RESEARCH ARTICLE

Dual-synergistic effects of citric acid on atmospheric leaching of manganese ore

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OBJECTIVES In the current study, the leaching of manganese from Indonesian manganese ores using citric acid as a leaching reagent without the addition of a reducing agent was investigated. Citric acid was used simultaneously as a leaching reagent and a reducing agent due to its reducing power. **METHODS** Several leaching parameters were examined to determine the optimum conditions for citric acid leaching of manganese from these ores. **RESULTS** Low-grade manganese ores from two mining areas in Indonesia contained 36.77% Mn (WK ore) and 12.5% Mn (TG ore), both with different mineral compositions, yet both could be leached using citric acid without an additional reducing agent. The optimum manganese recovery was obtained at approximately 76% and 69% for WK ore and TG ore, respectively, under the conditions of 1M citric acid concentration, 5% pulp density, 150 rpm shaker speed, 30°C leaching temperature, 1-hour leaching period, and <75 µm ore particle size. This condition was also selective for iron recovery leaching. Despite its effectiveness, citric acid leaching presents certain limitations, including relatively high reagent consumption, prolonged leaching duration compared to conventional acid leaching, and potential challenges in reagent recycling. **CONCLUSIONS** Nevertheless, this study demonstrates that citric acid exhibits a dual-synergistic effect as both an effective leaching reagent and a reducing agent for manganese leaching from manganese ores.

KEYWORDS leaching; manganese; low-grade ore; citric acid

1. INTRODUCTION

Manganese is one of the main metal elements that have many benefits, including being widely used in the steel industry, batteries, fertilizers, dyes, dietary, pharmaceuticals, chemical industries, and so on (Lasheen et al. 2009b). Manganese production for the steel industry is usually carried out using the pyrometallurgical method, which is a method of processing manganese ore or other manganese sources using high temperatures in a smelting furnace to produce manganese alloys or ferromanganese as an alloying agent in the steel industry. While manganese products for other needs are generally produced using the hydrometallurgical method which is preceded by a manganese leaching process either from manganese ore as a primary source or from secondary sources of manganese such as spent batteries, ferromanganese smelting slag, and so on (Tian et al. 2010).

The main sources of manganese are manganese ores, that most manganese ores are usually found in the form of manganese dioxide (MnO_2) or pyrolusite ore. Manganese from spent batteries is also in the form of MnO_2. Hydrometallurgical treatment of manganese ores has attracted great attention in recent years. Hence, many efforts have been applied recently to develop a commercial hydrometallurgical process to recover manganese from manganese ores. Extraction of manganese from manganese ores using the leaching method has been widely carried out using the reductive leaching method, namely the acid leaching method with the addition of a reducing agent. Various types of reducing agents have been used in various studies including molasses, oxalic acid, hydrogen peroxide, glucose, pyrite, CaS, iron (II) sulfate, aqueous sulfur dioxide, methanol, ethanol, oxalic acid, tannic acid, sucrose, glucose, lignite, phenol, metallic iron, various types of biomasses such as sawdust, corn cobs and other organic/carbon based reducing agents (Su et al. 2008; Ismail et al. 2004; Xie et al. 2013; Sun et al. 2017; Prasetyo et al. 2019; Tian et al. 2010; Astuti et al. 2019). Organic reducing agents are advantageous due to their biodegradability and lower environmental impact but often require longer reaction times and higher concentrations. In contrast, inor-

ganic reducers are highly efficient but pose safety and environmental hazards (LI et al. 2015; Das et al. 2012).

Among the leaching reagents studied, sulfuric acid is the most widely used due to its effectiveness and cost-efficiency. However, alternative acids such as nitric acid and hydrochloric acid have also been employed (El Hazek et al. 2006; Lasheen et al. 2009b). While sulfuric acid provides high leaching efficiency, it generates sulfate-rich wastewater that requires careful disposal. Nitric acid is highly reactive but expensive, and hydrochloric acid can lead to chlorine gas emissions, posing safety challenges (Pagnanelli et al. 2004; Lasheen et al. 2009a). Citric acid, a weak organic acid, has gained attention due to its dual function as both a leaching reagent and a reducing agent. It has been successfully used for the extraction of various metals, including Ni, Co, Li, and Mn, due to its chelating properties (Astuti et al. 2016a; Santana et al. 2017; Zheng et al. 2020; Punt et al. 2021). Compared to conventional inorganic acids, citric acid offers environmental benefits such as lower toxicity and biodegradability, although its leaching kinetics may be slower and require optimization (Meng et al. 2020).

Several studies have demonstrated the effectiveness of citric acid in the leaching and reduction of manganese. For instance, Meng et al. (2020) reported that citric acid successfully leached manganese from spent batteries with a recovery rate of over 89% under optimized conditions. Astuti et al. (2016a) found that citric acid effectively dissolved manganese from low-grade ores, though reaction times were longer compared to sulfuric acid leaching. Zheng et al. (2020) highlighted the role of citric acid as a complexing agent that enhances manganese solubility by stabilizing Mn(II) ions in solution. Additionally, Punt et al. (2021) demonstrated that citric acid leaching is highly selective, reducing the co-dissolution of impurities, which is advantageous for purification processes. These findings collectively indicate that citric acid has strong potential as an alternative leaching agent for manganese extraction, particularly in environmentally sensitive applications where traditional acids pose risks.

Indonesia possesses substantial manganese ore resources distributed across multiple regions. Despite previous studies exploring citric acid as a leaching agent for manganese extraction, there has been limited research on its effectiveness across different manganese ore mineralogies. The novelty of this study lies in its investigation of the dual-synergistic effects of citric acid as both a leaching reagent and a reducing agent in the atmospheric leaching of Indonesian manganese ores. Unlike previous studies that primarily focused on citric acid as a supplementary reducing agent alongside sulfuric acid, this research evaluates its standalone performance under varying leaching conditions. By assessing manganese dissolution from ores with distinct mineralogical compositions, this study aims to establish citric acid as a sustainable alternative for manganese extraction. Several leaching parameters were investigated to obtain the optimum condition of citric acid leaching of manganese from manganese ores.
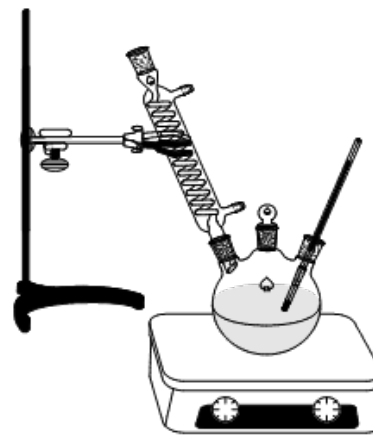
2. MATERIALS AND METHOD

FIGURE 1. Experimental setup for manganese leaching.

2.1 Materials

Manganese ores originated from two mining areas i.e. Way Kanan (WK ore) dan Tanggamus (TG ore) in Lampung Province, Indonesia. The same manganese ores have been used in our previous study (Astuti et al. 2019). The samples (WK ore and TG ore) were mineralogically and chemically characterized. Before characterization, the ore samples were initially dried at 105°C for 24 hours to remove moisture content. The dried samples were then crushed using a laboratory jaw crusher and ground in a ball mill to achieve a particle size of <75 µm. The ground samples were homogenized by mixing thoroughly before further analysis to ensure representative results. X-ray fluorescence (XRF, Epsilon 3XLE, PANalytical, Netherland) was used to determine the chemical compositions of these samples. The mineral phases of the raw ore samples were identified by X-ray diffraction (XRD, X'Pert 3 Powder, PANalytical, Netherland), using CuKα radiation, in the 2θ range 5°to 80°. Scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDS; JEOL, USA) was used to determine the nickel contents of the different minerals by metal mapping.

2.2 Leaching method

To compare the effectiveness of citric acid with other leaching reagents in leaching manganese without the addition of a reducing agent, the leaching experiment began with leaching without the addition of reducing agent using various types of acids namely sulfuric acid, nitric acid, hydrochloric acid, citric acid, and oxalic acid. Each leaching test was conducted under the same operating conditions: acid concentration of 1M, pulp density of 5% w/v (ore weight/acid volume), shaker speed of 150 rpm, leaching temperature of 50°C, and a leaching period of 2 hour. The selection of 1M citric acid concentration was based on prior studies that demonstrated effective manganese dissolution within this concentration range (Astuti et al. 2016b). Additionally, preliminary experiments were conducted to assess the leaching efficiency at different citric acid concentrations, and 1M was found to provide a balance between manganese recovery and reagent consumption.

The experimental setup is shown in Figure 1, consisting of a round-bottom flask on a hotplate stirrer, a condenser to prevent solvent loss, and a thermometer for temperature monitoring. This setup ensures controlled heating and effi-

cient mixing during the leaching process.

Further, the effectiveness of citric acid on the manganese ore leaching was investigated by atmospheric leaching at 150 rpm of shaker speed, various leaching temperatures (30, 50, 70°C), and various leaching periods (15 minutes – 8 hours). The ore particle size was less than 75 µm. Pulp density was kept constant at 5% w/v (ore weight/citric acid volume). Analytical grades of all chemicals (Merck and Sigma-Aldrich, Germany) were used to prepare the leaching solutions. Ultrapure water with a resistivity of 18 ΩM was used throughout this work. Liquid samples after leaching were analyzed by Induced Couple Plasma-Optical Emission Spectroscopy (ICP-OES; Perkin Elmer 8500, Waltham, MA, USA) to calculate metal concentrations in the pregnant leaching solution.

The metal recovery value presented in Figure 5 was calculated using the following equation:

$$\text{Metal Recovery (\%)} = \frac{C_l \times V_l}{C_o \times W_o} \times 100\% \quad (1)$$

where C_l is the metal concentration in the pregnant leaching solution (mg/L), V_l is the volume of the leaching solution (L), C_o is the initial metal content in the ore (mg/g), and W_o is the weight of the ore used in the experiment (g).

3. RESULTS AND DISCUSSION

3.1 Ores characterization

XRF analyses in Table 1 show that manganese ores used have different chemical compositions. WK ore has higher manganese content (36.77%) than TG ore (12.5), while the iron content of TG ore (11.02%) is greater than that of WK ore (6.59%). In addition, Si and Al content in both samples are similar. Figure 2 shows the XRD pattern of WK ore and TG ore. From this figure, it can be seen that the major minerals in WK ore are quartz and pyrolusite (MnO_2), while TG ore contains quartz and Fe_2MnSi (Astuti et al. 2019). Manganese in both samples is incorporated in different minerals. Figures 2 and 3 present the metal mapping of each sample respectively by SEM/EDS analysis. Figure 3 shows that manganese in WK ore is closely associated with oxygen as manganese dioxide (MnO_2), whereas from Figure 4, it can be seen that manganese in TG ore is incorporated with iron.

The effectiveness of manganese leaching using various acids as leaching reagents without the addition of reducing agents is shown in Figure 5. From Figures 5a and 5b, it can be observed that citric acid is the most effective leaching reagent, dissolving manganese up to approximately 62% for both ores under the conditions of a leaching temperature of 50°C, an acid concentration of 1M, a pulp density of 5%, and a leaching time of 2 hours. In contrast, sulfuric acid, nitric acid, hydrochloric acid, and oxalic acid exhibit significantly lower leaching efficiencies in the absence of a reducing agent.

For WK ore, sulfuric acid, hydrochloric acid, and nitric acid each dissolve less than 2% of manganese, while oxalic acid achieves a slightly higher dissolution of approximately 5%. Meanwhile, for TG ore, the dissolution rates are slightly higher, with sulfuric acid, hydrochloric acid, and nitric acid dissolving less than 10% of manganese, while oxalic acid achieves a dissolution of around 15%. Notably, oxalic acid preferentially dissolves iron rather than manganese, as indicated by the higher Fe recovery observed in Figure 5. The greater manganese dissolution observed for TG ore compared to WK ore is attributed to differences in mineral composition, as confirmed by the XRD data (Figure 2). The data indicate that manganese in WK ore is entirely in the form of pyrolusite (MnO_2), which is highly resistant to acid dissolution without a reducing agent. In contrast, TG ore contains manganese in phases other than pyrolusite, which are likely more readily leached in acidic conditions. The low dissolution of pyrolusite in acidic media can be attributed to its high oxidation state (+4) and strong Mn–O bond energy, which makes direct acid attack ineffective without the presence of a reducing agent. According to the Pourbaix diagram, MnO_2 is thermodynamically stable at low pH and oxidizing conditions, necessitating a reducing environment to convert Mn^{4+} to Mn^{2+}, which is soluble in aqueous solutions.

The variations in leaching efficiency among the acids can be explained by their distinct dissolution mechanisms. Citric acid exhibits the highest leaching efficiency due to its dual function as both a leaching agent and a reducing agent. As an organic acid containing carbon, citric acid undergoes partial oxidation, facilitating the reduction of Mn^{4+} (from MnO_2) to Mn^{2+}, which is soluble in aqueous solutions. Additionally, citric acid provides H^+ ions that enhance the dissolution of Mn^{2+} into the leachate. The formation of Mn-citrate complexes further promotes dissolution by stabilizing Mn^{2+} in solution and preventing its reprecipitation.

Oxalic acid is another organic acid widely used as a reducing agent in manganese leaching, typically in conjunction with sulfuric acid. However, in this study, oxalic acid alone was found to be less effective than citric acid. This inefficiency is likely due to the formation of insoluble manganese oxalate (MnC_2O_4), which precipitates and prevents manganese from remaining in the leachate. This is supported by the absence of manganese in the leach filtrate during ICP-OES analysis. Another possible reason for the lower manganese dissolution is that oxalic acid preferentially leaches iron rather than manganese, thereby limiting the availability of Mn in the solution.

The comparative effectiveness of citric acid and oxalic acid, along with other leaching agents, is further summarized in Table 2, which presents the recovery rates and conditions for different metal sources. This table provides a clearer

TABLE 1. Chemical composition of manganese ores (determined using XRF) (Astuti et al. 2019).

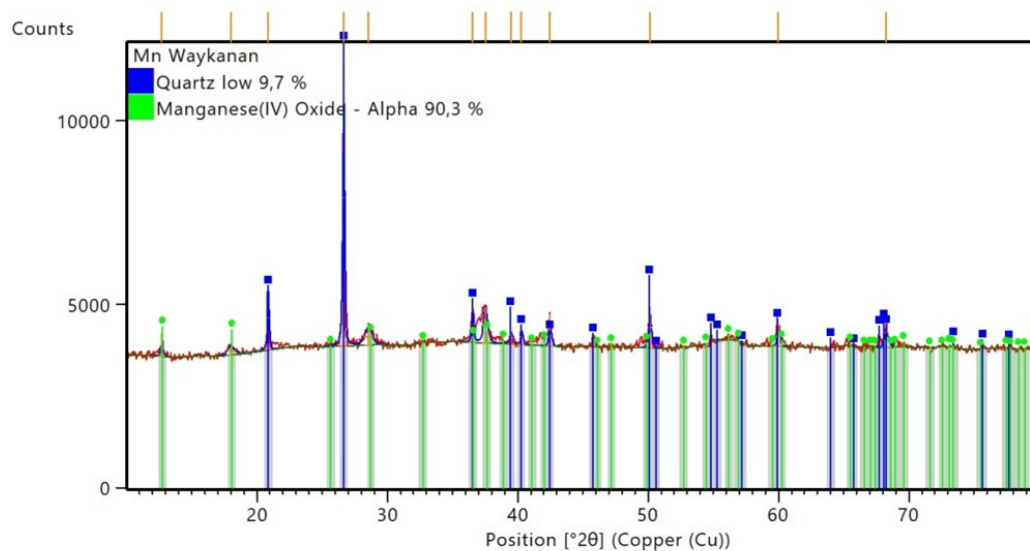| Sample	Elements (wt %)				
	Si	Mn	Fe	S	Al
WK ore	4.13	36.77	6.59	0.31	0.45
TG ore	6.89	12.50	11.02	0.59	0.69

TABLE 2. Manganese recovery from different sources under various leaching conditions.

| Metal Source	Leaching Conditions	Mn Recovery (%)	Description	References
Anodic sludge residue	60°C, solid/liquid ratio 420 g/L, 30 min + reducing agent H2O2	98,5%	Citric acid effectively leached manganese, confirming its potential as a leaching agent.	Kauppinen et al. (2020)
Industrial waste lithium-ion batteries (LIBs)	0.5 mol/L citric acid, S/L ratio 80 g/L, 90°C, 80 min	89%	Citric acid effectively leached Mn along with Li, Co, and Ni under optimized conditions	Meng et al. (2020)
Spent Zn-C and alkaline batteries	2 M citric acid, 80°C, 3 hours	51.32%	Citric acid is more effective in dissolving manganese than malic acid and aspartic acid.	Yuliusman et al. (2018)

insight into how leaching parameters influence manganese recovery, reinforcing the superior performance of citric acid in dissolving manganese under the tested conditions.

3.2 Manganese leaching using citric acid

From previous experiments, it has been proven that citric acid is effective for leaching manganese from manganese ore without the addition of reducing agent. Furthermore, to determine the optimum leaching conditions using citric

(a)
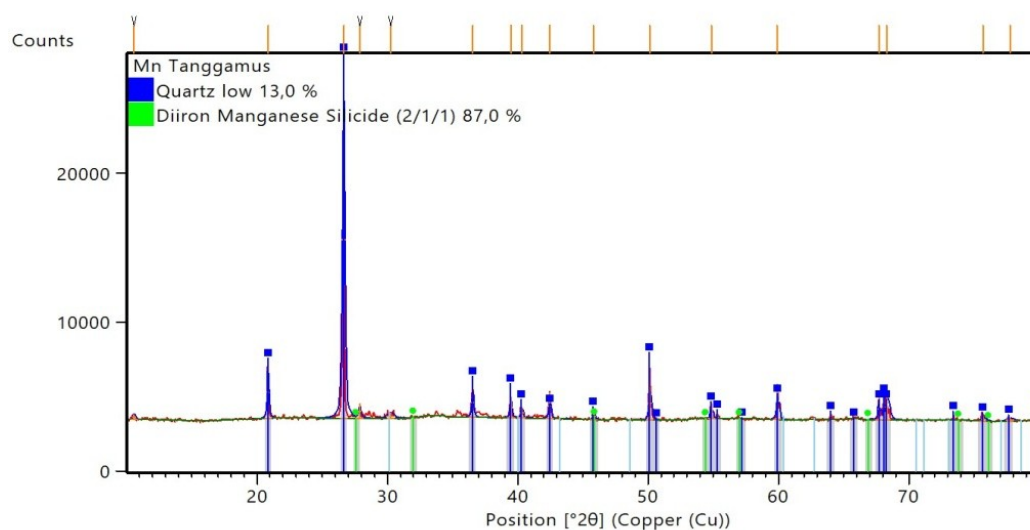
(b)

FIGURE 2. XRD pattern of manganese ores (a) WK ore; (b) TG ore (Astuti et al. 2019).

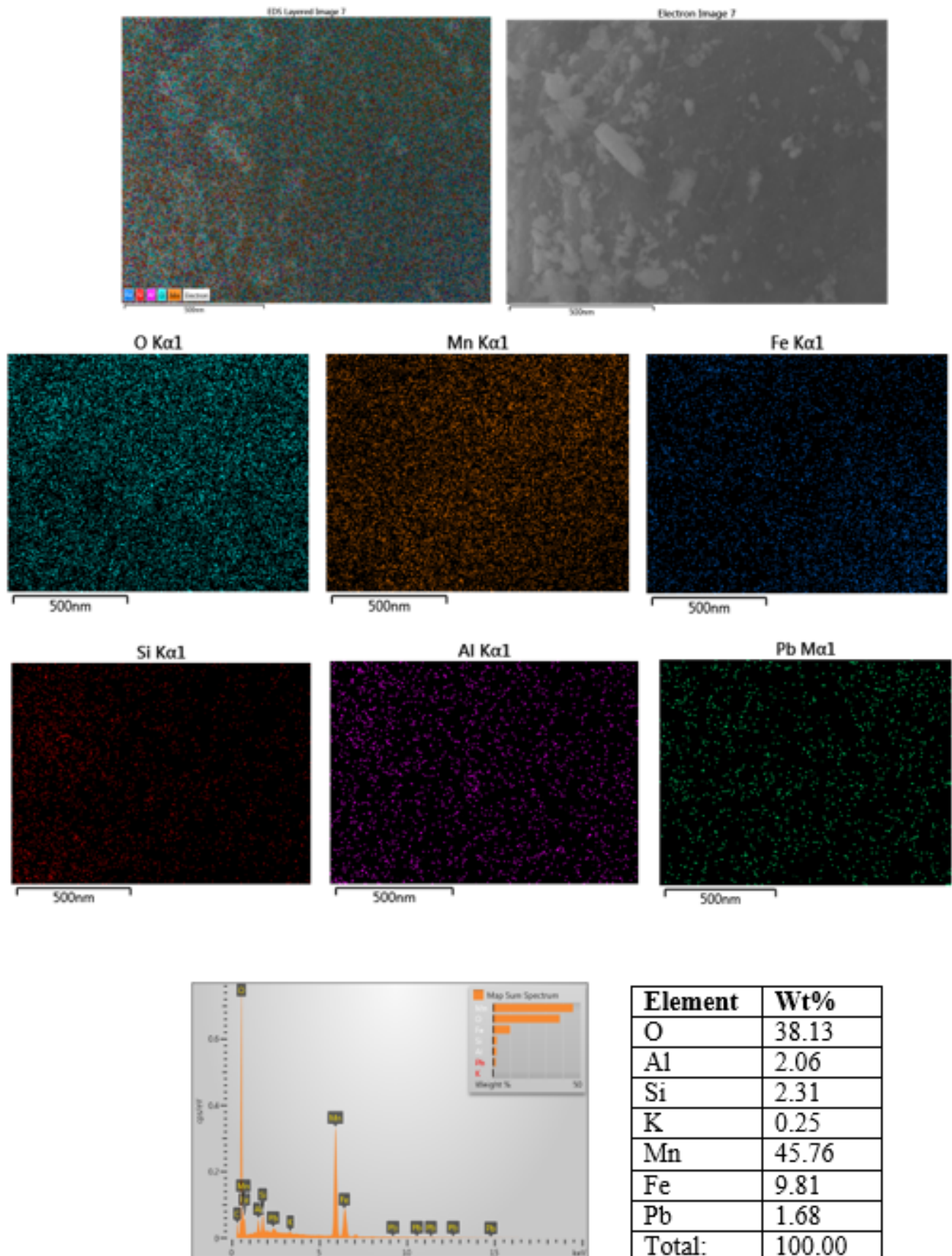| Element	Wt%
O	38.13
Al	2.06
Si	2.31
K	0.25
Mn	45.76
Fe	9.81
Pb	1.68
Total:	100.00

FIGURE 3. SEM/EDS metal mapping of WK ore (Astuti et al. 2019).

acid, the effect of leaching temperature and leaching time was studied. Figure 6a shows an interesting phenomenon, namely manganese from WK ore can leach quickly at higher temperatures. Manganese can be leached up to 76% at 70°C and 60 minutes (1 hour), whereas at 50°C only 62-63% can be leached after 120-240 minutes (2-4 hours) of leaching. At 30°C, manganese can be leached up to 70% but at longer leaching time of 8 hours. However, at 70°C, a decrease in manganese leaching recovery was observed after 1 hour of leaching time. This is likely due to the crystallization of man-
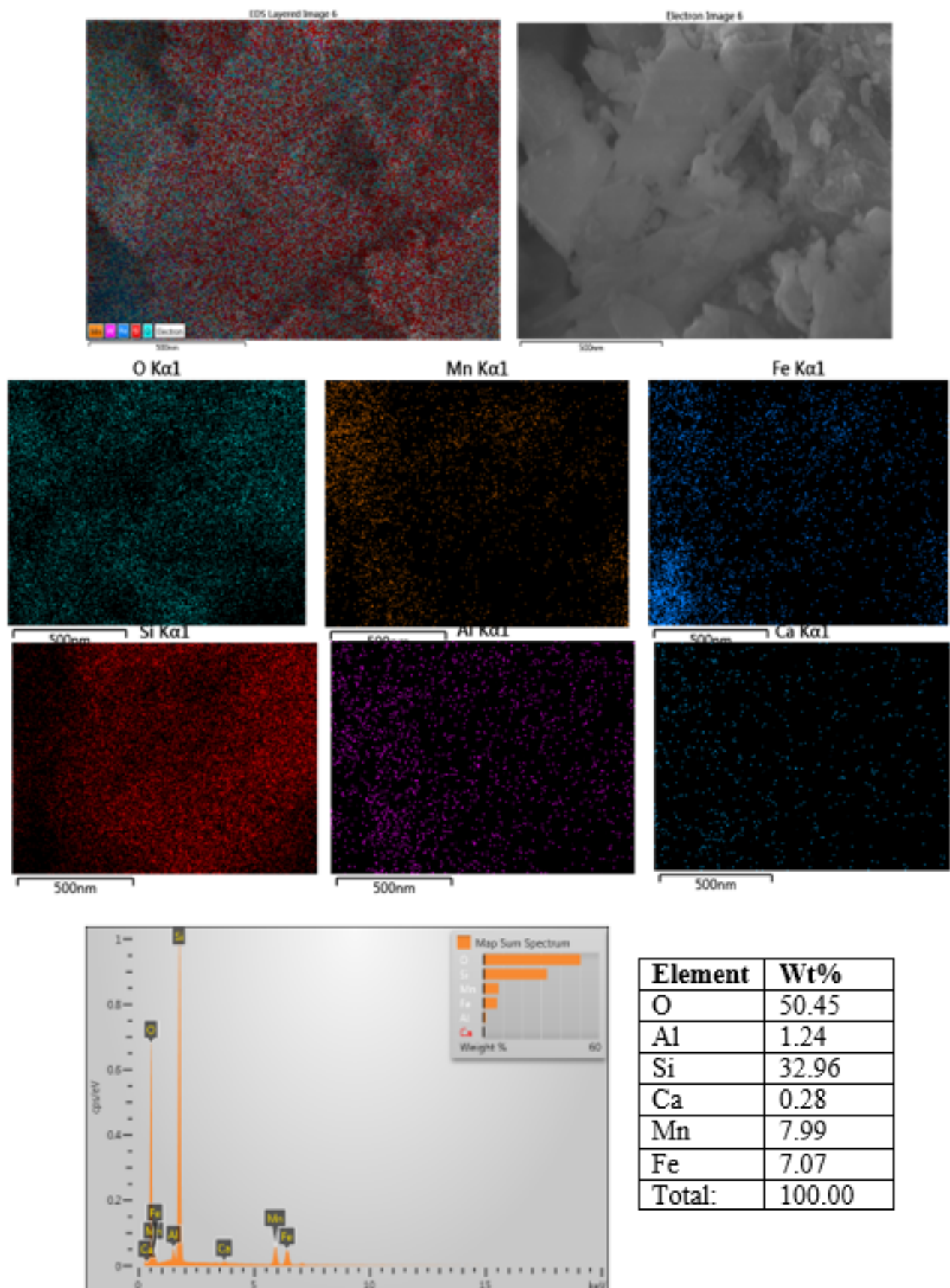
FIGURE 4. SEM/EDS metal mapping of TG ore (Astuti et al. 2019).

ganese citrate, as indicated by the appearance of white crystals after 1 hour of leaching at 70°C, which causes a reduction in the concentration of manganese ions in the leaching solution and a decrease in manganese recovery. Additionally, prolonged exposure at high temperatures may promote secondary reactions, such as the reprecipitation of manganese species, further reducing dissolution efficiency (Baba et al. 2014).
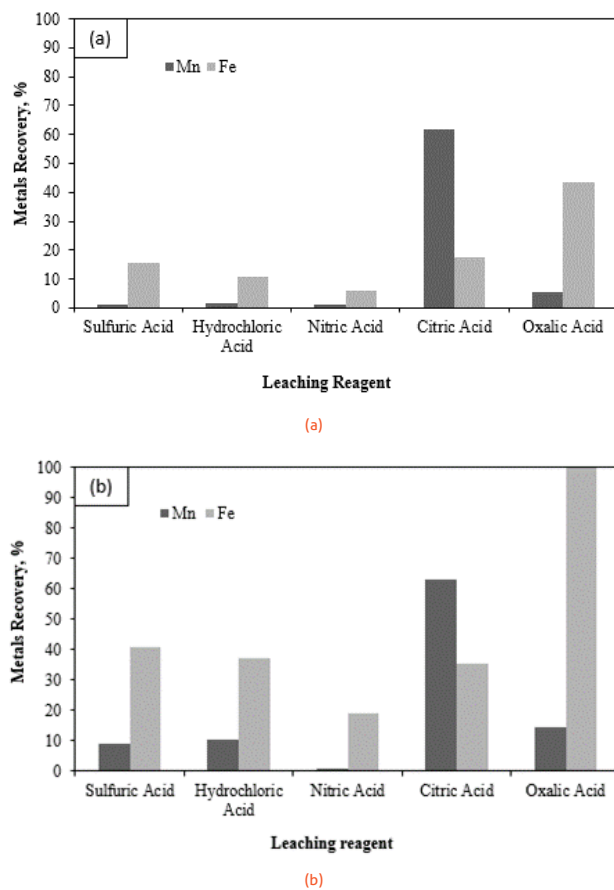
(a)

(b)

FIGURE 5. Effect of leaching reagents on the metals recovery from (a) WK ore and (b) TG ore (Temperature of 50°C; 1 M of acid concentration; 5% pulp density; 2 hours leaching period; 150 rpm of shaker speed).

(a)

(b)

FIGURE 6. Effect of temperature and time on the manganese recovery from (a) WK ore and (b) TG ore in the citric acid leaching.

For TG ore, as shown in Figure 6b, the common leaching phenomenon that occurs is that the higher the temperature and time, the higher the manganese recoveries. The highest manganese recovery was obtained at 70°C and 2 hours of leaching time, which was around 70%. However, at 1 hour of leaching time, manganese recovery of about 69% was also achieved. Hence, it would be more profitable to use 1 hour of leaching time because the difference in manganese recovery of 1 and 2 hours of leaching time is not significant.

The recrystallization of manganese citrate occurs in WK ore but not in TG ore, likely due to differences in ore composition and leachate chemistry. As confirmed by XRD data (Figure 2), WK ore consists predominantly of pyrolusite (MnO_2), which requires citric acid to function as both a leaching and reducing agent. This process releases a higher local concentration of Mn^{2+}, which, under elevated temperatures, can react with citrate anions and precipitate as manganese citrate once supersaturation is reached. In contrast, TG ore contains manganese in more acid-soluble phases, leading to a more controlled dissolution process and preventing manganese citrate precipitation. A similar phenomenon of manganese citrate crystallization at elevated temperatures has been reported in previous studies (Astuti et al. 2016b), further supporting this observation.

From Figure 6, it is known that the difference in the mineral composition of manganese ore can produce different leaching phenomena. However, in general, it can be concluded that the best leaching conditions that produce o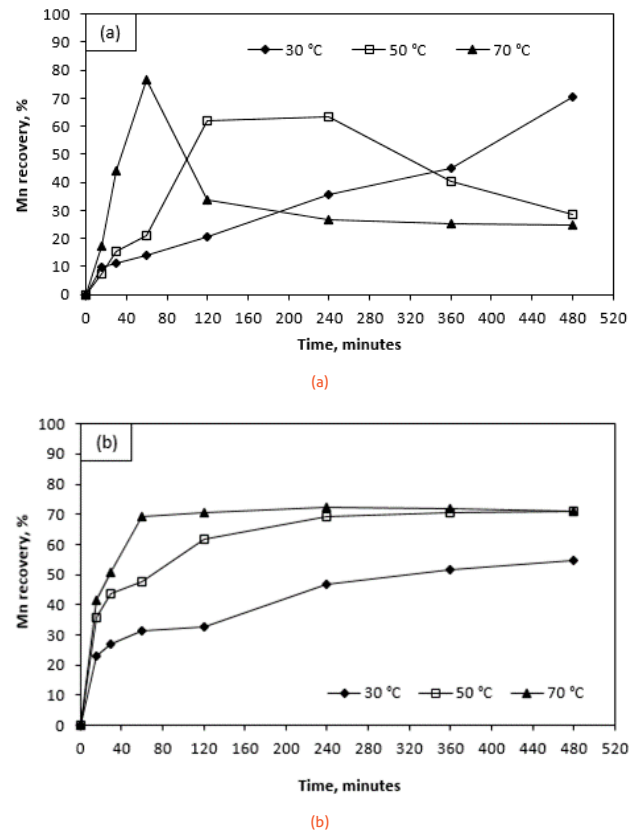ptimum manganese recovery are at 70°C and 1 hour leaching time. For industrial application, citric acid leaching must consider reagent recycling and waste management. The crystallization of manganese citrate at high temperatures may reduce efficiency, requiring process optimization. Additionally, the economic feasibility depends on citric acid recovery to minimize costs. Further studies on reagent reuse and byproduct handling are needed to improve scalability.

4. CONCLUSION

Low-grade manganese ores containing 36.77% Mn (WK ore) and 12.5% (TG ore) with different mineral types can be leached using citric acid as leaching reagent without the addition of reducing agent. The optimum Mn recovery was obtained at around 76% and 69% respectively for WK ore and TG ore with citric acid of 1M, 5% of pulp density, 150 rpm of shaker speed, 30°C of leaching temperature, 1 hour of leaching period, and <75 µm of sample particle size. This condition was also selective for iron recovery leaching.

This study demonstrates the potential of citric acid as an environmentally friendly leaching reagent for manganese extraction, offering advantages such as biodegradability and reduced hazardous waste. However, high reagent consumption and manganese citrate precipitation at elevated temperatures remain challenges for industrial applications. Further research is needed to optimize reagent efficiency, leaching kinetics, and process scalability to enhance its feasibility for large-scale operations.

5. AUTHOR STATEMENT

Widi Astuti: Corresponding author, supply idea, give the finance, and draft the manuscript; Fika Rofiek Mufakhir and

Amru Daulay: do experiments, characterize the samples; analyze data; Yayat Iman Supriyatna, Moch. Setyadji, Jilda Sofiana Dewi: threat some data and edit the manuscript.

6. DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

7. ACKNOWLEDGMENT

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