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RESEARCH ARTICLE

Extraction of manganese from Indonesian manganese ore using sugarcane bagasse-acid reductive leaching

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OBJECTIVES In this study, sugarcane bagasse was used as a reducing agent in manganese leaching from manganese ore from Way Kanan, Lampung under acidic conditions using sulfuric acid as a leaching reagent. Bagasse is an agricultural waste from the cane sugar manufacturing industry which is commonly found in Lampung Province. This agricultural waste has the potential to become a reducing agent in manganese leaching because it contains carbon in the form of cellulose and sugar. METHODS The optimization of the leaching conditions has been investigated with respect to H_2SO_4 concentration, temperature, and pulp density. RESULTS The highest manganese leaching recovery was obtained under the following optimized conditions: 1 M H_2SO_4 concentration, 14 g sugarcane bagasse/20 g MnO2, 50 g/L pulp density, 80 °C leaching temperature, 200 rpm stirring rate and 4 hours of reaction time. CONCLUSIONS The present process therefore deals with achieving the effective recovery of value-added product from low grade manganese ore using agriculture waste as reducing agent.

KEYWORDS Indonesian Manganese Ore; Sugarcane Bagasse; Reductive Leaching

1. INTRODUCTION

Manganese ore is a mineral resource in Indonesia in large quantities but has not been processed optimally. One of these potentials is in Way Kanan Regency, Lampung Province. The most widely available manganese compound in nature is manganese ore in the form of pyrolusite (MnO_2). Various methods are employed to extract manganese, including pyrometallurgical and hydrometallurgical approaches, which are widely used for metal recovery from minerals (Raj et al. 2022; Yang et al. 2021). Among these, the hydrometallurgical approach is the most extensively studied and commercialized due to its efficiency. This method involves key steps such as leaching, where reductants facilitate metal solubilization (Cheng et al. 2022; Okonkwo et al. 2021). Reducing agents play a crucial role by lowering the oxidation state of transition metals like manganese, making them more soluble in acidic solutions and enhancing metal recovery (Cheng et al. 2022; Ma et al. 2021).

The reduction process can be performed separately from the leaching process, as in reduction roasting followed by leaching, or it can be integrated directly into the leaching process by adding reducing agents under acidic conditions, known as hydrometallurgical reductive leaching (Sun et al. 2017; You et al. 2018; Deng et al. 2018; Sinha et al. 2020; Du et al. 2023). However, the reduction roasting method has significant drawbacks, including high energy consumption, substantial investment and operational costs, and the potential release of harmful gases. In contrast, hydrometallurgical reductive leaching offers advantages such as energy efficiency and a one-step process (Sinha and Purcell 2019). This method requires a reducing agent to convert Mn^{4+}, which is stable in acidic solutions, into Mn^{2+}, which is more soluble and can form manganese salt compounds(Xiong et al. 2018; Zhou et al. 2021; Zou et al. 2023).

Commonly used reducing agents include carbon-based and organic compounds such as oxalic acid, tannic acid, and glucose (Xue et al. 2016; Zhang et al. 2018; Prasetyo et al. 2019; Astuti et al. 2019b; Anggraeni et al. 2019). Additionally, agricultural waste materials such as sugarcane bagasse, which contain high levels of carbon, can serve as environmentally friendly reductants. Research by Sun et al. (2017) demonstrated that using straw as a reductant in the reduction of low-grade manganese oxide resulted in a manganese leach-

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ing efficiency of 92.8% under optimized conditions: a stirring speed of 300 rpm, a reductant/ore mass ratio of 0.3, a sulfuric acid concentration of 9% (v/v), a particle size of 45–75 µm, and a temperature of 85°C for 180 minutes.

Other studies have also explored leaching using organic reductants in sulfuric acid media. Zou et al. (2023) investigated the reductive leaching of pyrolusite using orange peel as a reductant over four hours, achieving a manganese dissolution efficiency of 98.1% under optimum conditions: a temperature of 90°C, a sulfuric acid concentration of 0.1 M, and an R/P of 1.5 (w/w). The leaching of manganese using sulfuric acid and organic reductants such as straw, orange peel, and bagasse requires sufficient reaction time to ensure optimal performance. Research indicates that a duration of approximately four hours is effective for achieving high manganese extraction rates. This timeframe allows for complete interactions between sulfuric acid, organic reductants, and manganese ore, maximizing dissolution efficiency.

Sulfuric acid is widely used as a leaching agent in hydrometallurgical processes due to its high efficiency, cost-effectiveness, and scalability (Deng et al. 2018). Compared to other acids such as hydrochloric acid (HCl), nitric acid (HNO_3), and organic acids like oxalic and citric acid, sulfuric acid offers several advantages. HCl effectively dissolves MnO_2 by forming stable Mn^{2+} chloride complexes but poses corrosion risks (Wang et al. 2017), while HNO_3 generates toxic nitrogen oxides (NO_x), leading to environmental concerns (Wu et al. 2020). Organic acids like oxalic acid facilitate manganese dissolution through complex formation but are limited by their high cost and slower reaction kinetics (Xiong et al. 2018). In addition to its effectiveness as a leaching agent, sulfuric acid plays a crucial role in breaking down cellulose-based materials such as sugarcane bagasse, promoting the release of reducing sugars that enhance the reduction of Mn^{4+} to Mn^{2+} (Wu et al. 2014). This decrystallization process disrupts hydrogen bonds in cellulose and lignin, rendering them amorphous and facilitating the hydrolysis of cellulose into monomeric sugars such as glucose, which act as effective reductants (Sun et al. 2017). Due to these properties, carbohydrates, particularly glucose and sucrose, have been widely utilized for extracting transition metals like manganese from mineral ores and spent zinc-alkaline batteries (Wang et al. 2017).

Most studies in the literature have focused on direct manganese leaching using sulfuric acid, often requiring high acid concentrations, which pose environmental concerns. To address this, the present study employs a lower concentration of sulfuric acid combined with sugarcane bagasse as a reducing agent to enhance manganese recovery. The use of bagasse offers significant advantages: it is readily available as an industrial byproduct, environmentally friendly, and non-toxic. This study investigates the potential of sugarcane bagasse, sourced from Way Kanan, Lampung, as a reducing agent under acidic conditions using sulfuric acid. Bagasse, a byproduct of the sugarcane industry, is abundant in Lampung Province. To the best of our knowledge, no prior study has examined the direct leaching of manganese using this combination. This research explores the influence of pulp density, sulfuric acid concentration, and temperature on manganese recovery, aiming to optimize the leaching process while minimizing environmental impact.

2. EXPERIMENTAL

2.1 Materials

The manganese ore was originated from a local mine in Way Kanan Regency, Lampung Province, Indonesia. All chemicals of analytical grade were used as supplied. Concentrated sulfuric acid used was from Merck, Germany. Manganese ore was characterized using X-ray fluorescence (XRF, Epsilon 3^{XLE}, PANalytical, Netherland) and X-ray diffraction (XRD, X'Pert 3 Powder, PANalytical, Netherland), with CuKα radiation and 2θ between 5°to 80°. Scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDS; JEOL, USA) was used to determine the manganese and other metal contents by metal mapping.

Prior to the leaching studies, manganese ore was mechanically crushed, ground, milled, and screened to < 200 mesh. Dry bagasse was crushed using a ball mill and sieved with a size of 200 mesh and then weighed according to stoichiometric calculations shown in equation (1)-(5). In addition to the analysis of manganese ore, an analysis of the cellulose content in bagasse was also carried out at the Agricultural Product Technology Laboratory of the Lampung State Polytechnic. Cellulose content can be calculated using the Chesson method analysis . The Chesson method is a gravimetric analysis for each component (cellulose) after hydrolysis or dissolution. Based on this analysis, the cellulose content of bagasse used in this experiment was 20.30%.

The weight of bagasse required as a reducing agent in the leaching of 20 g MnO_2 in 400 ml of sulfuric acid is calculated by the following reaction equation.

$$\begin{aligned} 12MnO_2 + C_6H_{10}O_5 + 12H_2SO_4 \rightarrow 12MnSO_4 + \\ 6H_2O + 17CO_2 \end{aligned} \tag{1}$$

$$mole\ MnO_2 = \frac{mass}{Mr} = \frac{20\,g}{87\,g/mol} = 0.23\,mole \tag{2}$$

$$mole\ C_6H_{10}O_5 = \frac{1}{12} \times 0.23\,mole = 0.02\,mole \tag{3}$$

$$\begin{aligned} massC_6H_0O_5 = mole \times Mr = 0.02 mole \times 142\,g/mole \\ = 2.84\,g \end{aligned} \tag{4}$$

The results of the analysis of the cellulose content in bagasse obtained by 20.30%. So that the mass of bagasse needed in the manganese leaching process of 20 g MnO_2 is as follows.

$$\begin{aligned} Mass of baggase = \frac{100\%}{20.30} \times 2.84 \\ = 14\,g\ baggase/20\,g\ MnO_2 \end{aligned} \tag{5}$$

2.2 Leaching manganese

Various leaching parameters such as temperature, concentration of sulfuric acid, and solid/liquid ratio (pulp density) were investigated to optimize the leaching conditions. The selection of these parameters is based on previous studies that have demonstrated their significant influence on the

TABLE 1. Chemical composition of manganese ore sample determined by XRF.

Elements	Mn	Fe	Si	Pb	Zn	Al
Wt.%	36.78	6.60	4.13	1.14	0.55	0.46

leaching efficiency of manganese (Zhang et al. 2017; Sinha et al. 2020). The temperature range (30°C, 55°C, and 80°C) was chosen based on the kinetic behavior of manganese leaching, where increased temperature is known to enhance leaching rates due to improved diffusion and reaction kinetics (Zhang et al. 2017). The selected sulfuric acid concentrations (0.1 M, 0.5 M, and 1 M) were determined based on previous studies, which reported that an increase in acid concentration enhances the dissolution of manganese oxides but may also lead to excessive dissolution of iron impurities (Sinha et al. 2020). The pulp density variations (50 g/L, 100 g/L, and 200 g/L) were selected to evaluate their impact on mass transfer and leaching kinetics, where lower pulp densities generally facilitate better metal recovery due to improved reagent accessibility.

The leaching experiments were conducted for 4 h in an incubator shaker. In a typical experiment, 20 g of manganese oxide ore sample and 14 g of bagasse were first added to the agitated dilute sulfuric acid solution at the required temperature. The obtained ore slurry was then agitated at the required stirring speed (200 rpm). After completion of the leaching experiment, the slurry was filtered to separate leach liquor and leach residue. The leach residue was then washed three times with deionized water. The manganese ore leaching process was performed under the aforementioned variations in leaching parameters to determine the optimum conditions for maximizing manganese recovery.

During leaching, a small amount of the aliquot of leach solution was withdrawn for analyzing the metal ion concentration using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES; PlasmaQuant (PQ) 9100) after proper dilution. The recovery of manganese leached (%) was calculated according to the following equation:.

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$$\% \text{ Recovery Mn} = \frac{c\,V}{M_0} \times 100\% \qquad (6)$$

where c and M_0 are the mass of Mn in the leaching solution and the original sample, respectively; V is the volume of the leaching solution after filtration

3. RESULTS AND DISSCUSSION

3.1 Characterization of manganese ore

Chemical analysis of the manganese ore sample (Table 1) shows the presence of Mn (36.78%), Fe (6.60%) and Si (4.13%) as the major elements while Al, Pb and Zn respectively are present in minor quantities. The mineralogical phases of manganese ore and its surface morphology were determined using XRD and SEM/EDS. The XRD results in Figure 1 indicate that the manganese ore mainly consists of pyrolusite (MnO_2) and quartz (SiO_2). The X-ray diffraction pattern confirms that pyrolusite is the dominant phase, with a content of approximately 90.3%, while quartz is present in a smaller amount, around 9.7%. The three highest diffraction peaks in the XRD pattern are observed at 2θ 26.6°, 37.3°, and 55.1°. The peak at 26.6°corresponds to the presence of quartz (SiO_2), while the peaks at 37.3°and 55.1°are characteristic of pyrolusite (MnO_2), indicating its dominant crystalline structure.

Fig. 2a presents the metal mapping of manganese ore (WK) based on SEM/EDS analysis. The results elemental map-

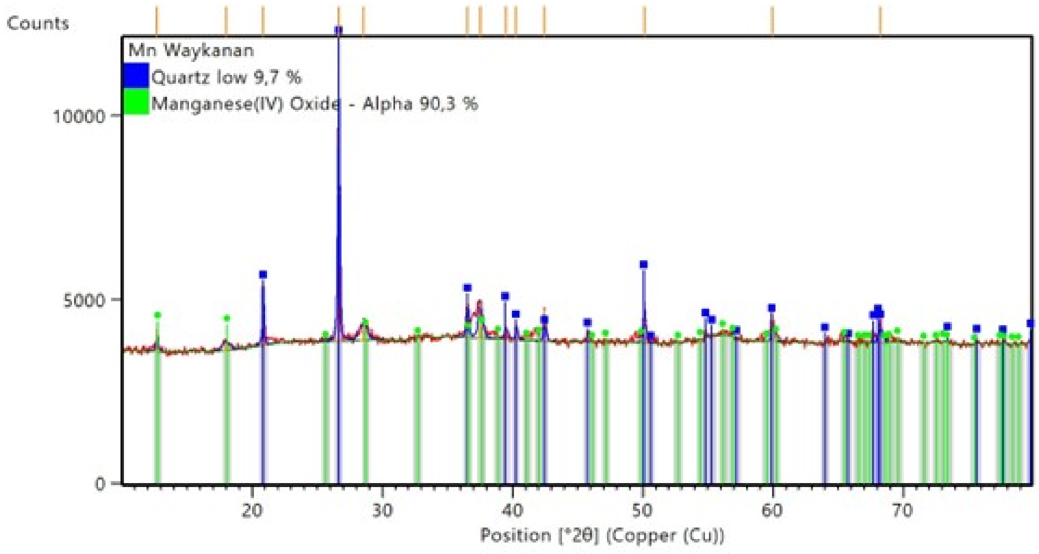
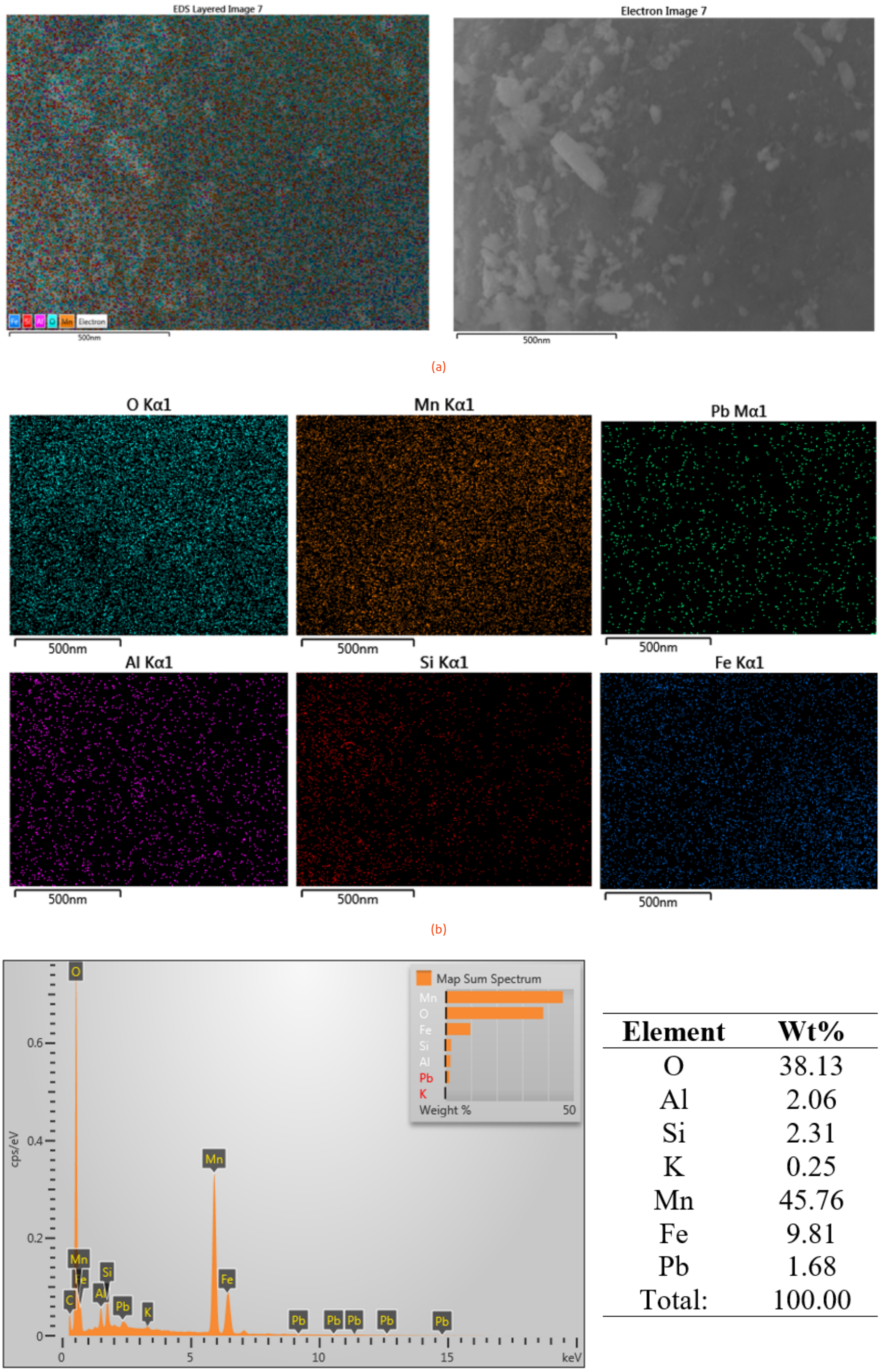


FIGURE 1. XRD pattern of manganese ore (Astuti et al. 2019a).



(a)

(b)

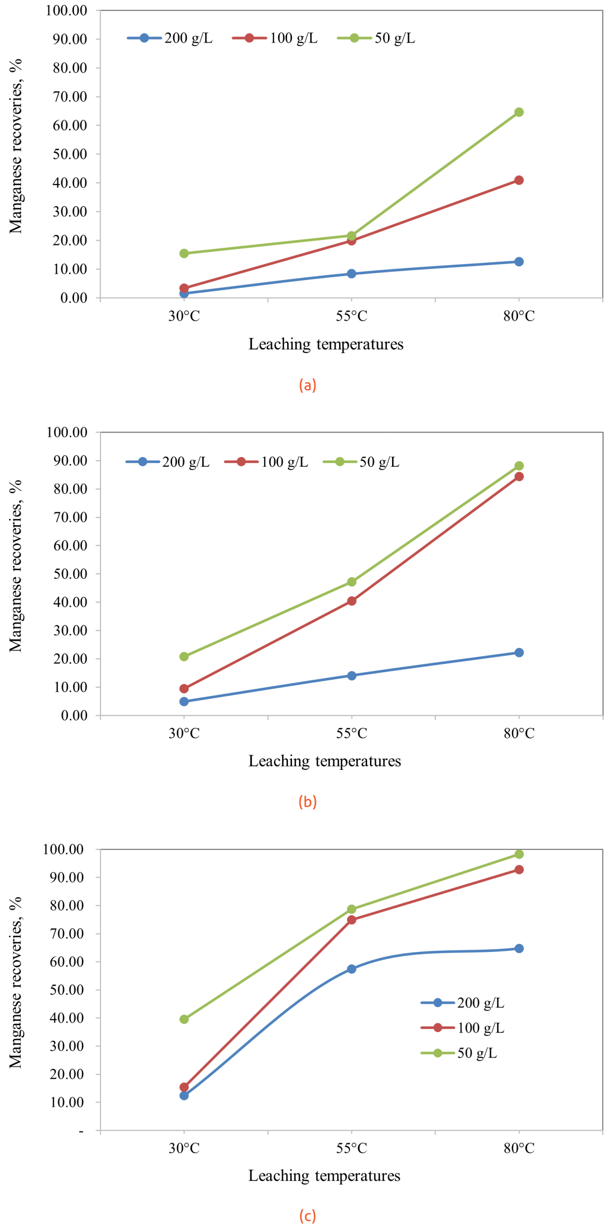
Element	Wt%
O	38.13
Al	2.06
Si	2.31
K	0.25
Mn	45.76
Fe	9.81
Pb	1.68
Total:	100.00

(c)

FIGURE 2. (Result (a)SEM; (b) elemental mapping; (c) EDS spectrum and elemental composition from manganese ore (Astuti et al. 2019a).

ping (Fig. 2b) indicated that manganese in WK ore is closely associated with oxygen, forming manganese dioxide (MnO_2). Meanwhile, Fe, Si, and Al exhibit a more dispersed distribution, indicating heterogeneity in the sample composition. These findings align with previous studies, which reported that natural manganese ores often contain various impurities, including silica and iron oxides (Astuti et al. 2019a).

The EDS spectrum (Fig. 2c) reveals major peaks corresponding to Mn, Fe, Si, Al, Pb, and K, indicating the diverse mineral composition of the sample. The elemental composition (Fig. 2c) obtained from the EDS analysis shows that manganese (Mn) is the dominant element, accounting for 45.76%,

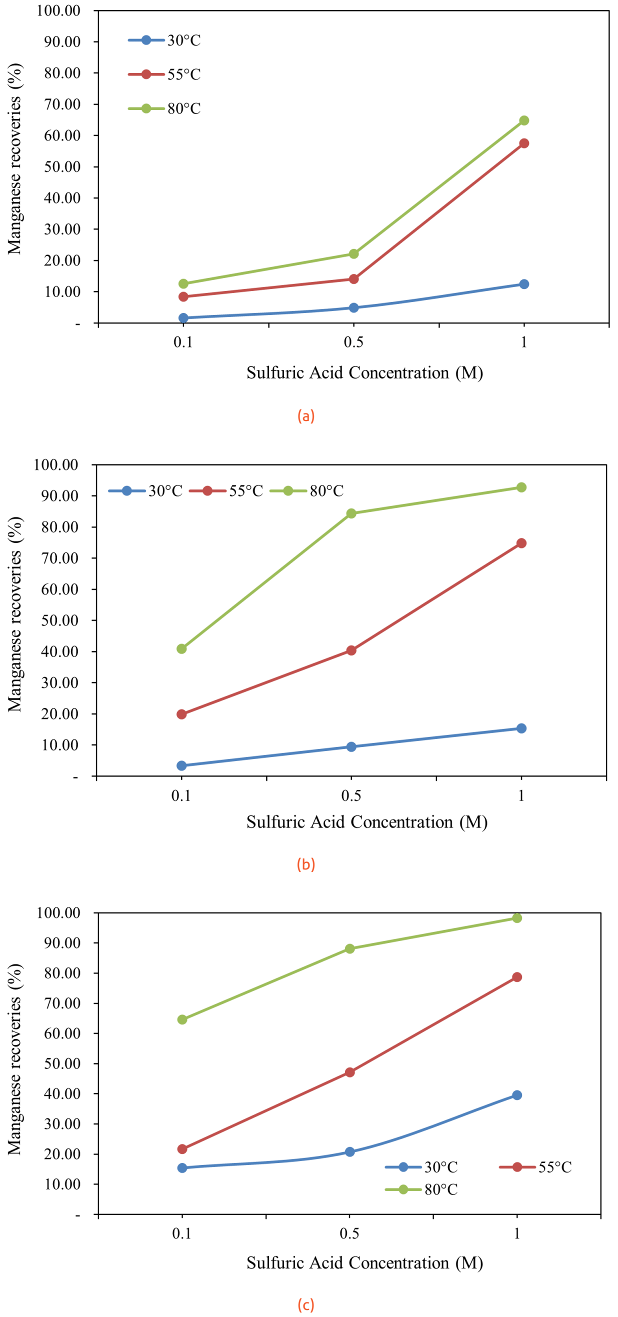


(a)

(b)

(c)

FIGURE 3. **Effect of leaching temperatures on the manganese recoveries at different pulp densities (50, 100, 200 g/L) and sulfuric acid concentrations (a) 0.1 M; (b) 0.5 M; (c) 1M.**



(a)

(b)

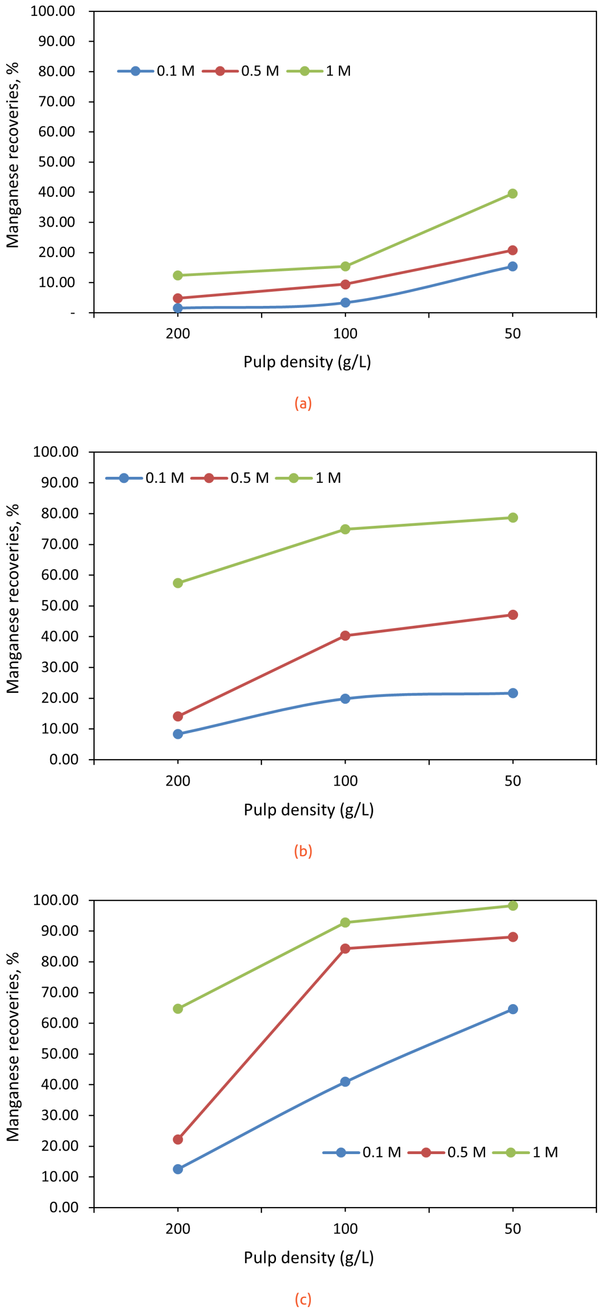
(c)

FIGURE 4. **Effect of sulfuric acid concentration on the manganese recoveries at different temperature (30, 55, 80 °C) and pulp density (a) 200 g/L; (b) 100 g/L; (c) 50 g/L.**

followed by oxygen (O) at 38.13%, iron (Fe) at 9.81%, and minor amounts of other elements such as silicon (Si) 2.31%, aluminum (Al) 2.06%, lead (Pb) 1.68%, and potassium (K) 0.25%. The presence of iron (Fe) in a significant amount suggests possible impurities such as hematite or goethite within the ore sample.

To investigate the effect of leaching temperature on manganese leaching efficiency, the ore was leached at 30, 55, and 80 °C under various sulfuric acid concentrations (0.1 M, 0.5 M, 1 M) and pulp densities (50 g/L, 100 g/L, 200 g/L). Bagasse was used as a reducing agent in a stoichiometric ratio of 14 g per 20 g MnO_2. The recovery of Mn increased with rising temperature across all sulfuric acid concentrations and pulp densities. At 30 °C, manganese leaching progressed gradually, reaching a maximum efficiency of 39.54% Mn at 1 M H_2SO_4 and 50 g/L pulp density. Increasing the temperature to 55 °C significantly enhanced manganese dissolution, achieving 78.66% Mn recovery. At 80 °C, the leaching efficiency peaked at 98.31% Mn dissolution.

The observed trends in manganese recovery can be explained by the dissolution kinetics of MnO_2 in an acidic medium in the presence of a reducing agent. The leaching process follows a redox reaction mechanism where Mn^{4+} in pyrolusite is reduced to soluble Mn^{2+} via electron transfer from organic compounds present in sugarcane bagasse. The rate of this reaction is highly temperature-dependent, as increasing temperature enhances reaction kinetics by promoting faster diffusion of H^+ ions to the ore surface and increasing the oxidation rate of reducing agents (Zhang et al. 2017). Moreover, higher temperatures reduce the viscosity of the leaching solution, decreasing mass transfer resistance and facilitating better interaction between reactants (Sinha et al. 2020). The effectiveness of bagasse as a reducing agent is



(a)

(b)

(c)

FIGURE 5. Effect of pulp density on the manganese recoveries at different sulfuric acid concentration (0.1; 0.5; 1M) and leaching temperature (a) 30°C; (b) 55°C; (c) 80°C.

attributed to its organic content, such as cellulose. During the leaching process, these components undergo hydrolysis and degradation, producing various organic acids, such as oxalic acid and acetic acid, which act as reducing agents (Cheng et al. 2022).

The use of bagasse as a reducing agent was compared to other organic reductants, such as glucose and tannic acid, which have been previously studied for manganese leaching. Glucose is commonly employed due to its ability to facilitate the reduction of Mn^{4+} to Mn^{2+} through an oxidation reaction in acidic media, yielding high manganese recoveries of approximately 85–97% under optimized conditions (Ma et al. 2021). However, glucose tends to degrade at elevated temperatures, which can limit its long-term efficiency in high-temperature leaching processes. Tannic acid, another organic reductant, has shown promising results, achieving Mn recoveries above 95% due to its high polyphenol content, which acts as an effective electron donor (Wu et al. 2020). Nevertheless, the higher cost and limited availability of tannic acid may pose economic challenges for large-scale applications. Compared to these reductants, bagasse provides a sustainable and cost-effective alternative while maintaining comparable leaching performance.

3.2 Effect of sulfuric acid concentration on manganese leaching

The effect of sulfuric acid concentration (0.1; 0.5; 1M) on the leaching efficiency of manganese and iron from the manganese ore was studied at various leaching temperatures (30 °C; 55 °C; 80 °C), various pulp density (50 g/L; 100 g/L; 200 g/L) at 200 rpm shaker speed, 4 hours leaching time using stoichiometric amount of baggase as reductant. Results (Fig. 4) show that the leaching of Mn is strongly affected by the acid concentration and leaching efficiency increases with increase in the H_2SO_4 concentration. It was observed that recovery of manganese increased from 64.63% using 0.1 M H_2SO_4 to 88.14% using 0.5 M H_2SO_4. Then, the manganese recovery increased from 88.14% to 98.31% when using 0.5 M to 1 M H_2SO_4.

3.3 Effects of pulp density on manganese leaching

The change in the leaching rate and recovery of manganese with changing the solid-to-liquid ratio (pulp density) was investigated using 50 g/L to 200 g/L ratios of the manganese ore in various sulfuric acid concentration (0.1; 0.5; 1M) and various leaching temperature (30 °C; 55 °C; 80 °C) with 14 g baggase/20 g MnO_2 and 200 rpm shaker speed for 4 hours. Fig. 5 shows that the rate of leaching and the maximum percentage recovery of manganese increase with decreasing solid–liquid ratio. The maximum recovery of manganese increased from 64.77% to 92.75% at a pulp density of 200 g/L and 100 g/L respectively in 4 hours leaching time while quantitative (98.31%) recovery of Mn was observed with 1 M H_2SO_4 at a pulp density of 50 g/L. Since complete leaching of Mn was observed at 50 g/L pulp density, it was considered as the optimum solid-to-liquid ratio for the manganese recovery. These results show that decreasing pulp density creates mass transfer rate between the ore surface and the sulphuric acid solution faster than the denser pulp density (Sinha et al. 2020). It can be concluded that the maximum recovery of Mn can be obtained by leaching the manganese ore with 1 M H_2SO_4 for 4 hours at 80 °C and 50 g/L pulp density using baggase 14 g/20 g MnO_2 as a reducing agent.

4. CONCLUSION

The results of this study demonstrate that manganese leaching efficiency is significantly influenced by temperature, sulfuric acid concentration, and pulp density. The highest manganese recovery of 98.31% was achieved at an optimum condition of 80 °C, 1 M H_2SO_4, and 50 g/L pulp density using bagasse as a reducing agent. This optimal condition enhances manganese dissolution due to the combined effects of increased reaction kinetics at higher temperatures, improved acid penetration, and reduced mass transfer resistance at lower pulp densities. The use of bagasse effectively facilitated the reduction of Mn^{4+} to Mn^{2+}, allowing for better solubility in the sulfuric acid medium.

These findings highlight the potential of agricultural waste as an environmentally friendly and cost-effective reductant for manganese extraction, providing a sustainable alternative to conventional reductants. The application of this method in industrial settings could contribute to reducing reliance on synthetic reducing agents, lowering processing costs, and minimizing environmental impact. However, further studies are needed to evaluate the scalability of this process, including the handling of large-scale waste generation, continuous process optimization, and the economic feasibility of using bagasse in commercial manganese recovery operations. Additionally, potential limitations such as the variability in bagasse composition and its effect on process consistency should be explored to ensure reproducibility and efficiency in industrial applications.

5. DECLARE

All the authors have no interest conflict.

6. AUTHOR STATEMENT

Widi Astuti: Corresponding author, supply idea, give the finance, and draft the manuscript; Kherani Hana Pinania, Slamet Sumardi, and Harry Supriyadi: do experiments, characterize the samples; analyze data; and Donny Lesmana, Agus Prakosa, and Jilda Sofiana Dewi: threat some data.

7. 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.

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