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Recovery of Iron-Based Coagulant from Waste Laterite Ore through Chemical Leaching for Efficient Water Treatment / Bawani, M.R., Rajput, A.A., Gadhi, T.A., Sheikh, Z., Ansari, K., Ahmed, Z., Chalgri, S.R., Bonelli, B.. - In: ACS OMEGA. - ISSN 2470-1343. - 11:2(2026), pp. 2431-2440. [10.1021/acsomega.5c01985]

Availability:
This version is available at: 11583/3014801 since: 2026-08-24T11:11:07Z

Publisher:
American Chemical Society - ACS

Published
DOI:10.1021/acsomega.5c01985

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14 October 2026

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Article

Recovery of Iron-Based Coagulant from Waste Laterite Ore through Chemical Leaching for Efficient Water Treatment

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Cite This: *ACS Omega* 2026, 11, 2431−2440

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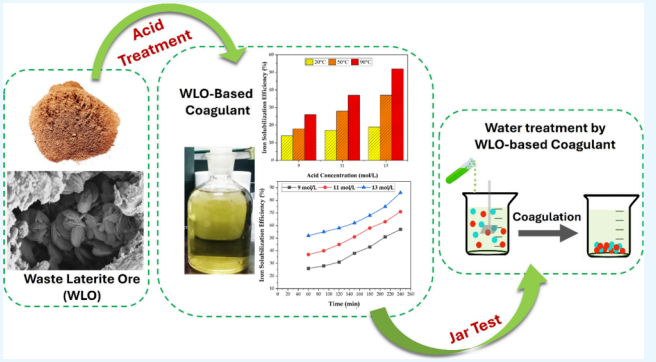
ABSTRACT: Open dumping and disposal of waste iron ore in freshwater and landfills are increasing environmental concerns. Therefore, following the environmental sustainability and circular economy approach, this study recovered iron-based coagulants from waste laterite ore (WLO) and assessed their performance in water treatment. Chemical leaching of WLO was performed using HCl with varying acid concentrations, temperatures, and treatment times. The optimum iron solubilization efficiency of 86% was achieved at an optimized HCl concentration of 13 mol/L, a reaction temperature of 90 °C, a mixing speed of 500 rpm, and a treatment time of 240 min. The recovered WLO-based iron coagulant exhibited a greenish color and showed promising performance in treating water from the River Indus Canal, particularly in removing turbidity and heavy metals. This performance of the WLO-based iron coagulant was nearly identical to that of conventional ferric chloride. Overall, recovering iron-based coagulants from WLO may reduce the cost of coagulating drinking water or wastewater. Furthermore, the present study's findings will be useful in protecting the environment by either not discharging the WLO into freshwater bodies or by dry stacking.



1. INTRODUCTION

Pakistan contains an estimated 430 MT of iron reserves.[1,2] However, a considerable amount of waste ore is generated during the mining operations.[3,4] These waste materials are openly dumped near mining fields or disposed of in the nearest freshwater body.[5] After the processing stage, disposing of mining waste ore material into the water body is worrisome. On the other hand, converting and reusing these waste materials into valuable products has gained significant importance in recent years to make the mining industry more sustainable, with better economic, social, and environmental outcomes.

The laterite deposits in Jhimpir, Thatta district, Pakistan, are exploited by conventional surface mining.[6−8] An estimated 7−8 tons of waste laterite ore (WLO) is generated during the processing stage. The disposal of WLO is putting an extra cost burden on the processing industry. At the same time, current disposal methods also increase the major environmental concerns regarding air and water pollution. The conversion of waste iron ore into valuable products, the reduction of waste ore and dry disposal of waste materials are under consideration for its safe disposal.[9] Recently, many studies reported the use of waste iron ore in the construction of civil engineering projects in the form of concrete compounds and cement materials[10−12] as road construction material,[13] as a pigment, and used in the ceramic industry.

Coagulation and flocculation are primary treatments that enhance overall treatment efficiency in water and wastewater treatment, but they also incur high associated costs. Various coagulants, including aluminum-based, iron-based, and natural coagulants, are utilized in water and wastewater treatment. Compared to aluminum-based coagulants, iron-based coagulants, such as ferric chloride, have a negative impact on sludge dewaterability and odor emission.[14] Nevertheless, they are widely used in water and wastewater treatment.[15] However, iron-based coagulants have a high cost, ultimately increasing the water and wastewater treatment costs. It will be economically beneficial to filter and treat raw water from rivers or canals for drinking purposes, such as in the River Indus in Pakistan, which heavily relies on coagulation for its municipal and drinking water supply in major urban cities.[16]

Received: March 3, 2025
Revised: December 24, 2025
Accepted: December 25, 2025
Published: January 5, 2026

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Therefore, recovery of iron-based coagulants from WLO could appear as a sustainable solution, lowering the cost of coagulants.

Leaching is a chemical process used to extract valuable metals from ores that contain low concentrations of desired elements through the percolation of a specific leaching solution. Various acidic and basic solutions are generally used to obtain desired elements.[17−20] The leaching efficiencies are influenced by various factors, such as the type of leaching solution, solution concentration, reaction temperature, mixing intensity, reaction time, and reactor design.[21,22]

Various attempts have been made to recover iron from the ore and tailings. Alafara et al[23] studied the chemical and biological leaching of iron ore and analyzed various factors, including mixing, acid dosing, time, and temperature. Another study also reported the chemical leaching of an iron-based ore (a blend of limonite and saprolite) using HCl at different concentrations to recover Fe, Ni, and Mg.[24] Furthermore, another study reported 93% Fe recovery from iron tailings using acid leaching.[25] These reported factors are important in recovering iron-based coagulant and their sustainable utilization in water treatment systems to promote waste resource recovery and circularity.

The literature already reports the recovery of iron-based coagulant from iron ore tailings and its utilization for water treatment.[26] However, the impact of various factors, different types of ore and waste material for iron recovery and its subsequent usage for water treatment still needs to be explored. Furthermore, iron recovery from WLO may also provide new insights into chemical leaching from waste. Moreover, iron recovery from WLO and its utilization in water treatment could be a sustainable practice for managing waste materials.

Previous studies have used laterite as a natural coagulant in water treatment, particularly in treating landfill leachate[27] and removing fluoride from contaminated drinking water.[28] However, this study introduced optimized methods for recovering iron-based coagulant as a liquor from WLO through the chemical leaching process by optimizing the various variables such as acid concentrations, reaction temperatures and treatment time. Furthermore, another objective of this study was to assess the effectiveness of recovered WLO-based iron coagulants on raw water treatment.

2. MATERIALS AND METHODS

Laterite surface mining in Jhampir, Thatta District, Pakistan, produces significant amounts of waste laterite ore. After mining, the laterite ore is passed through an 8 mm screen. The material below 8 mm is considered undersized and discarded as waste. An estimated 7−8 tons/day of waste laterite ore is generated. This WLO was collected from the mining site, located at coordinates 25°04′18”N and 68°05′42”E. This study consisted of two separate laboratory-based experiments. In the first part, the chemical leaching of WLO was performed under various conditions to optimize the parameters during the leaching process. The second part analyzed the effectiveness of the obtained product in water treatment experiments. For leaching experiments, a lab-grade Merck HCl was used.

2.1. Preparation of Raw Material

The particle size of the WLO is less than 8 mm, which is quite coarse for the leaching experiment. Therefore, the collected WLO samples were fed to the disc crusher, and a fine powder WLO was prepared to increase the surface area, consequently enhancing the surface reactivity of metals during leaching. The particle size distribution analysis of the prepared WLO was made using a Horriba LA-300 Particle Size analyzer. Additionally, 1 mL of sodium hexametaphosphate (1 mol/L solution) was added to prevent particle coagulation.[29] The ground product was placed in airtight bags until used.

2.2. Chemical Leaching Experiment

Chemical leaching experiments of WLO were performed with 30 g of WLO and 500 mL of prepared leaching solution (HCl acid and deionized water). To optimize different parameters of the leaching experiments, in the first part of the study, the effect of seven concentrations of HCl, viz 3, 5, 7, 9, 11, and 13 mol/L, was assessed on the leaching of WLO for 60 min at room temperature with 500 rpm. Prior studies have reported the effective dissolution of iron from laterite using HCl in the 1−6 mol/L range.[30,31] Since the material is waste and contains impurities, the HCl concentration was extended to 13 mol/L. After optimizing the acid concentration, the optimal concentrations were used to investigate the effect of different reaction temperatures, i.e., 20, 50, and 90 °C.[31,32] The leaching times were studied after optimizing the HCl concentration and reaction temperature at a fixed mixing speed, i.e., 500 rpm. The assessed chemical leaching experiment times were 60, 90, 120, 150, 180, 210, and 240 min.[31,32] After the leaching step, the liquor (a WLO-based iron coagulant) was filtered to get an impurity-free product, and the solid residue was preserved as a byproduct. The iron solubilization Eefficiency (ISE) of each experiment was evaluated to determine the effectiveness of acid leaching in recovering iron from WLO. The ISE was calculated using eq 1.

$$\mathrm{ISE}(\%) = \frac{C_f}{C_i} \times 100 \tag{1}$$

Where the ISE is iron solubilization efficiency (%), C_i is the initial iron concentration (%) in WLO, and C_f is the iron concentration (%) in liquor (WLO-based iron coagulant) produced after acid leaching. C_f is also known as the absolute iron concentration (%). The mass balance equation for the iron concentration in the leaching step is described in eq 2.

$$C_i = C_f + C_{SR} \tag{2}$$

where C_i and C_f were described earlier, however, C_{SR} is the iron concentration in the solid residue left after the acid leaching, which was calculated by the difference method.

2.3. Jar Test Experiment

Experiments were performed using the conventional jar test apparatus, which consisted of six mixing paddles connected to a power supply panel attached to the motor of a Chicago, USA-based company, model 60618, with a power requirement of 24 V, 2.6 A, 1/18 HP, and a rotation speed of 417 rpm. Six 1-L breakers were used with each 1 L of raw water collected from the KB feeder canal at the River Indus to assess the WLO-based iron coagulant (WLO-IC) performance in water treatment. Additionally, an industrial-grade ferric chloride (40%), a product of Guangxi Yukexin Industrial Co., Ltd., was used for comparative analysis and referred to as conventional ferric chloride (CFC). Referring to the APHA water treatment procedure,[33] a standard solution of different doses, i.e., 5, 10, 20, and 30 ppm, was prepared from the produced WLO-IC and CFC for the Jar test. After the dose addition, the solution was rapidly mixed for 1−2 min, and the speed was increased to

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30−40 rpm for the next 15−20 min. Subsequently, the mixing was stopped, and the samples were left undisturbed without agitation for 60 min to allow the solids to settle. The raw water and treated water were analyzed for pH, total dissolved salts (TDS), color, turbidity, electrical conductivity (EC), and various elements (Al, As, Pb, Mn, Cu, Cd, Cr, Zn, PO_4, Mg, Fe, Ca, K, Na, Co, Li, B, Ba, Ni). The mass balance of Fe% in the Jar test was obtained from the following equation,

$$C_{(\mathrm{WLO-IC}),\,\mathrm{sol}} + C_{\mathrm{RW}} = C_{\mathrm{TW}} + C_{\mathrm{flocs}} \quad (3)$$

where, $C_{(\mathrm{WLO\text{-}IC}),\,\mathrm{sol}}$ is the iron concentration (mg/L) measured in the prepared solution of WLO-IC for the jar test, C_{RW} iron concentration (mg/L) measured in the raw water, C_{TW} is the iron concentration (mg/L) measured in the treated water after the jar test, C_{flocs} is the iron concentration (mg/L) in the flocs settled after the jar test. This value (C_{flocs}) was calculated by the difference method.

2.4. Analytical Methods

The particle size distribution of powder WLO was determined using the Horiba LA-300 particle size analyzer. An Olympus Vanta X-ray fluorescence (XRF) analyzer was used to determine the elemental composition of WLO. The morphology of WLO was studied using a scanning electron microscope (SEM, Zeiss Sigma 500 VP). The specific surface area of WLO was obtained by applying the Brunauer−Emmett−Teller (BET) method to N_2 isotherms measured at −196 °C. PANalytical X'Pert Pro X-ray diffractometer (XRD) was used to investigate the mineral phases before and after leaching under optimal conditions. The software HighScore Plus (v. 6.0) and the Crystallography Open Database (COD) were used for peak identification and automated search−match to analyze the results of the diffraction patterns, and as well as the semiquantitative analysis. ICP-OES 5100 VDV AGILENT was used to determine the Fe content of the WLO-based iron coagulant produced from each leaching experiment and the elemental analysis of raw and treated water samples. Simultaneously, a TDS probe meter (Hanna), a pH meter (Hanna) and a Lovibond meter for turbidity were used. The fluoride concentration before and after coagulation-jar tests was determined using the SPADNS colorimetric method with a UV−Vis spectrophotometer. The chloride concentration was measured through argentometric titration using a standard silver nitrate solution with a potassium chromate indicator, in accordance with the APHA Standard procedure.[33]

2.5. Statistical Analysis

All leaching experiments and analyses were performed in triplicate to minimize random errors and ensure the data represent consistent trends. ANOVA was also performed to find the significant differences between the leaching conditions and Jar tests results. ANOVA analyses were performed using the SPSS-16 software package.

3. RESULTS AND DISCUSSION

3.1. Characterization of Waste Laterite Ore

The particle size range of prepared WLO (Figure 1) i.e., 0.766 to 394 μm, with a D_{50} of 79.54 μm and a D_{90} of 162.71 μm. It reveals that most particles have (fine) average sizes below 100 μm. Fine particle sizes generally enhance metal dissolution due to increased surface area, facilitating better contact with the leaching agent.[34−30] Studies show that finer particles lead to higher extraction rates of metals, including iron, due to more efficient acid penetration and reaction.[19,36] Table 1 highlights the fundamental characteristics of WLO. The prepared WLO for treatment has a surface area of 11.32 m^2/g, similar to that of iron ore tailings reported in the literature.[26] The elemental analysis revealed that WLO is rich in silicon, followed by iron and alumina, indicating the presence of silicate and iron-bearing minerals.[17]

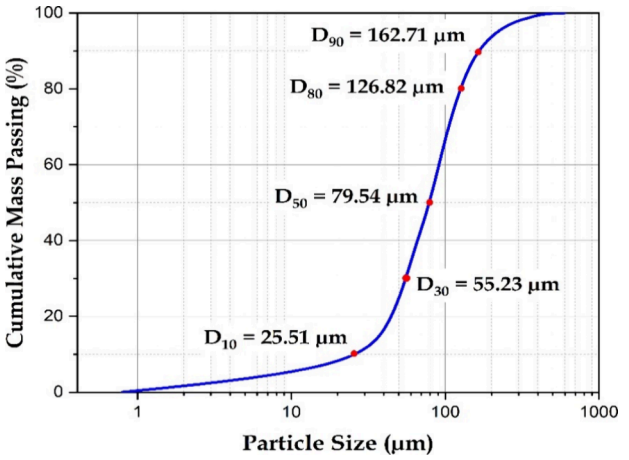

Figure 1. Particle size distribution of the prepared WLO.

Table 1. Relevant Characteristics of WLO

Property	WLO
surface area (m^2/g)	11.32
elemental analysis (%)	
Si	42.4
Fe	21.3
Al	1.32
Ti	0.09
Mn	0.01
Mg	0.047
Sr	0.012
Ca	0.02
Zn	0.022
S	0.18
LE[a]	remaining

[a]LE = light elements.

The SEM morphological analysis of laterite ore waste (Figures 2A and 2B) reveals that it is highly porous and rough, exhibiting needle-shaped particles and agglomerates of ultrafine particles (Figure 2B), as well as flaky structures within the pores (Figure 2A). These characteristics enhance surface area and acid penetration, thereby improving leaching efficacy for metal extraction, as validated by Wahab et al.[37] The needle shape and flaky structures are typical of clays or iron-rich oxides in lateritic ores[38] and refer to the roughness and porosity increase surface reactivity.[39] The agglomerates of ultrafine particles in Figure 2B represent grinding effects,[40] which enhance the reactive surface sites.[41] Figure 2 exhibits morphological evidence suggesting laterite ore waste has the potential for sustainable resource utilization.

3.2. Effect of Acid Concentrations on Leaching

The ISE and absolute iron concentration (C_f) as a function of different HCl concentrations from WLO is shown in Figure 3. Various studies have reported that the acid concentration is

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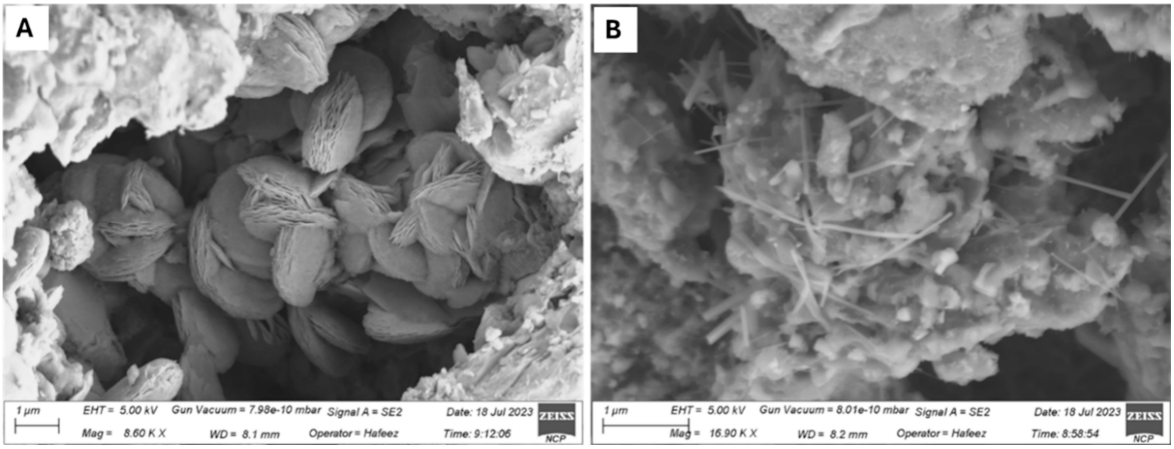

Figure 2. SEM images of WLO: (A) flaky structures present within the pores, and (B) needle-shaped and ultrafine agglomerates on the surface of particles.

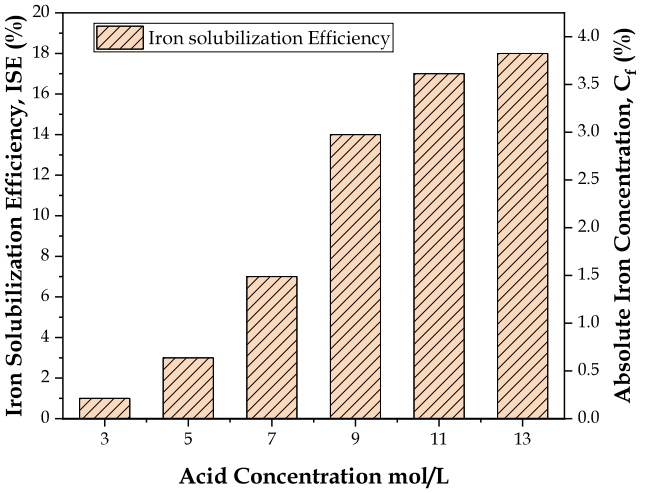

Figure 3. Effect of acid concentration on ISE and C_f at room temperature.

crucial in optimizing the leaching recovery of iron ore.[34] The leaching of WLO was performed at different HCl acid concentrations, i.e., 3, 5, 7, 9, 11, and 13 mol/L at constant experimental times of 1 h at room temperature. Figure 3 shows the increase in ISE as the HCl concentration increased. The highest ISE of 18% was achieved at a 13 mol/L HCl concentration. It was lowest at the HCl concentration of 3 mol/L with just 1%. It shows that higher acid (HCl) concentrations will also increase the dissolved percentage of iron. The increase in ISE was also reported in previous studies as the HCl concentrations increased.[42] Previous studies on laterite deposits from the same region have reported highly heterogeneous mineralogical characteristics, with finely distributed siliceous and alumina-based mineral phases.[7] Since the material investigated in this study represents the waste fraction generated while mining laterite deposits, its mineral composition is expected to be even more complex. Consequently, such waste laterite ore requires more intensive activation and stronger leaching conditions to extract iron effectively.

3.3. Effect of Reaction Temperature on Leaching

To assess the extraction of iron solutions at different temperatures, agitation leaching experiments were performed at 20, 50, and 90 °C. The role of reaction temperature during the leaching of WLO was assessed and presented in Figure 4.

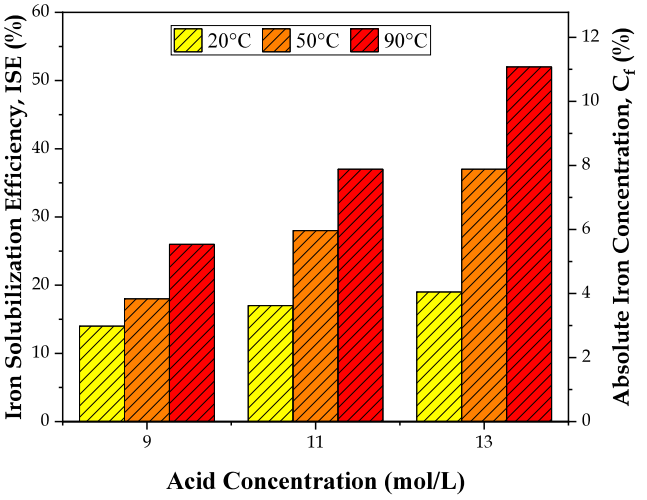

Figure 4. Effect of variable temperatures on ISE and C_f.

In the experiment with three optimum HCl acid concentrations, i.e., 9, 11, and 13 mol/L, the obtained ISE values were 14, 17, and 18%, respectively, during the leaching process at a reaction temperature of 20 °C. However, when the reaction temperature of the leaching process was 50 °C, the ISE values were 18, 28 and 37%, respectively. The increase in ISE was further observed during the leaching experiment, which was performed at 90 °C. At 90 °C, the ISEs reached 26, 37 and 52%. During leaching experiments, the ISE also increased as the reaction temperature increased. These results align with those of the previous study, indicating that increased reaction temperature enhances leaching efficiencies.[43,44] This work achieved an optimum 52% ISE at an acid concentration of 13 mol/L HCl and a temperature of 90 °C.

The ANOVA analysis shows that the ISE is significantly enhanced ($p < 0.05$) due to different temperatures and acid concentrations. However, these findings on the temperature-related leaching studies agree with previous leaching studies of limonitic laterites at 95 °C.[45] The reaction temperatures played a vital role in increasing the ISE during the leaching process

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because, as the temperature increased, the ISE also increased, as shown in Figure 4.

3.4. Effect of Reaction Time on Leaching

Figure 5 illustrates the impact of HCl acid concentration and reaction time on ISE at constant temperature, i.e., 90 °C. Iron

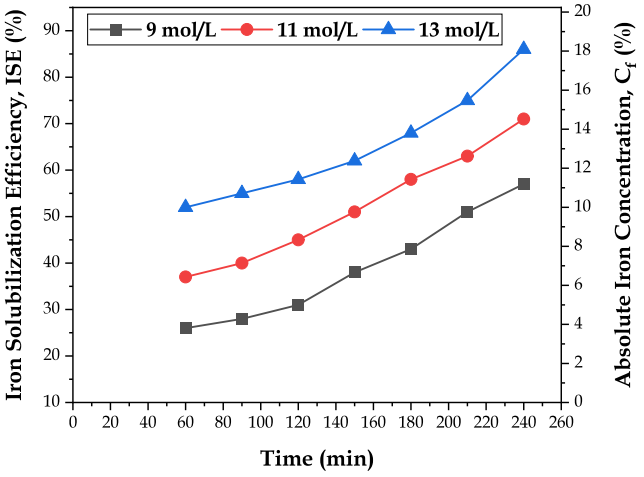

Figure 5. Effect of treatment time on ISE and C_f.

solubilization increases with higher HCl acid concentrations, as the curve for 13 mol/L consistently shows the highest ISE, followed by 11 and 9 mol/L. This trend suggests increased acid concentrations enhance iron solubility, perhaps due to higher chemical reactivity.

Furthermore, ISE consistently increases over time at all acid concentrations, showing an almost linear trend. At the end of the reaction period (240 min), the maximum ISEs achieved are about 86% for 13 mol/L, 71% for 11 mol/L, and 57% for 9 mol/L. The ISE significantly enhanced ($p < 0.05$) due to different treatment times and acid concentrations, as indicated by ANOVA analysis. The results highlight the significant influence of acid strength and reaction time on the efficacy of iron dissolution, providing essential insights for improving leaching processes in WLO treatment. It was observed that the WLO-based iron coagulant was greenish in color and got darker with increasing reaction time.

3.5. Leaching Mechanism

The optimal ISE of 86% was attained at a concentration of 13 mol/L HCl, a temperature of 90 °C, and a treatment time of 240 min, representing the most effective leaching conditions for WLO (Figure 5). The mass balance of iron content under optimal conditions was calculated using equation 2. The iron content in liquor (WLO-IC) was determined experimentally as 18.32% (Figure 5), while the corresponding iron content in the solid residue (SR-13 mol/L;13 mol/L, 90 °C, 240 min) was calculated to be 2.98%. The WLO and SR-13 mol/L were characterized to determine the mineral dissolution mechanism before and after acid leaching. Figure 6 shows the presence of quartz, kaolinite, goethite and hematite minerals in WLO and SR-13 mol/L samples, similar to the most common minerals found in laterite globally.[17,46,47] In WLO, the goethite peaks were detected at 2θ of 21.19°, 33.15° and 17.74°, respectively. These peaks diminished substantially after leaching, indicating that a significant portion of the goethite was dissolved under acidic conditions, as previously reported.[17,34] The goethite dissolved due to lower crystallinity, hydrated nature and high

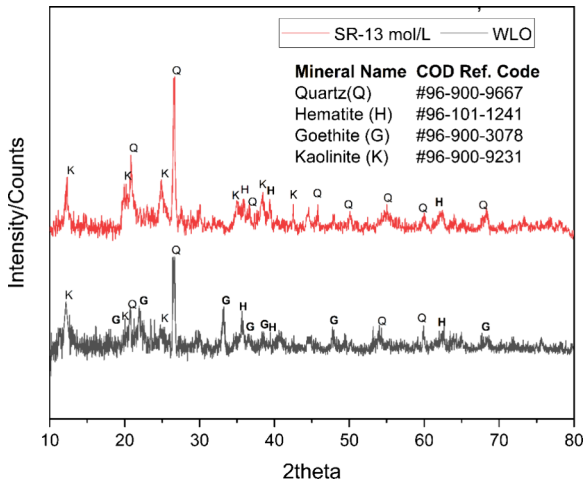

Figure 6. XRD analysis of waste laterite ore (WLO) and solid residue left after leaching at optimal conditions (SR-13 mol/L).

reactivity.[48,49] Hematite peaks at 35.6°, 39.26°, and 62.39°, with low intensity counts, revealing the presence of a smaller amount in WLO. After leaching, the hematite loses the peak intensity at 35.6°, suggesting partial dissolution.[17] The hematite is highly crystalline compared to goethite and dissolves slowly.[17,49,34] However, finer particle size can improve the hematite dissolution.[50] Kaolinite and quartz are major mineral phases in WLO (Figure 6). Quartz peaks were detected at 20.84°, 26.62° and 59.92°, respectively. However, the quartz relative peak intensity at 26.62° appears higher after leaching due to the selective dissolution of other phases, mostly iron-bearing minerals (goethite and hematite), as shown in Figure 6. Also, more peaks of quartz were detected after leaching (SR-13 mol/L) in the region of 35°−70°. In WLO, kaolinite peaks were observed at 12.3°, 20.34° and 24.87°, which became more prominent after leaching (SR-13 mol/L), as kaolinite is more resistant to HCl.[51] The prominent and sharper kaolinite peaks are likely due to the removal of amorphous phases, leading to increased crystallinity or better ordering of the kaolinite layers.[51,52] Apart from the intensity counts, the semiquantitative analysis of SR-13 mol/L (Table 2) indicates an increased in the proportion of quartz and kaolinite, and a decreased in the amount of iron-bearing minerals.

Table 2. Semiquantitative Analysis of Optimized Samples before and after Leaching

			semiquantittative analysis (%)	
sr#	mineral name	highscore code	WLO	SR-13mol/L
1	quartz	96−900−9667	38.4	45.9
2	kaolinite	96−900−9231	32.3	40.8
3	goethite	96−900−3078	16.2	5.1
4	hematite	96−101−1241	13.1	8.2

Overall, the WLO reveals low counts of iron-bearing minerals and high counts of silicate minerals, which is supported by the elemental analysis reported in Table 1. The leaching of WLO under optimal conditions primarily targets iron-bearing phases, with goethite dissolved substantially and hematite partially. Under high HCl acid concentration, heating and prolonged exposure, the Fe−O bonds in goethite and

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Table 3. Analyzed Raw and Treated Water Quality Parameters by WLO-IC (20 ppm) and CFC (10 ppm)[a]

Sr#	parameters	units	raw water, (mean ± S.D)	treated water (WLO-IC), (mean ± S.D)	treated water (CFC), (mean ± S.D)
1	pH	–	6.90 ± 0.05	7.12 ± 0.04	6.80 ± 0.04
3	TDS	mg/L	253 ± 3	212 ± 2	202 ± 2
4	Color	TCU	11.0 ± 0.20	6.2 ± 0.10	5.4 ± 0.10
5	EC	μS/cm	377 ± 5	324 ± 4	338 ± 4
6	turbidity	NTU	25.5 ± 0.40	7.36 ± 0.11	6.78 ± 0.10
7	chlorides	mg/L	37 ± 0.50	39 ± 0.50	41 ± 0.50
8	Fe	mg/L	0.060 ± 0.002	0.272 ± 0.004	0.226 ± 0.004
9	Al	mg/L	0.010 ± 0.00	0.010 ± 0.000	0.010 ± 0.000
10	Pb	mg/L	0.006 ± 0.000	0.006 ± 0.000	0.006 ± 0.000
11	Mn	mg/L	0.0020 ± 0.0002	0.086 ± 0.002	0.209 ± 0.004
12	Cu	mg/L	0.0050 ± 0.0002	0.0060 ± 0.0002	0.0050 ± 0.0002
13	Cd	mg/L	0.0011 ± 0.0001	0.0009 ± 0.0001	0.0006 ± 0.0001
14	Cr	mg/L	0.020 ± 0.001	0.030 ± 0.001	0.030 ± 0.001
15	Zn	mg/L	0.012 ± 0.0002	0.006 ± 0.0002	0.007 ± 0.0002
16	PO_4	mg/L	0.039 ± 0.001	0.030 ± 0.001	0.031 ± 0.001
17	Mg	mg/L	8.289 ± 0.09	8.150 ± 0.08	7.507 ± 0.07
18	Ca	mg/L	11.249 ± 0.07	12.360 ± 0.08	12.541 ± 0.08
19	K	mg/L	1.541 ± 0.02	2.905 ± 0.025	3.204 ± 0.025
20	Na	mg/L	23.164 ± 0.16	20.168 ± 0.115	20.414 ± 0.115
21	As	mg/L	0.0013 ± 0.0001	0.0014 ± 0.0001	0.0090 ± 0.0002
22	Co	mg/L	0.0050 ± 0.0001	0.0040 ± 0.0001	0.0040 ± 0.0001
23	Li	mg/L	0.0050 ± 0.0002	0.0050 ± 0.0002	0.0020 ± 0.0001
24	B	mg/L	0.010 ± 0.0002	0.001 ± 0.0001	0.010 ± 0.0002
25	Ba	mg/L	0.014 ± 0.0002	0.009 ± 0.0002	0.008 ± 0.0001
26	Ni	mg/L	0.009 ± 0.0002	0.012 ± 0.0002	0.003 ± 0.0001
27	F	mg/L	0.64 ± 0.01	0.27 ± 0.01	0.24 ± 0.01

[a]SD = Standard deviation.

hematite break down, releasing Fe^{3+} ions, which react with chloride ions (Cl^-) to form ferric chloride ($FeCl_3$).[26,53] The XRD results (Figure 6) support the high ISE, i.e., 86% (Figure 5), leaving behind chemically inert quartz and stable kaolinite in solid residue, i.e., SR-13 mol/L (Figure 6). These changes reflect a selective dissolution mechanism of iron-bearing minerals controlled by mineral crystalline, reactivity, and acid solubility.

3.6. Water Treatment Performance

Following the APHA standard procedure,[33] different doses of WLO-IC and CFC were added to the raw water. The optimal coagulant dosage was found to be 20 ppm for WLO-IC and 10 ppm for CFC, when normalized to the Fe content of each coagulant. The difference in dosage can be attributed to the distinct nature of the coagulants, as CFC is a conventional commercial coagulant, while WLO-IC is a complex waste-derived coagulant prepared from WLO. The results obtained from the jar test experiment are summarized in Table 3, showing good coagulation characteristics in removing the turbidity and other contaminants from the raw water. The results of WLO-IC were also compared with CFC, as presented in Table 3. The results showed a pH shift from 6.9 to 7.1 and 6.8 for WLO-IC and CFC, respectively, approaching neutrality and falling within the desired ranges as described by the World Health Organization (WHO). The low dosage of WLO-IC indicated that the acidifying potential was constrained in comparison to the buffering capability of the raw water, which is likely the reason the pH increased somewhat post-treatment rather than remaining acidic. Comparable findings have been recorded in coagulation studies, where the alkalinity alleviates the acidity of metal salt coagulants.[54] The color index decreased from 11 TCU to 6.2 and 5.4 TCU for WLO-IC and CFC (Table 3), and electrical conductivity decreased from 377 μS/cm to 324 and 338 μS/cm from WLO-IC and CFC, respectively, indicating a decrease in the ionic content of the cleared water. Interestingly, the TDS value dropped from 253 mg/L to 212 and 202 mg/L from WLO-IC and CFC, while turbidity decreased significantly from 25.5 NTU to 7.36 and 6.78 NTU from WLO and CFC. Turbidity removal efficiencies from WLO and CFC were 71.2 and 73.5%, respectively, resulting in improved transparency of the treated water sample. A study reported similar results to those of the present study regarding turbidity removal.[55,56] In the reported studies, conventional coagulants such as ferric chloride and ferrate achieved 83% turbidity removal in wet flow conditions[57] and 93.5% turbidity removal from textile wastewater.[58] It reveals that the existing WLO-IC efficiency is close to the conventional coagulant and may be enhanced by further process optimization.

The detailed elemental analysis of the raw and coagulant-treated water revealed considerable reductions in metal concentrations (Table 3). The Al and Pb concentrations remained the same, at 0.01 and 0.006 mg/L, respectively, in the raw and coagulated samples from WLO-IC and CFC. However, slight increases in trace elements such as Cu, Mn, and Cr were observed. The increase in these trace elements might be due to the addition of recovered coagulant from WLO and CFC, as shown in Table 3. However, Cd, Zn, Mg and Co decreased in WLO and CFC. In addition, Phosphate (PO_4) concentrations also decreased slightly after jar testing in both cases of the coagulants tested. The changes in elemental concentrations highlight the chemical interaction of the released Fe ions from the coagulant with other elements in water, as well as the formation of complex ionic flocs, along

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with sand and silt flocs.[59−61] The mass balance of iron content in the jar test was calculated using eq 3 and presented in Table 4. The residual iron concentration in the treated water (C_{TW})

Table 4. Mass Balance of Iron Content in the Jar Test Using WLO-IC and CFC

Sr#	parameters	units	WLO-IC	CFC
1	$C_{(WLO-IC),\ sol}$	mg/L	20	10
2	C_{RW}	mg/L	0.06	0.06
3	C_{TW}	mg/L	0.272	0.226
4	C_{flocs}	mg/L	19.788	9.834

was low in both conditions (0.272 mg/L for WLO-IC and 0.226 mg/L for CFC), indicating that the iron was effectively removed from the solution. The concentration of iron in raw water was minimal (0.06 mg/L) and did not significantly impact the balance. The results indicate that WLO-IC is effective for floc formation; nevertheless, it requires almost double the amount compared to CFC to achieve equivalent water purity.

The concentration of arsenic (As) was slightly increased in the treated water sample using WLO-IC, but decreased after the CFC treatment, as shown in Table 3. A previous study also showed an increase in As concentration using a ferric chloride coagulant.[56] Ni also increased in the treated water sample due to WLO-IC, which might be due to the presence of Ni in the lateritic ore.[62] Conversely, Li remained the same in raw water and water samples treated with WLO-IC. However, it decreased slightly in the sample treated with CFC. B decreased in the sample treated with WLO-IC, as shown in Table 3. The fluoride concentration in the raw sample was 0.64 mg/L. After the treatment, the fluoride concentration was reduced from 0.47 mg/L to 0.27 mg/L using the WLO-IC and to 0.24 mg/L by CFC. The WHO recommends that drinking water does not exceed 1.5 mg/L of fluoride. WLO-IC and CFC reduced fluorite levels by 57.8% and 62.5%, respectively. The chloride concentration is slightly increased in both the WLO-IC and CFC, i.e., from 37 mg/L in raw water to 39 mg/L in WLO-IC and 41 mg/L in CFC, respectively. The increase in chloride was due to the release of chloride ions from both tested coagulants; however, these levels were found to be under the WHO threshold limits, i.e., less than 250 mg/L. The data shows that WLO-IC is likely to replace traditional coagulants more sustainably. It is often applicable in regions with nearby laterite waste, where cost and environmental concerns play a significant role.

The obtained results highlight the enhanced performance, coagulation, and flocculation characteristics of the recovered iron-coagulant, specifically in terms of suspended particles, turbidity, and the removal of significant metal ions. Nonetheless, a minor increase in trace elements, i.e., Cu and Cr, treated with WLO-IC, indicated their origination from the produced iron-based coagulant, which has been documented in the literature for several naturally derived coagulants.[63−27] These trace-level contaminations could be reduced by improving the coagulant's purity and chemical leaching to prevent secondary contamination. Moreover, the exact mechanism of complex chemical and/or ionic interactions, the composition of the ionic flocs, and other related aspects could be studied to upscale the methods and iron-based coagulant recovery from WLO for practical and commercial applications.

4. CONCLUSIONS

This study presents a sustainable and value-added approach for recovering an iron-based coagulant from waste laterite ore (WLO) through acid leaching and its successful application in water treatment. Pakistan's laterite deposits, particularly in Thatta (Sindh), represent a significant iron resource. During mining operations, undersized material (<8 mm) is often discarded, producing 7−8 tons/day of laterite waste. Our study utilized this waste by recovering soluble iron under optimized conditions (13 mol/L HCl, 90 °C, 240 min), achieving an iron solubilization efficiency (ISE) of 86%.

The WLO-based iron coagulant (WLO-IC) was tested for its performance in treating raw water. Compared to conventional ferric chloride (CFC), the WLO-IC coagulant exhibited nearly identical removal efficiencies for several parameters, including turbidity, fluoride, and specific trace metals (such as Mn, Zn, and Cd). WLO requires almost double the amount compared to CFC to achieve the same level of water purity. It highlights the potential of WLO-IC as a technically suitable and eco-friendly alternative in water treatment, promoting circular economy practices and waste-to-resource conversion.

The study focused only on the WLO-IC preparation and did not assess the potential reuse of the solid residue left after leaching. The long-term stability, shelf life, and cost-effectiveness of the WLO-IC compared to CFC were not evaluated.

Future work may also evaluate the blending of WLO-IC with other coagulants to enhance performance under specific treatment scenarios (e.g., industrial wastewater). The solid residue, rich in quartz and kaolinite, has potential applications as an adsorbent in water and wastewater treatment, or as a raw material for geopolymer composites. Future studies should also explore its characterization and reuse potential. Techno-economic feasibility, lifecycle assessment (LCA), and carbon footprint analysis should be carried out to fully establish the environmental and economic viability of scaling up this process.

■ ASSOCIATED CONTENT

Data Availability Statement

All data supporting the findings of this study are included within the article.

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https://doi.org/10.1021/acsomega.5c01985
ACS Omega 2026, 11, 2431−2440

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Funding

This research on finding alternative materials for the Indus River water treatment was performed under the research project funding from the Higher Education Commission, HEDP Centre of Excellence Grants (HEC−COE-37).

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

Authors acknowledge the technical support of Dr. Fahad Irfan Siddiqui, Chairman of the Mining Engineering Department, MUET, Jamshoro, Pakistan, and Prof. Dr. Rasool Bux Mahar, Vice Chancellor, BBSUTSD, Khairpur, Pakistan. The authors would like to express their sincere thanks to Engr. Ashraf Mallah, from the SLCMC Limited, for his valuable assistance in sample acquisition. Special thanks are also extended to Engr. Muhammad Hassan Mangrio, from the Mines and Minerals Department, Government of Sindh, for facilitating access to waste generation data, with the support of the mine supervisor. The Authors duly acknowledge the support of Mr. Mumtaz Tunio, a research assistant at USPCASW, MUET, Jamshoro, Pakistan, in conducting water quality analyses.

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