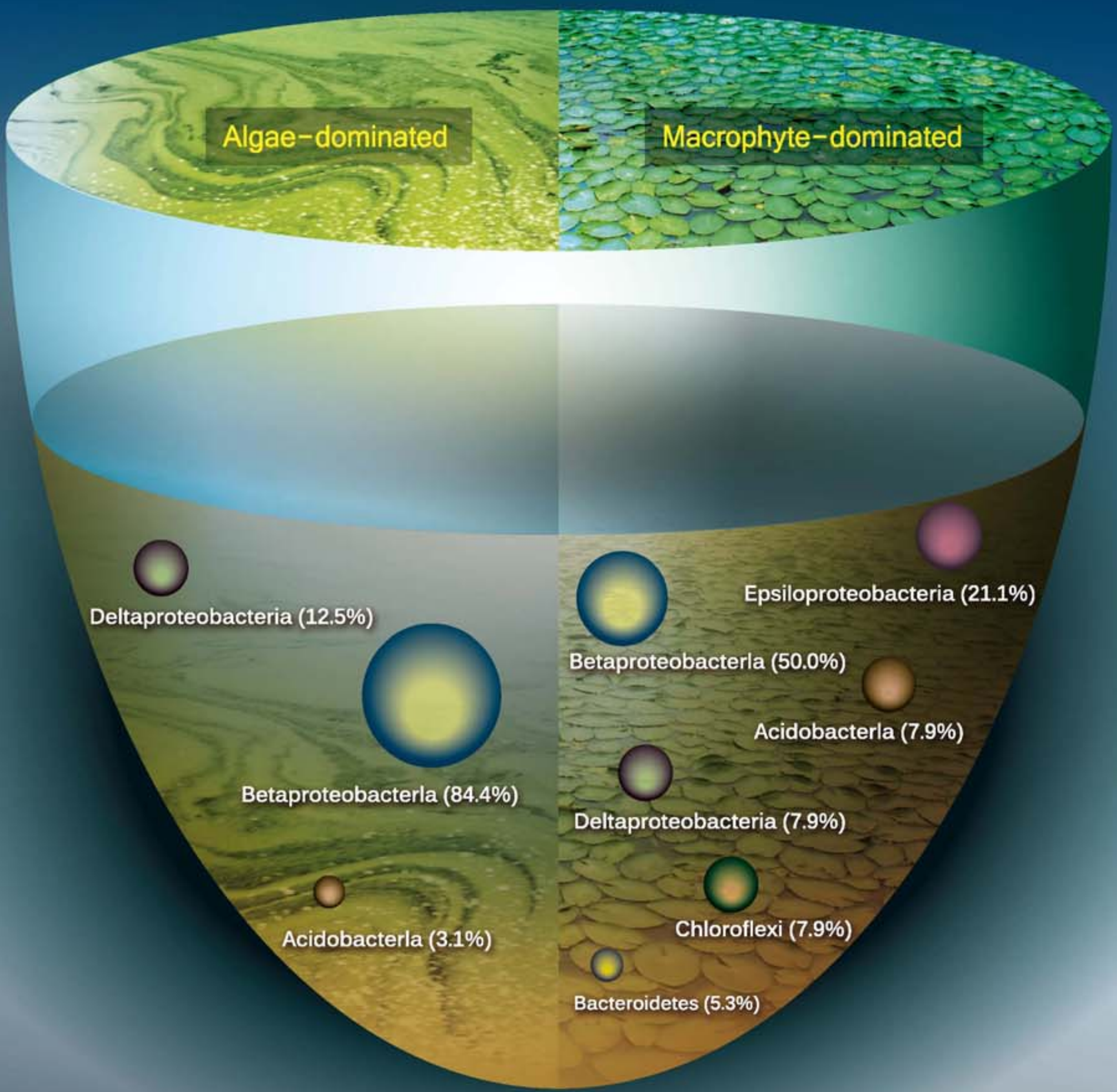

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CONTENTS

Environmental biology

Environmental catalysis and materials

Environmental analytical methods

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Evaluation of remediation process with soapberry derived saponin for removal of heavy metals from contaminated soils in Hai-Pu, Taiwan

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Abstract

The use of a biodegradable natural plant-based surfactant extracted from soapberry is proposed for the remediation of Ni, Cr and Mn from industrial soil site in Hai-Pu, Taiwan. Batch experiments were performed under variation of fundamental factors (saponin concentration, pH, and incubation time) for metal remediation. Removal of Ni and Mn were increased with increasing saponin concentration (0.015–0.150 g/L), whereas the removal of Cr was increased upto 0.075 g/L saponin. The Ni, Cr and Mn were removed significantly ($p \leq 0.05$) at near to the neutral and slightly acidic (pH 5 to 8) conditions. Removal efficiency of Ni (99%) from the soil was found to be greater than that of Cr (73%) or Mn (25%) in the presence of saponin at a concentration of 0.150 g/L at pH 5. The removal percentage increased with incubation time where the removal of Ni was faster than that of Cr and Mn. The result indicates the feasibility of eco-friendly removal of heavy metal (Ni, Cr and Mn) from industrial soil by soil washing process in presence of plant derived saponin.

Key words: heavy metal; remediation; contaminated soil; soapberry

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Introduction

Heavy metal including trace element contamination in soil, groundwater, and the food chain associated with human health problems arising from metal poisoning have been widely reported (Rudolf and Cervinka, 2010; Lin et al., 2009; Fozia et al., 2008; Prasad, 2004). Neurodegenerative disorders are related to manganese (Mn) exposure (Weiss, 2011). Exposure to nickel (Ni) and its soluble compounds could be carcinogenic, and causes skin allergy, resulting in itchy, red skin. Ni could inhibit enzyme activity (Wyszkowska et al., 2005). Chromium (Cr) enters cells and can lead to DNA damage. The high amount of heavy metal compounds are often found in soil and groundwater at abandoned industrial and mining waste sites (Rodriguez-Castillo and Armienta, 1994; Hansen et al., 2003; Winder and Carmody, 2002; Nestle et al., 2002). At present, the environmental clean-up and remediation of these heavy metals is needed.

Soil washing process is effective to remove the heavy metal from contaminated soil (Dermont et al., 2008). The chelating agents (EDTA, D-gluconic acid, D-glucaric acid, citric acid, and tartaric acid) have the ability to remove heavy metals (Cd, Cr, Cu, Ni, Pb, Zn) from contaminated soil (Fischer and Bipp, 2002; Wuana et al., 2010). The organic chelating acids showed that the soil washing efficiencies increased in the order of: ethylenediaminetetraacetic acid > citric acid > tartaric acid with metal removal in the order of Cu > Ni > Zn > Cd > Pb (Wuana et al., 2010). The main drawbacks of EDTA and other chelating agents are the chemical products and more expensive, low biodegradability and causes of serious environmental hazard if it is not recycled/destroyed during/after the soil washing process. Biodegradable, eco-friendly removal of metal by soil-washing procedures can be improved by using biosurfactant (Chen et al., 2008; Aşccı et al., 2007;

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Massara et al., 2007; Kim and Vipulanandan, 2006; Mulligan, 2005). The saponin successfully competes with the kaolin clay for complexing with the Cu and Ni ions by the Lewis acid-base interaction and metal-desorption efficiencies are documented in the order of EDTA > saponin ≫ SDS (Chen et al., 2008). An available washing mechanism involves, biosurfactant is able to remove heavy metals through sorption at the soil-liquid interface, desorption by reducing the interfacial tension and heightening the forces of liquid streams, and then formation of micellar surfactant-metal complexes in solution. The researchers have been reported for the removal of heavy metal (Cd, Cu, Pb and Zn) from the contaminated soils by saponin (Song et al., 2008; Chen et al., 2008; Hong et al., 2002).

In this study, we investigated the potential removal of Cr, Ni and Mn from polluted soil using Soapberry-derived saponin. The objective of this study consists of the removal process optimization and efficiency assessment in different levels such as pH, incubation time and saponin concentration.

1 Materials and methods

1.1 Soil collection, sample preparation and estimation soil properties

Soil samples were collected from Hai-Pu Industrial Park in Kaohsiung of Taiwan, China and the soil had been contaminated by heavy metals for a long period of time (> 10 years). The collected soils were air-dried, ground and sieved through a 2-mm sieve. The homogenized soil samples were stored in a plastic container for subsequent experiments.

Soil pH was measured in deionized water using a solid:liquid ratio of 1:1 after 1 hr of stirring to achieve equilibration (McLean, 1982). Soil electric conductivity (EC) was measured in deionized water using a solid:liquid ratio of 1:1 following 1 hr of shaking and filtration through Whatman No. 42 paper (Rhoades, 1982). Selective total metals were determined by ICP-MS following extraction with aqua regia (4:1, V/V, HCl to HNO_3) (McGrath and Cunliffe, 1985). Soil particle size analysis was carried out by the hydrometer method after pretreatment (Gee and Bauder, 1986). Selected chemical characteristics of the soil samples are presented in **Table 1**.

1.2 Soil properties

The Hai-Pu industrial soil is mainly sandy-clay soil type with higher amount of organic mater (10,000 mg/kg). The high content of organic matter indicates the high metal binding capacity of soil (Heredia et al., 2002; Lo et al., 1992). The soil properties indicated that the pH of the soil was (7.80 ± 0.02) (mean ± SE) (**Table 1**). The EC and CEC of the soil were (0.23 ± 0.01) dS/m and (2.80 ± 0.01) $cmol_c$/kg, respectively. Heavy metals Cr and Ni were present at (70.00 ± 1.12) mg/kg and (151.00 ± 1.98) mg/kg, respectively. The other heavy metal such as Mn, K, Ca and Mg were noticed at low level, which were measured at (476.00 ± 3.50), (54.00 ± 0.25), (602.00 ± 3.56), and (12.00 ± 0.15) mg/kg, respectively.

Table 1 Selected chemical properties of the soil samples in this study

Parameter	Concentration	Parameter	Concentration
Soil type	Sandy-clay	Organic matter (mg/kg)	10000.00 ± 4.36
pH (1:1, V/W)	7.80 ± 0.02	Ni (mg/kg)	151.00 ± 1.98
EC (dS/m)	0.23 ± 0.01	Cr (mg/kg)	70.00 ± 1.12
CEC ($cmol_c$/kg)	2.80 ± 0.01	Mn (mg/kg)	476.00 ± 3.50
Sand (%)	78.40 ± 2.21	K (mg/kg)	54.00 ± 0.25
Clay (%)	9.56 ± 1.01	Ca (mg/kg)	602.00 ± 3.56
Silt (%)	12.00 ± 1.11	Mg (mg/kg)	12.00 ± 0.15

Data represent mean ± SE, where $n = 6$, $p < 0.05$.

1.3 Properties and estimation of saponin

The saponin biosurfactant (local name of the plant: soapberry; scientific name of the plant: *Sapindus mukorossi* L.) was extracted from soapberry as described by Shiau et al. (2009). Saponin is non-volatile, surface–active compounds with the composition of hydrophilic glycoside backbone and lipophilic triterpene derivative. It contains several functional groups such as carboxylate, OH, COOH, esteric band, and acetate group (Shiau et al., 2009; Sharma et al., 2013). The concentration of saponin was measured by colorimetric assay following the procedure of Shiau et al. (2009). Aqueous solutions of the organic extract of crude saponin were prepared in different dilution factors for further analysis. Standard saponin (Quillaja) (Sigma Chemical Company, St. Louis, USA) was used to standardized the experiment (**Fig. 1**) and the experimental error was ± 2.324%.

1.4 Soil-washing and estimation of heavy metal

The soil-washing process with saponin was performed in a batch experiment as a function of concentration, pH and time at room temperature (26–29°C). To determine the optimum concentration of saponin and the optimum

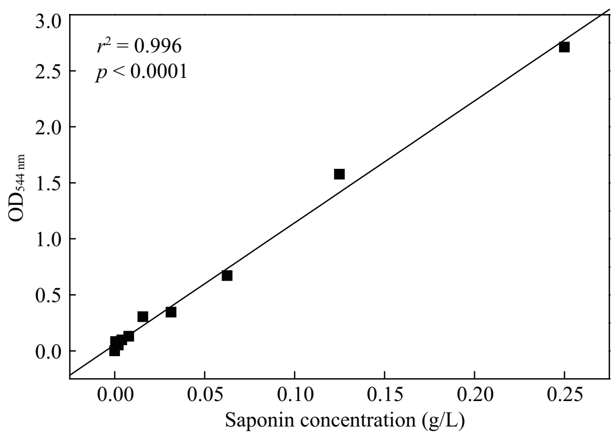

Fig. 1 Standard optical density ($OD_{544\ nm}$) curve of reference saponin (quillaja).

pH for soil treatment, 1 g of soil was placed in a series of polycarbonate centrifuge tubes and 25 mL of solution containing saponin at various concentrations was added with deionized water. To quantify the effects of variation of saponin concentration and pH level on heavy metal removal, it performed different sets of experiments in which the saponin concentration was varied (0.015–0.15 g/L). At an instant, when saponin was used at different concentrations the pH remained unchanged, to test the heavy metal removal efficiency. The pH values such as 5.0, 6.0, 7.0, 8.0 and 9.0 were taken under study, whereas the incubation time was varied upto 72 hr with 6 hr interval. The suspensions were shaken and centrifuged using a refrigerated centrifuge 10000 r/min for 10 min (before estimation of heavy metal). The supernatant was subsequently withdrawn and filtered through a 0.45-μm nitrocellulose membrane filter. The heavy metals of supernatant and soil (before and after washing process) were analyzed by ICP-MS (Hewlett-Packard, Yamanashi-Ken, Japan). A standard material was used for this study (SRM-2704 and SRM-2711). The experimental errors of ICP-MS were within the range of ± 2.163% to ± 3.521%.

1.5 Statistical analysis

The statistical significance of this study was analyzed using analysis of variance (ANOVA) followed by the *T*-test, and the significance level was set at 0.05. Data summary and calculations were performed using Microsoft Excel and Statistica 5.1, and graphs were drawn using Origin 6.1 software.

2 Results and discussion

2.1 Heavy metal removal from soil with different concentrations of saponin and variation of pH

The removal of heavy metal was changed with the factor of saponin concentration and pH. The effect of saponin concentration on metal removal was expressed as a percentage with respect to the initial concentration of the metal in the soil. Removal efficiency of Ni was found to be dependent on saponin concentration (**Fig. 2a**). The lowest concentration of saponin (0.015 g/L) was capable of removing 39% of Ni at pH 5, and 50% of Ni was removed at a saponin concentration of 0.030 g/L at the same pH. Maximum removal (99%) of Ni was achieved at a saponin concentration of 0.150 g/L at pH 5. The removal efficiency of Ni decreased with an increase in pH from 5 to 9. At pH 7, the removal efficiency of Ni was found to be nearly 69% at saponin concentration 0.150 g/L. Fischer and Bipp (2002) reported that 43% of Ni can be removed from sewage sludge by sugar acid. Hong et al. (2002) reported that saponin was effective for removal of heavy metals from soils at pH 3. In present study, the removal efficiencies of Ni are function of mainly two factors such as pH and saponin concentration.

In case of Cr, the removal efficiency was found to be nearly 3% at a saponin concentration of 0.015 g/L at pH 5 (**Fig. 2b**). And 50% of the Cr was removed at a saponin concentration of 0.150 g/L at pH 5. Similar to the result of Ni, the removal efficiency of Cr decreased with increasing pH. These results are in agreement with the report of Kiliç et al. (2011), which indicated that the efficiency of Cr removal from tannery waste decreased with increasing pH and increased with increasing saponin concentration. The amount of sorbed saponin (onto soil) is increased with decreasing pH due to the increased electrostatic attraction between saponin and the soil surface (Hong et al., 2002). The increasing amount of saponin may cause of greater metal removal at a constant pH. At pH 7, the Cr removal efficiency was found to be nearly 36% when the saponin concentration was 0.150 g/L. In compares between Ni and Cr, the saponin was capable to remove Ni more efficiently than Cr from the soil. At a saponin concentration of 0.150 g/L the removal efficiency decreased from 50% to 15% with an increase in pH from 5 to 9 (in case of Ni: the removal efficiency was decreased from 100% to 46%). Interestingly, 50% of the Cr was removed at a saponin concentration of 0.150 g/L at pH 5, whereas 50% of the Ni was removed at a saponin concentration of 0.030 g/L at pH 5 or at saponin concentration of 0.075 g/L in the pH range of 5 to 7. The previous researcher reported that the percentage of Cr extraction with the saponin increased

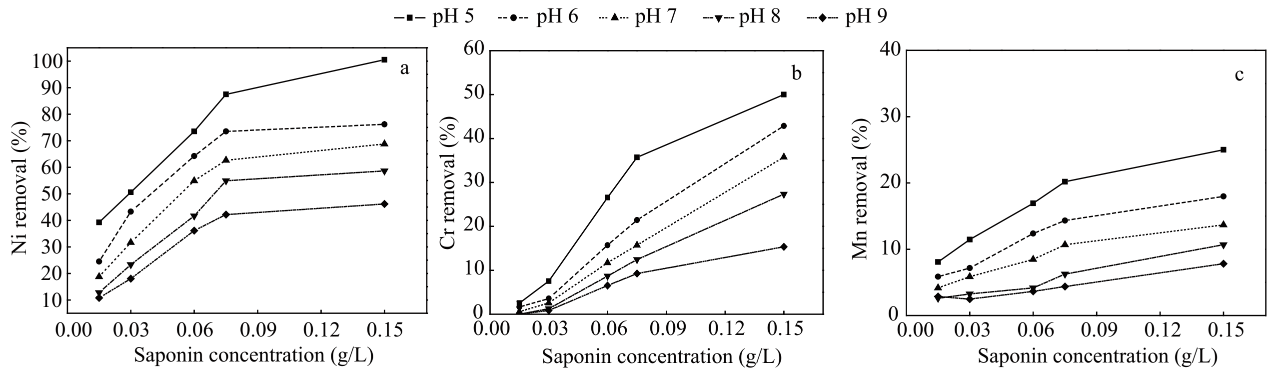

Fig. 2 Removal of heavy metals from contaminated soil at different saponin concentrations (0.015, 0.030, 0.060, 0.105, and 0.150 g/L) and different pH values (5–9). (a) Ni, (b) Cr, (c) Mn. Data represent percentage of mean ± SE, $n = 6$ ($p < 0.05$).

upto 45% with decreasing pH (4–7) from fly ash and it exhibited slightly higher percentage than EDTA (Hong et al., 2000).

The efficiency of Mn removal from the soil was found to be lower than that of Cr and Ni. Only 25% of Mn was removed from the soil at a saponin concentration of 0.150 g/L at pH 5 (**Fig. 2c**). Similar trends in the removal pattern to those of Cr and Ni were observed in the case of Mn. **Figure 3** clearly indicated that the metal removal are varied and/or increased with the increase of saponin concentration and decreasing with working pH. The variations of metal removal were noticed higher in increasing pH (**Fig. 3b, d,** and **f**) in compare to the increasing saponin concentration (**Fig. 3a**, **c**, and **e**). The removal efficiency of a certain heavy metal is not only related to COO– or –OH but also to the concentration of saponin (Yuan et al., 2008). In this study, NaOH was added to adjust the pH of saponin solution. The Na-saponin complexes could have been formed during pH adjustment and replaced by the other heavy metal ions after use, thereby leading to the removal of a metal-saponin complex (Hong et al., 2002). The removal efficiencies were noticed in the order of Ni > Cr > Mn. This result is consistent with the report of Hong et al. (2002). In another study, the removal of Cr increases not only from soil but also from fly ash with increasing concentrations of saponin (Hong et al., 2000). Therefore, saponin is an effective agent for washing heavy metals from contaminated soil. The results of present study

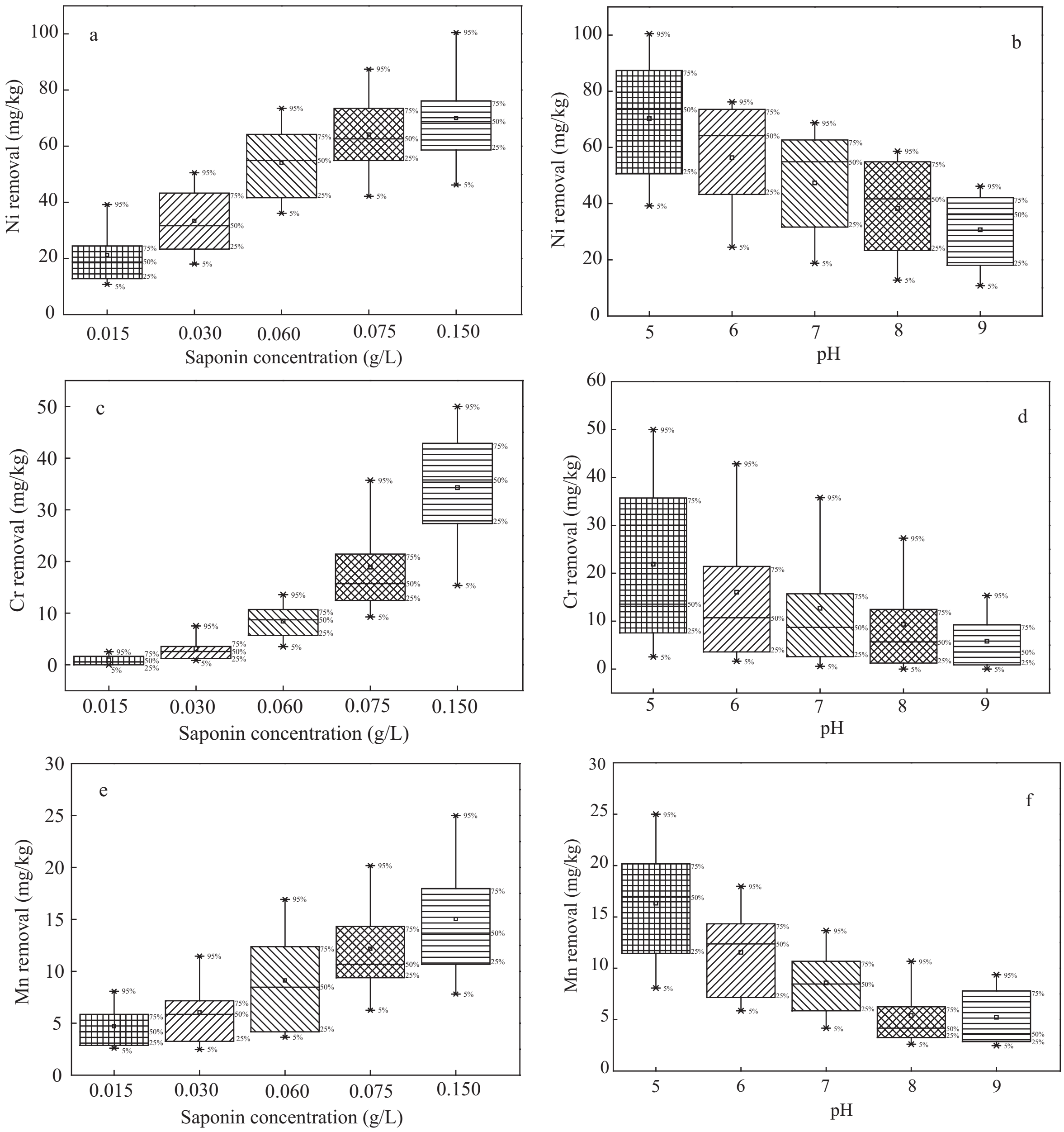

Fig. 3 Variation in heavy metal removal at different saponin concentrations and different pH values: (a) and (b) Ni, (c) and (d) Cr, (e) and (f) Mn. The Box-whisker plot represents maximum score, 75th percentile (Upper Quartile), Median, 25th percentile (Upper Quartile) and Minimum Score.

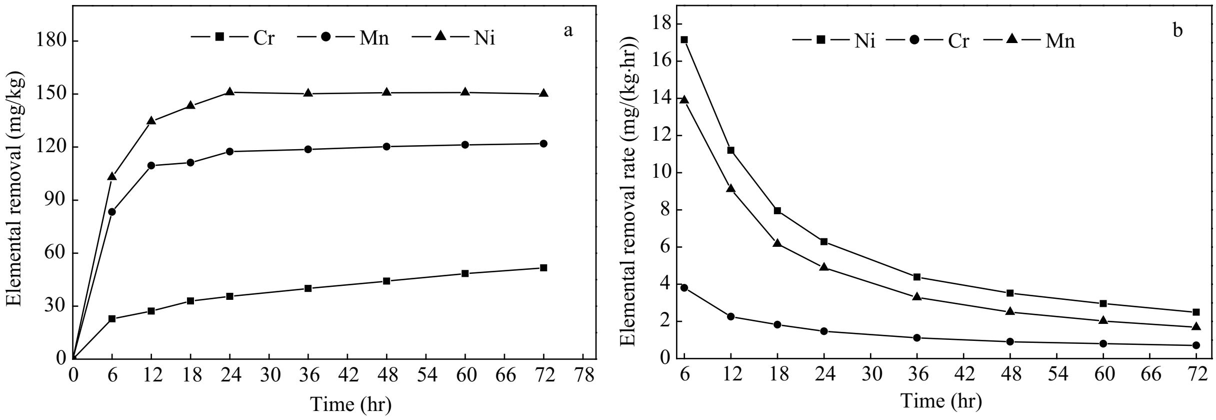

Fig. 4 (a) Removal of Ni, Cr and Mn in different incubation time upto 72 hr (6 hr interval), data represent percentage of mean ± SE, $n = 6$ ($p < 0.05$); (b) removal rate of metal (Ni, Cr and Mn) with time from contaminated soil at a constant saponin concentration of 0.150 g/L at pH 5.

clearly showed that the saponin concentration needs to be high in order to remove metal from soil if the soil pH and initial concentration metal is high.

2.2 Time dependent metal removal at a constant saponin concentration and pH

The removal of heavy metal from contaminated soil in different time durations (upto 72 hr with 6 hr interval) by 0.150 g/L saponin at pH 5 is represented in **Fig. 4a**. The results showed that 68.19% of Ni (with respect to the initial concentration in the soil) was removed after a 6 hr incubation period, whereas Cr and Mn was removed 32.57% and 17.50% from contaminated soil, respectively (the same incubation time). Complete removal of Ni (100%) was observed after 24 hr of incubation; however, only 50.70% (51.64 mg/kg) of Cr and 24.67% (121.94 mg/kg) of Mn was released from the contaminated soil after the same duration (24 hr). According to previous reports, the saponin is effective (85%) for desorption of Ni from kaolin clay at pH 5.4 within 3 hr (Chen et al., 2008). Present results reflect the better removal efficiency of Ni (100%) within 24 hr. The different incubation time indicates the efficiency of metal removal depends upon soil compositions and inter-elemental interaction. Removal of Mn was insignificant after 24–72 hr of incubation (nearly 1% removal efficiency). However, the removal efficiency of Cr increased from 50.70% to 73.77% with an increase in the incubation period from 24 to 72 hr, respectively, which indicates the optimization of contact time between soil and saponin is very important for the heavy metal removal from contaminated soil.

The elemental removal rates are shown in **Fig. 4b**. After 6 hr of incubation, the Ni removal rate was found to be greater (17.16 mg/(kg·hr)) than the Mn (13.89 mg/(kg·hr)) and Cr removal rates (3.80 mg/(kg·hr)). A sharp decreasing trend in the removal rate was noticed with an increase in incubation time from 6 to 36 hr in the case of Ni (4.39 mg/(kg·hr)) and Mn (3.29 mg/(kg·hr)). The removal of Ni was faster than that of Cr and Mn. The slow removal of Cr may be due to that the sorbed metal more strongly bound to interior surface sites. Care needs to be taken for the format of chromium in the polluted soil. They could be cationic Cr(III) and anionic Cr(VI). The former might form a complex with saponin, while there is no evidence supporting the complexation between saponin and Cr(VI), which also leading to the low removal of Cr (Kiliç et al., 2011). These results are consistent with the previous report, where the removal rate of Ni is greater than Cr with sugar acid extraction after 72 hr (Fischer and Bipp, 2002). In the present study, it was probably the case that Cr and Mn were tightly bound to the organic matter, delaying the formation of soluble saponin-Cr or saponin-Mn complexes. More detailed study of the mechanisms in the cases of Ni, Cr and Mn needs to be performed. However, biosurfactants have significant advantages of lower toxicity, higher biodegradability, better environmental compatibility, higher selectivity for metal ions removal, less expensive, less sensitive to pH and temperature variations. So it can be applicable for elemental removal process in environment.

3 Conclusions

Saponin of soapberry is able to remove heavy metals such as Ni, Cr and Mn from contaminated soil. The removal efficiency is strongly influenced by the saponin concentration, pH and incubation time. The removal efficiency increases with increasing saponin concentration and incubation time. Removal capability is very high at near-neutral and slightly acidic pH values (5–7) at a high saponin concentration of 0.150 g/L, but the removal potential drastically decreases between pH 8 and 9. The Ni removal was faster than that of Cr and Mn. Cr was released slowly due to its strong binding with soil molecules in comparison with Ni and Mn.

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