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Mitigation of Contamination Levels and Ecological Risk of Toxic Metal Contaminated Soil using CA-EKSR Process

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Abstract : Many studies have focussed on decontamination of the soil by incorporating electrokinetic technologies. However, these not considered to determine the contamination and decontamination levels before and after treatment. We addressed this problem and determined the decontamination levels of toxic metals(TMs) after treatment. In this study, TMs contaminated granite mining soil was treated with the chelating agents and electrokinetic soil remediation (CA-EKSR) process. After 20 days of treatment, we determined the decontamination levels and ecological risk indices of TMs through pollution index (PI), Geo-accumulation index (I_{geo}) and potential ecological risk index (RI). The removal performance of TMs was found in CA-EKSR (citric acid and EDTA as chelating agents) treatment about 5-6 times more than when conventional treatment. The overall ecological risk index (RI) was reduced from 224 to 35.6, after treatment with chelating agent, which indicated that the soil has low environmental risk. It is observed that the CA-EKSR treatment was an effective in the remediation and mitigation of TMs from contaminated mining waste. The study is useful for the researchers to investigate the soil quality, contamination levels and ecological risk indices particularly in the soil decontamination studies.

Keywords : Electrokinetic technology, Heavy metals, chelating agents, Pollution index, Geo-accumulation index.

1. Introduction

Several efforts focussed on decontamination by incorporating electrokinetic soil remediation technologies¹⁻⁴. However, these studies have not considered to assess the contamination and decontamination levels before and after treatment. We addressed this problem and determined the decontamination levels of toxic metals (TMs) after electrokinetic treatment. The electrokinetic soil remediation (EKSR) technology has been emerged as a prominent in the remediation of toxic metals from contaminated soils/sediments/sludge. The principle involved in this process is electrolysis of water, electromigration, electro-osmosis and electrophoresis⁵.

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Therefore, the assessment of soil quality, contamination levels and risk indices of contaminated soils are necessary and useful in the effective removal of toxic metals to improve the environmental quality. Many studies have been performed to assess the environmental impact and risk of mining and metallurgy industries based on their geochemical content and concentrations^{5,6}. Recently⁷, investigated the contamination levels and ecological risk indices of environmentally hazardous metals for granite mining waste including dump, stock yard and mineral bench soils. They reported that the dump soil has more contamination and more ecological risk than mineral and stock yard soils due to continuous accumulation of granite waste at dump yard in the mining quarries.

In the present study, we used the same dump yard soil to study the electrokinetic removal of toxic metals from granite mining waste. The study is divided into two sections. First section focussed on chelating agents enhanced-electrokinetic soil remediation (CA-EKSR) process. Subsequent section, focussed on assessment of contamination levels and ecological risk indices imposed by TMs through the various indices such as pollution index (PI), Geo-accumulation index (I_{geo}) and potential ecological risk index (RI) after CA-EKSR treatment for 20 days.

2. Experimental

2.1. Determination of Toxic Metals (TMs) Concentration

Mining dump soil was collected from the top layer of soil (0-30cm) from the granite industry at Chimakurthy, India. The collected samples were sieved through a series of 4.75-0.002 mm mesh nylon fibre sieves and removed any vegetation fragments and large material. Finally, samples were well homogenized and kept in inert zip plastic bags at room temperature for further study^{8,9,10}. The acid digested soil solutions were subjected to atomic absorption spectrometry (AAS) in order to measure the TMs concentration¹¹, prior to electrokinetic remediation. The concentrations of TMs are reported in **Table 1**.

Table 1. The Concentrations of TMs in Granite Mining Soil

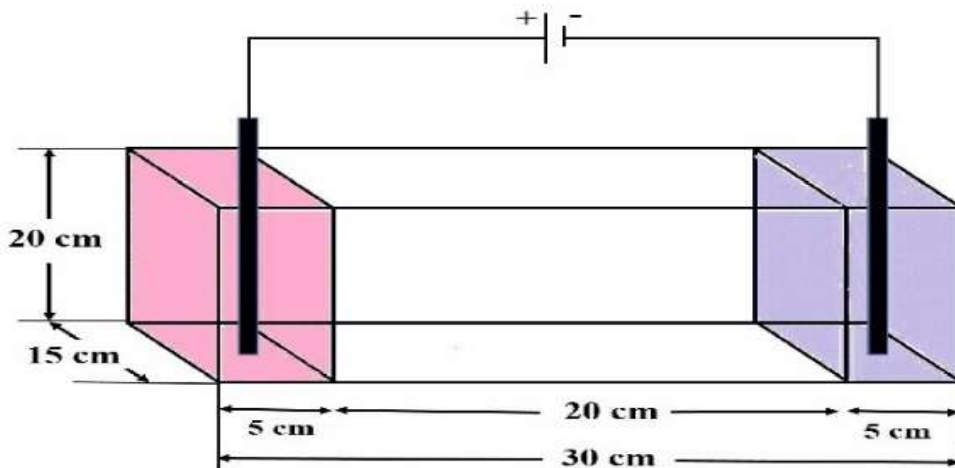
Soil characteristics	Units	Value	Method
Chromium (Cr)	mg kg ⁻¹	192	Acid digestion/AAS
Cobalt (Co)	mg kg ⁻¹	175	Acid digestion/AAS
Nickel (Ni)	mg kg ⁻¹	204	Acid digestion/AAS
Copper (Cu)	mg kg ⁻¹	363	Acid digestion/AAS
Zinc (Zn)	mg kg ⁻¹	505.1	Acid digestion/AAS
Manganese (Mn)	mg kg ⁻¹	704	Acid digestion/AAS

2.2. Experimental Apparatus

A laboratory-scale electrokinetic reactor was used in this study^{3,12-18}. The reactor was made of Thermocol box with dimensions of 30cm x 20cm x 15cm and two electrode compartments with dimensions of 5cm x 20cm x 15cm and soil matrix with dimensions of 20cm x 15cm x 5cm with a working volume of 1.5L. The two folded filter papers (Whatman 42) were placed between the soil matrix and therefore the electrode compartments to avoid the outflow of the soil from the soil matrix to the electrode chambers. The graphite electrodes with dimensions of 15cm in height and 1.5cm in diameter were used as anode and cathode. Multi-meter was used to measure the current intensity and voltage drop during EKSR process. Digital pH meter was used to test the pH of the anode reservoir, cathode reservoir and different soil sections periodically during the operation. Regulated DC power supply with voltage ranged from 0-128V and 5A maximum current flow in output, was used during EKSR experiments. As seen in **Table 2**, electrode compartments were filled with prior prepared processing fluids. **Fig. 1** represents the schematic experimental design set-up for EKSR reactor.

Table 2. Experimental Condition of Four EKSR Experiments

Exp. No	Anolyte purging solution	Soil saturation	pH	Catholyte purging solution	Duration (Days)	Voltage (V/cm)
1	Distilled water	Distilled water	9.7	Distilled water	5,10,15,20	2
2	Distilled water	0.1M Citric acid	5.7	0.1M Citric acid	5,10,15,20	2
3	Distilled water	0.1M Citric acid	5.9	0.1M EDTA	5,10,15,20	2
4	0.1M NaOH	Distilled water	9.7	Distilled water	5,10,15,20	2

**Fig.1. Experimental Design Set-up for EKSR Reactor**

2.3. Experimental Design

In this study, four EKSR experiments were carried out for four electrokinetic manipulation patterns as shown in **Table 2**. In the case of unenhanced-EKSR Exp.1, the anode and cathode compartments were filled with double distilled water. In the case of citric acid enhanced EKSR Exp.2, the soil was saturated with 0.1M of citric acid and pH was adjusted to 5.7 prior to the experimentation. The anode chamber was stuffed with double distilled water and the cathode chamber was stuffed with 0.1M of citric acid about 500ml. In the case of EDTA-enhanced EKSR Exp.3, the soil was pre-saturated with 0.1M of citric acid and pH was adjusted to 5.9.

In Exp.4, the soil was pre-saturated with double distilled water. The anode compartment was filled with 0.1M of NaOH to maintain the alkali environment at the anode side. The anolyte and the catholyte were refreshed daily once to maintain the electrolyte properties at constant in all experiments. Prior to experimenting, approximately, 2000g of dry soil mixed with 1000ml of distilled water in a separate plastic tray and stirred manually for several minutes to achieve homogeneity for Exp.1. In Exp.2 and 3, approximately, 2000g of dry soil mixed with 1000ml of citric acid to reduce the soil pH and in Exp.4, 2000g of dry soil mixed with 1000ml of distilled water, and stirred manually for 30 minutes to achieve homogeneity.

A uniform voltage gradient 2V/cm was provided to the slurred soil matrix for four EKSR experiments and treated for twenty days. Two graphite electrodes as anode and cathode had placed within the electrokinetic cell and supplied the electric current. The current intensity and the pH were continuously monitored daily by inserting multi-meter and pH probes directly into the anode and cathode reservoirs under steady state conditions. The anolyte and catholyte were drained once each day and kept aside for analysis of TM concentrations. The extracted 1g of soil and 5ml of distilled water were placed in a 50ml beaker and stirred with glass rod for 10min., and then the pH was recorded, similar trend was repeated for other sections of extracted soil from soil matrix. Soil samples from four soil sections were collected at the end of each experiment after 5, 10, 15 and 20 days of operation further analysis of heavy metal concentrations. Further, the removal performance of TMs during EKSR process was determined as following Eqn. (1) ¹⁹⁻²²Removal efficiency (%):

$$\frac{C_0 - C_f}{C_0} \times 100 \quad (1)$$

Where, C_0 , C_f are initial and final concentration (mg/kg) of the soil after EKSR treatment.

2.4. Pollution Index (PI)

The pollution index PI was defined by the following relation and integrated pollution index IPI is defined as the mean value of the pollution index PI of an element ²³:

$$PI = \frac{C_i}{C_{b,i}} \quad (2)$$

Where, The C_i and $C_{b,i}$ are the measured and the background concentration of the soil. **Table 3** shows the classification of PI.

Table 3. Classification of PI²³

PI value	Level of pollution
$PI \leq 1$	Low
$PI \leq 3$	Middle
$PI > 3$	High

2.5. Geo-accumulation Index (I_{geo})

The geo-accumulation index (I_{geo}) used to estimate the soil contamination levels by potentially hazardous chemical species particularly, TMs, radionuclides and organic pollutants²⁴. In this study, we determined the geo-accumulation index (I_{geo}) by using the following expression:

$$I_{geo} = \log_2 \left(\frac{C_i}{1.5 \times C_{b,i}} \right) \quad (3)$$

Where, I_{geo} , the geo-accumulation index used to estimate the levels of the environmental contamination via hazardous chemical species in the soil. The C_i and $C_{b,i}$ are the determined and background concentration of the soil. The arbitrary constant value 1.5 used for natural fluctuations in the environment of a chemical species and allows for the identification of any small influences of anthropogenic activity²³. The geo-accumulation index values are graded in to seven types and the highest grade indicates (>5) that the soil has contaminated extremely by TMs. The seven grades of I_{geo} reported in **Table 4**.

Table 4. The Seven Grades of the I_{geo} ^{5,6}

Grade	Value	Soil quality
0	$I_{geo} \leq 0$	Practically uncontaminated
1	$0 < I_{geo} \leq 1$	Uncontaminated to moderately contaminated
2	$1 < I_{geo} \leq 2$	Moderately contaminated
3	$2 < I_{geo} \leq 3$	Moderately to heavily contaminated
4	$3 < I_{geo} \leq 4$	Heavily contaminated
5	$4 < I_{geo} \leq 5$	Heavily to extremely contaminated
6	$I_{geo} > 5$	Extremely contaminated

2.6. Ecological Risk Assessment

The potential ecological risk of a given contaminant is defined as ^{25,26}:

$$RI = \sum_{i=1}^n E_r^i = \sum_{i=1}^n \left[T_r^i \times \left(\frac{C_i}{C_{b,i}} \right) \right] \quad (4)$$

Where, RI is the environmental risk index to assess the ecological toxicity levels by potentially harmful elements in the soil sample. The E_r is the potential ecological risk index for i^{th} chemical species, T_r is the biological toxicity factor for i^{th} chemical species. The C_i and $C_{b,i}$ are the measured concentration and the expected background concentration of the soil. Environmental risk assessment can be performed by using a wide variety of factors including the multi-element synergy, toxicity level, contaminated concentration and sensitivity to HM contamination to the environment.

Eq.1 considered these factors to assess the environmental risk index or ecological risk index. The classification and the standard values of potential ecological risk index (E_r) for i^{th} chemical species and the ecological risk index (RI) were reported in the **Table 5**. The toxic-response factor for the TMs in the ascending order of Hg, As, Pb, Co, Ni, Cu, Cr, Zn and Mn are 40, 10, 5, 5, 5, 5, 2, 1 and 1 respectively^{27,28}. In the study, we determined the environmental risk assessment in order to investigate the level of potential ecological risk posed by the hazardous TMs in the four groups of soils including mineral bench site, dump yard, stock yard and agricultural soils in the vicinity of the mining area.

Table 5. Classification and Standard Values of E_r^i and RI^{6,14}

E_r^i	Single pollutant degree of environmental risk	RI	Comprehensive environmental risk level
$E_r^i \leq 40$	Low ecological risk	$RI \leq 150$	Low ecological risk
$40 < E_r^i \leq 80$	Moderate ecological risk	$150 < RI \leq 300$	Moderate ecological risk
$80 < E_r^i \leq 160$	Considerable ecological risk	$300 < RI \leq 600$	Considerable ecological risk
$160 < E_r^i \leq 320$	High ecological risk	$RI > 600$	Very high ecological risk

3. Results and Discussion

3.1. Unenhanced EKSR Process

The acid digestion derived TM content residual within the soil cell varying with time during unenhanced EKSR treatment. Generally, TMs are moved between the anode and the cathode via electromigration and electroosmosis process. As observed, the removal efficiency of TMs increasing with increasing the treatment time from 5 to 20 days. It can be seen from **Fig. 2(a)**, in Exp.1, the percentage removal of TMs for chromium about 6.22%, for cobalt about 9.2%, for nickel about 16.34%, for copper about 23.7%, for zinc about 11.42% and for manganese about 32.45% respectively after 20 days of operation. The removal percentages of TMs were higher in anolyte than catholyte due to high acidic front generated at anode compartment, where TMs were precipitated. From this, for various remediation times, it is observed that longer remediation times increases the removal of TMs within the soil close to the anode due to high acidic front formation.

3.2. Chelating Agents-Enhanced Electrokinetic Soil Remediation (CA-EKSR) Process

The chelating agents extracted the TMs residual in soil cell depends on time during enhanced EKSR process. In Exp.2, the catholyte conditioning was carried out using 0.1M of citric acid, at the end of the EKSR process (20d), the removal efficiency of TMs increasing over treatment time because of the migration of positive charge TM ions towards cathode via electromigration. It is observed that, the removal of all TMs was more in citric acid enhanced EKSR treatment than unenhanced EKSR treatment (Exp.1), because of easy desorption and quick migration of metal ions onto the soil surface to pore water at acidic environment.

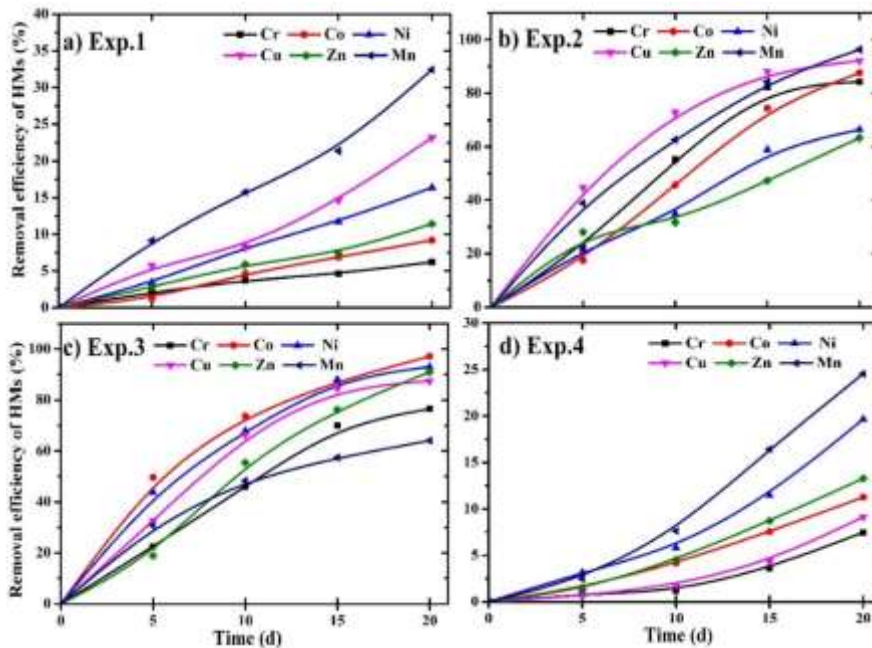


Fig.2. Removal of TMs during Time for Four Different EKSR Experiments.

After 20d of the operation, as seen from **Fig. 2 (b)**, in Exp.2, the percentage removal of TMs for chromium about 84.32%, for cobalt about 87.66%, for nickel about 66.41%, for copper about 92.16%, for zinc about 63.35% and for manganese about 96.34%, respectively. The results demonstrated that the electrokinetic removal of TMs in citricacid enhanced EKSR was about 5-6 times more when unenhanced EKSR process.

In Exp.3, the soil was saturated by 0.1M citric acid and catholyte pH controlling using 0.1M of EDTA solution were performed. The TMs migration were more significant due to the catholyte conditioning during EKSR treatment due to easy desorption of metal ions onto the soil surface to pore water under acidic environment. From **Fig. 2(c)**, in Exp.3, the percentage removal of TMs for chromium about 76.52%, for cobalt about 97.16%, for nickel about 93.04%, for copper about 87.48%, for zinc about 91.02% and for manganese about 64.07%, respectively. The results demonstrated that the electrokinetic removal of TMs in citricacid enhanced EKSR was about 5-6 times more than the unenhanced EKSR process.

In Exp.4, the soil was saturated by the double distilled water with anolyte pH controlling using 0.1M of NaOH solution was performed. The concentration profiles of TMs are rapidly decreased in the soil. After 20d of the operation, as seen in **Fig. 2(d)**, in Exp.4, the percentage removal of TMs for chromium about 7.48%, for cobalt about 11.26%, for nickel about 19.67%, for copper about 9.14%, for zinc about 13.28% and for manganese about 24.26%, respectively. The results demonstrated that the electrokinetic removal of TMs in the alkali (NaOH)-enhanced EKSR was about 1.5 times more than the unenhanced EKSR process. The overall results demonstrated that the electrokinetic removal of TMs in chelatingagents enhanced EKSR was about 6-7 times when unenhanced process.

As seen in Exp.1(unenhanced) the average removal efficiency of six TMs were observed from soil cell and the combination of the anolyte and catholyte (A+C) flow after 20 days of treatment, for Cr(VI), Co^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} were about 4.86%, 8.62%, 13.04%, 16.7%, 10.04%, and 25.53%, respectively. The removal performance of Cu^{2+} , Zn^{2+} and Mn^{2+} were more than those of Cr(VI), Co^{2+} , and Ni^{2+} . It might be the reason that high acidic environment at anode compartment in the electrokinetic reactor was more favourable for the migration of Cu^{2+} , Zn^{2+} and Mn^{2+} due to their high initial concentration in the dump soil. In the Exp.2, the average removal efficiency of TMs were observed from soil cell and the anolyte and catholyte flow after 20 days of treatment, as for Cr(VI), Co^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} were about 81.40%, 89.9%, 58.51%, 81.84%, 56.9% and 83.03% respectively. In the Exp.3, the average removal efficiency of TMs were observed from soil cell and anolyte and catholyte flow after 20 days of treatment, as for Cr(VI), Co^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} were about 83.92%, 98.58%, 96.52%, 86%, 76.29% and 65.25% respectively. In the Exp.4, the average removal efficiency of TMs were observed from soil cell and the anolyte and catholyte flow after 20 days of

treatment, as for Cr(VI), Co^{2+} , Ni^{2+} , Cu^{2+} , Zn^{2+} and Mn^{2+} were about 5.15%, 18.58%, 28.61%, 12.45%, 15.22% and 21.64% respectively. It is observed that, the citric acid/EDTA-metal complexes moved toward cathode by electroosmosis and free metal cations moved toward the cathode by the electromigration.

3.3. Final Risk Indices and Contamination Levels after EKSR Treatment

We estimated the risk indices and contamination levels after CA-EKSR process. The final pollution index (PI) values were determined for all six TMs after treatment in chelating agents enhanced EKSR experiments and shown in **Fig.3**. The $\text{PI} < 3$, for all TMs in Exp.2 (Citric acid enhanced EKSR) and Exp.3 (EDTA enhanced EKSR) than Exp.1 (Unenhanced EKSR) and Exp.4 (Alkali EKSR), which indicated that the treated soil has low level of contamination.

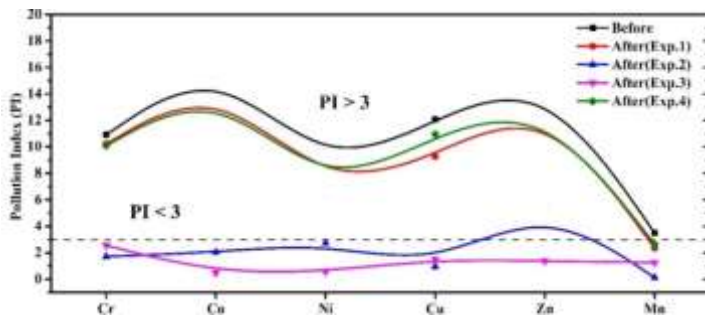


Fig. 3. Final Contamination Levels after EKSR Treatment

The final geo-accumulation index (I_{geo}) values were determined for all six TMs after treatment in chelating agents enhanced EKSR experiments and shown in **Fig. 4**. The treated soil had I_{geo} values lies in $2 < I_{\text{geo}} < 3$, which indicated that soil contamination levels were reduced during chelating agents enhanced EKSR process particularly in citric and EDTA as enhancing agents.

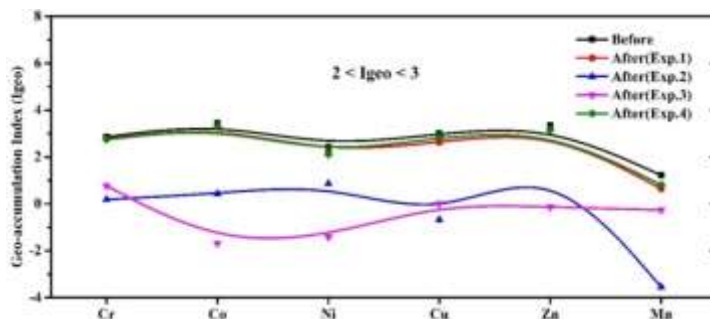


Fig.4. Final Geo-accumulation Index (I_{geo}) after EKSR Treatment

The final ecological risk index (E_r) values were determined for all six TMs after treatment in chelating agents enhanced EKSR experiments and shown in **Fig. 5**. The treated soil has $E_r < 40$, which indicated that the soil had low ecological risk after treatment particularly, in Exp.2 and 3 as citric and EDTA as enhancing agents.

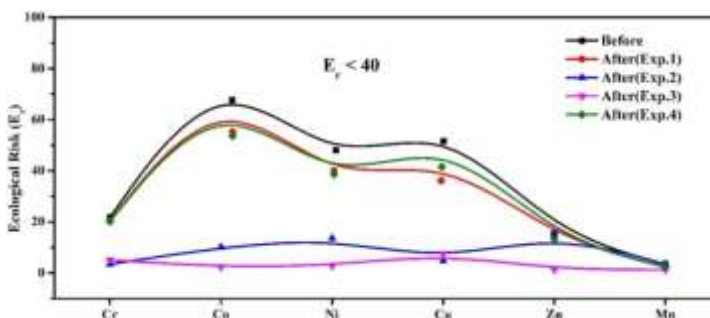


Fig.5. Final Ecological Risk Index (E_r) after EKSR Treatment

The overall risk index (RI) values were determined after treatment in chelating agents enhanced EKSR experiments and shown in Fig. 6. It is observed that the RI < 150 in case of citric acid and EDTA enhanced EKSR Exp.2 and 3, which indicated that the soil had low environmental risk after treatment.

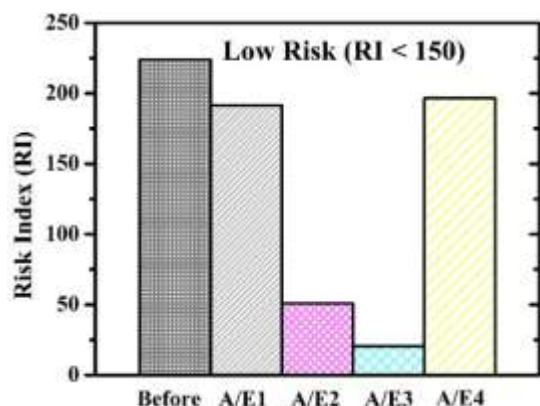


Fig.6. Final Risk Index (RI) after EKSR Treatments

4. Conclusion

In this study, the removal performance of toxic metals (TMs) was found in chelating agents (citric acid and EDTA) enhanced electrokinetic soil remediation (CA-EKSR) treatments about 5-6 times more than when unenhanced treatment. The pollution index (PI) < 3, for all TMs in the case of citric acid and EDTA enhanced than unenhanced EKSR treatment, which indicated that the treated soil has a low level of pollution. In case of geo-accumulation index (I_{geo}), the treated soil has I_{geo} values lying in $2 < I_{geo} < 3$, which indicated that soil contamination levels were reduced after CA-EKSR treatments. The treated soil has $E_r < 40$, which indicated that the soil has low ecological risk after treatment particularly, in citric and EDTA enhancing EKSR treatments. Moreover, the overall ecological risk index (RI) was reduced from 224 to 35.6 by means of RI < 150 of CA-EKSR treated soil, which indicated that the soil has low environmental risk after treatment. The environmental risk levels and contamination levels were reduced under CA-EKSR treatments than unenhanced treatment. It is observed that the CA-EKSR treatments were effective in the remediation and mitigation of TMs from granite mining waste particularly in dump soils. The study is useful for the researchers to investigate the soil quality, contamination levels and ecological risk assessments (EIA) particularly in the field of soil decontamination studies.

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