

Research Paper

Effect of Leaching Time of Paludda Manganese Ore Using Sulfuric Acid Solution

Sitti Ratmi Nurhawaisyah^{a*}, Muhammad Fajrul^a, Muhammad Idris Juradi^a, Mubdiana Arifin^a, Suriyanto Bakri^b

^a Mining Engineering Department, Faculty of Industrial Technology, Universitas Muslim Indonesia 90231, Indonesia

^b Metallurgical Engineering Study Program, Production Technology Department, Institut Teknologi Bacharuddin Jusuf Habibie, Indonesia

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ABSTRACT: In the mining industry, increasing the concentration of valuable minerals in ore is essential to enhance its economic value. Manganese ore is considered economically viable and marketable when it has a purity level exceeding 40%. However, not all extracted manganese minerals meet this purity threshold. Various processing methods can be employed to separate manganese-bearing minerals from their associated gangue minerals. One such method is leaching using sulfuric acid solution. The objective of this study is to investigate the effect of leaching time on manganese recovery. The leaching durations examined in this study were 90 minutes, 120 minutes, 150 minutes, and 180 minutes. In addition, the leaching process also employed sulfuric acid and molasses as additives, conducted at a temperature of 80 °C with a stirring speed of 200 rpm. The research began with the preparation of manganese ore to obtain the desired particle size, followed by sample division using the coning and quartering method to ensure representative sampling. A portion of the samples was sent to the laboratory for initial characterization using X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF) analyses. The remaining samples were subjected to the leaching process. Subsequently, the leachate was analyzed using Atomic Absorption Spectrophotometry (AAS). The results indicated that the highest dissolved manganese recovery, 14.4244638%, was achieved at a leaching time of 180 minutes. These findings suggest that leaching time is a critical variable significantly influencing the leaching process. The longer the leaching time, the greater the manganese recovery obtained.

Keywords: anganese ore; leaching time; sulfuric acid; molasses; Mn recovery

1. INTRODUCTION

Manganese ore is an essential raw material in the steel industry, primarily in the form of ferromanganese. It can also be utilized for non-metallurgical purposes, such as the production of dry cell batteries, animal feed, fertilizers, and in the chemical industry [1]. In addition, manganese is also used in the production of driers for paints and varnishes. It has significant applications in the ceramics and glass industries as a coloring agent [2]. Therefore, manganese ore holds considerable importance from an industrial perspective. The global demand for manganese ore is expected to increase annually.

Manganese (Mn) is one of the minerals in Indonesia that holds significant potential in terms of its economic value, diverse applications, and the availability of its reserves [3]. To date, low-grade manganese ore in Indonesia has not been optimally utilized [4]. The issuance of Government Regulation No. 7 of 2012, which prohibits the export of unprocessed mineral resources, underscores the necessity of processing these materials into semi-finished or final products to increase their added value. Among the 14 types of valuable metal ores restricted from export is manganese ore [5].

High-grade manganese ore, commonly referred to as metallurgical grade, is characterized by a manganese content exceeding 44% and is primarily utilized in the steel industry to produce ferromanganese or manganese-alloyed steel. In contrast, low-grade manganese ore is generally used for non-metallurgical purposes, such as the manufacture of manganese dioxide (MnO₂) for dry cell battery depolarizers, ceramics, or other chemical compounds [6]. The extraction of high-grade manganese ore is typically conducted via the pyrometallurgical route to produce ferromanganese and silicomanganese, for which demand continues to

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Department of Chemical Engineering
Faculty of Industrial Technology
Universitas Muslim Indonesia, Makassar

Address

Jalan Urip Sumohardjo km. 05 (Kampus 2 UMI) Makassar- Sulawesi Selatan
e-mail : jcpe@umi.ac.id

Corresponding Author *
sitti.nurhawaisyah@umi.ac.id



increase. Initially, ferromanganese was produced using blast furnaces with coke or charcoal as reducing agents. However, due to environmental concerns and the need for improved energy efficiency, current ferromanganese production predominantly employs electric arc furnaces [6]. Manganese ores containing less than 40% Mn are used in the production of chemical compounds such as potassium permanganate and MnO_2 , among others. Pyrolusite-type manganese minerals can undergo selective leaching under acidic conditions. This leaching process is reductive in nature, requiring specific reagents to reduce the oxidation state of manganese from Mn (IV) to Mn (II), thereby enabling its dissolution using acidic compounds [6].

Acidic solvents are commonly employed in the leaching process of manganese ores [7]. Selective leaching of manganese ore under acidic conditions is only feasible when manganese exists in the divalent state (Mn^{2+}). Frequently used acidic leachants include sulfuric acid [8], nitric acid, and hydrochloric acid [9]. In addition to these acids, other leaching agents such as SO_2 , FeS_2 , and Na_2SO_3 are also commonly used, typically at elevated temperatures [10]. Jiangang et al. investigated the leaching of manganese ore using sulfuric acid in combination with H_2O_2 as the reductant. Similarly, Qing-quan et al. studied the production of manganese sulfate from low-grade carbonate manganese ore using sulfuric acid as the leaching medium [11]. Leaching of manganese ore using molasses (a by-product of sugar processing) as a reductant in sulfuric acid solution was carried out by Sumardi et al., achieving a manganese recovery rate of 92.31% [12].

A variety of reductants have been developed to improve the efficiency of manganese recovery processes, including both inorganic substances, such as sulfur dioxide [13], hydrogen peroxide [8], and various organic materials. Inorganic reductants, however, may introduce metallic impurities into the processed sample and pose environmental hazards. As a result, considerable research has focused on the use of organic reductants, particularly carbohydrates such as glucose, sucrose, cellulose, lactose, and oxalic acid, due to their environmental friendliness and cost-effectiveness [14]. Organic reductants often possess oxidizable functional groups, such as the aldehyde and ketone groups present in carbohydrate monomers. Numerous studies have explored the use of organic materials as reductants for manganese recovery, including alcohol waste [15], cornstalk [16], corncob [14], carbohydrates [7], molasses [9], and sawdust [17].

The primary objective of this study is to develop an efficient method for processing manganese ore from Pujananting Subdistrict, Barru Regency, into economically valuable products. This research focuses on the leaching time of manganese ore under acidic conditions. Leaching studies on manganese ore have also been conducted, primarily focusing on the effect of leaching temperature. The results indicate that a temperature of 8°C yields the optimum manganese recovery. Furthermore, the use of a combination of sulfuric acid and molasses in the leaching process has shown a significant improvement in leaching performance [18]. For example, in manganese ore containing MnO_2 (Mn(IV)), the direct reaction with H^+ alone often is slow and incomplete because Mn(IV) oxide is stable and requires reduction to Mn(II) to become soluble. In the study with sucrose (reducing agent) and H_2SO_4 , leaching of MnO_2 and MnCO_3 reached 91,8% only when the reducing agent was present, whereas without the reducing agent, the MnO_2 dissolution was very low [19]. Another study using molasses as a reducing agent in the sulphuric acid leaching of pyrolusite achieved 97,6% Mn extraction with an optimum molasses concentration (50%) [20]. Molasses is a low-cost byproduct (sugar-cane industry), and using it as a reductant is more benign than some inorganic reductants [9]. The advantages of using sulfuric acid and molasses for leaching manganese ore can provide higher yield, faster kinetics, lower reagent cost, simpler equipment/operations, and a better environmental footprint, and are selectively compared to many traditional methods (e.g., non-reductive acid leaching, roasting+leaching, chemical reductants). It is therefore a compelling method, especially for low-grade or refractory manganese ores [12][19][21]. Previous studies in this region have primarily concentrated on ore grade enhancement through beneficiation, utilizing the magnetic properties of minerals to separate manganese ore from its impurities [22]. The novelty of this study lies in its focus on a specific raw material source manganese ore from the Palludda area, Barru Regency, which has rarely, if ever, been comprehensively investigated in terms of leaching-based processing methods.

This research thus offers a novel contribution within the geographical context and highlights the unique characteristics of the local mineral resources. This study is expected to provide insight into the utilization of local resources, particularly manganese ore, for industrial processing and purification. This aligns with the faculty's flagship program in engineering and environmental sustainability. Through the engineering of processing methods, it is possible to reduce operational costs and minimize the excessive use of chemical reagents during purification processes.

2. MATERIAL AND METHOD

This study was conducted using samples obtained from Palludda, Barru Regency, South Sulawesi Province. The location map of the study area is presented in Figure 1.

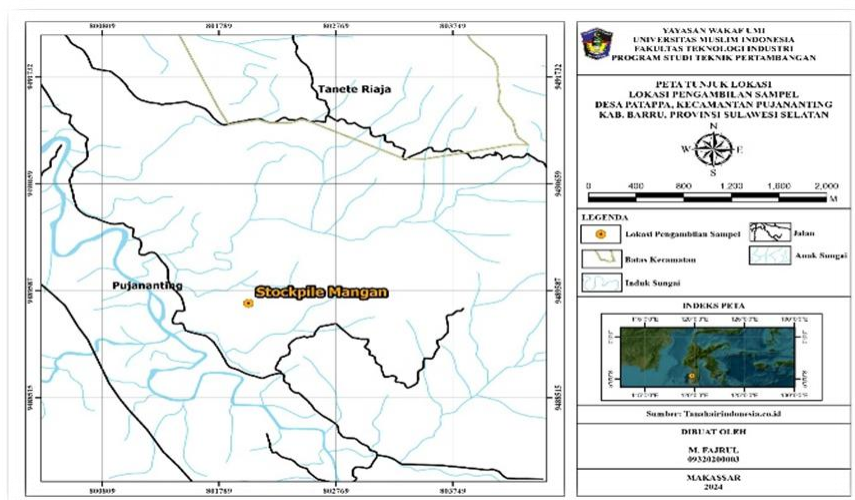


Figure 1. Research location map

The collected manganese ore samples were prepared through crushing, grinding, and sieving processes to obtain a particle size of -150 mesh, which was subsequently used for preliminary analysis and partially as feed material for the study. The preliminary analyses included X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), and Atomic Absorption Spectrophotometry (AAS) on solid samples, aimed at identifying the gangue minerals associated with the manganese ore and determining the elemental composition of the samples used. The extraction of manganese ore by leaching in this study was conducted using four different leaching durations: 90 minutes, 120 minutes, 150 minutes, and 180 minutes. Each experiment utilized 5 grams of manganese ore sample, 6% sulfuric acid, and 5 mL of molasses as an additive. Molasses (a sugar-industry by-product) is inexpensive and abundantly available in sugar-producing regions. Molasses is comparable to pure sugar (glucose/sucrose) or other biomass reductants in converting Mn (IV) to Mn (II) under acidic conditions [23]. Leaching was carried out at a temperature of 80°C with a stirring speed of 200 rpm. The samples were subsequently filtered to obtain the filtrate for Atomic Absorption Spectrophotometry (AAS) analysis. The obtained analytical data were processed to determine the manganese recovery percentage.

3. RESULT AND DISCUSSION

3.1. Initial Characterization of Manganese Ore

Initial characterization of manganese ore samples is necessary to determine their phases and chemical composition. The prepared samples, with a particle size of -150 mesh, were then used for preliminary analysis.

a. X-Ray Fluorescence (XRF) Analysis

In this study, the composition of manganese ore was determined using X-Ray Fluorescence (XRF) analysis. The purpose of the XRF analysis was to identify the elemental contents present in the samples. The

results of this analysis are presented as the percentage of elements in the form of oxide compounds. The XRF analysis of the manganese ore samples is presented in Table 1.

Table 1. XRF analysis results

Size (Mesh)	Compounds	Total (% weight)
-150	SiO ₂	-0.19
	Al ₂ O ₃	0.425
	TiO ₂	0.077
	Fe ₂ O ₃	16.718
	CaO	0.796
	MgO	-0.106
	K ₂ O	0.109
	P ₂ O ₅	0.591
	MnO	24.679
	Cr ₂ O ₃	0.07

Based on the chemical analysis using X-Ray Fluorescence (XRF) on the -150-mesh fraction of the initial manganese ore sample, the MnO compound content was found to be 24.679%. The percentage composition of Fe₂O₃ was 16.718%, while other compounds such as SiO₂, Al₂O₃, TiO₂, CaO, MgO, K₂O, P₂O₅, MnO, and Cr₂O₃ were present in amounts below 1%.

b. X-Ray Diffraction (XRD) Analysis

In addition to determining the chemical composition, the crystal structure of the sample was also analyzed. This process was conducted using X-Ray Diffraction (XRD), which operates based on the principle of X-ray diffraction. The measurement data were processed using Match software. The results of the XRD analysis are presented in Figure 2 and Table 2.

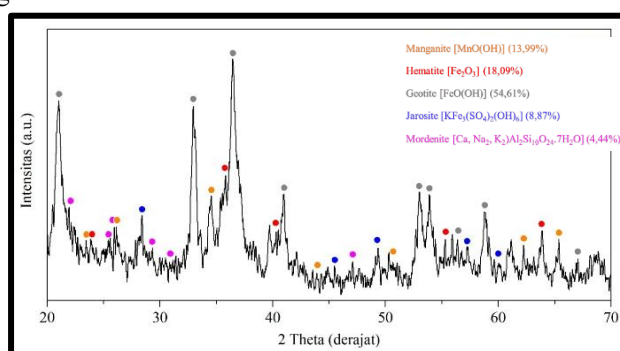


Figure 2. XRD analysis diffractogram

Table 2. X-Ray Diffraction (XRD) analysis result

No	Minerals	Chemical Composition	Presentase (%)
1	Manganite	MnO(OH)	13.99
2	Hematite	Fe ₂ O ₃	18.09
3	Goethite	FeO(OH)	54.61
4	Jarosite	KFe ₃ (SO ₄) ₂ (OH) ₆	8.87
5	Mordenite	Ca, Na ₂ , K ₂ Al ₂ Si ₁₀ O ₂₄ .7H ₂ O	4.44

Based on the results of the XRD analysis presented in Table 4.2 for the -150 mesh fraction of the initial manganese ore sample, the mineralogical composition consists of manganite at 13.99%, hematite at 18.09%, goethite at 54.61%, jarosite at 8.87%, and mordenite at 4.44%.

c. Atomic Absorption Spectrophotometry (AAS) Analysis

Atomic Absorption Spectrophotometry (AAS) analysis of the initial sample was conducted to determine the manganese content within the sample. The AAS results are presented in Table 3 below.

Table 3. AAS analysis result

Weight (g)	Sample Code	Absorbance	Mn concentration (ppm)
10	Initial sample	2.4533	195.2336

3.2. Manganese Ore Leaching Result

Each leaching experiment used 5 grams of manganese ore sample, 6% sulfuric acid, and 5 mL of molasses as an additive. Sulfuric acid supplies the protons and sulfate that dissolve manganese oxides to form soluble Mn^{2+} (as MnSO_4) and strongly controls reaction rate and mechanism. Molasses supplied reducing sugars that chemically reduce higher-valent Mn ($\text{Mn}^{4+}/\text{Mn}^{3+}$) to soluble Mn^{2+} , greatly increasing Mn extraction, lowering required temperature/energy, and speeding kinetics. The two together give much higher Mn recoveries than acid alone [24]. The leaching process was carried out using different leaching durations of 90, 120, 150, and 180 minutes. The resulting filtrates amount to 1.5 gr were then analyzed using Atomic Absorption Spectroscopy (AAS) to measure the concentration of dissolved manganese. The manganese content in the leachate (ppm), as determined by AAS analysis, is presented in Table 4.

Table 4. AAS results of dissolved Mn

Leaching time (minutes)	Dissolved Mn concentration (ppm)
90	28.1226
120	28.1568
150	28.1475
180	28.1614

Manganese recovery was determined by comparing the mass of metal in the pregnant solution with the mass of metal in the ore sample [25]. As illustrated in Figure 3, manganese recovery exhibited both increases and decreases across the different leaching time variations.

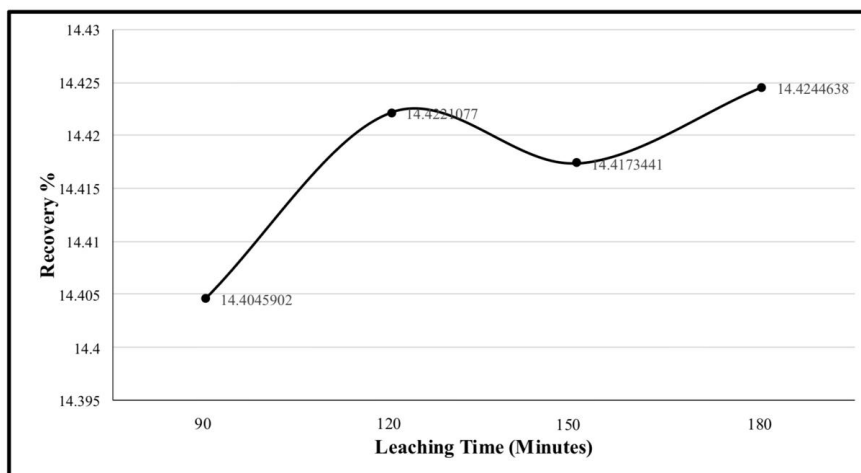


Figure 3. The effect of leaching time on Mn recovery

At 90, 120, and 180 minutes, manganese recovery increased to 14.4045902%, 14.4221077%, and 14.4244638%, respectively. This increase is attributed to the prolonged leaching duration, which allows for extended contact between the manganese and the leaching agent, thereby enhancing the leaching process. Based on the graphic show that the optimum leaching time is 120 minutes. Su et al. (reductive leaching with cane molasses): optimal conditions included 120 min at 90 °C ($1.9 \text{ mol}\cdot\text{L}^{-1} \text{ H}_2\text{SO}_4$, $60 \text{ g}\cdot\text{L}^{-1}$ molasses) to maximize recovery for the tested ore/particle size. The paper shows reaction time is a key parameter and that long times (minutes $\rightarrow$ hours) were used to reach high extraction for that ore [24]. MDPI (Wang et al., sucrose as reductant — similar reductive chemistry): showed most Mn leaching from mixed ores occurred quickly and that increasing time beyond a certain point produced only small gains; reaction time used in their kinetic fits and experiments is typically 30–120 min. This study is useful because it contains kinetic analysis and time-dependence plots [19]. According to Trinopiawan and Sumiarti [26], the duration of contact between the material and the solvent significantly influences the completeness of the reaction. Similarly, Prassanti [27] stated that a sufficiently long leaching time enables a more complete reaction between the material and sulfuric acid, resulting in higher yields. However, while longer leaching times improve contact between the sample and the solvent, the effect diminishes once equilibrium is reached, as the number of dissolved solids in the filtrate becomes constant [28][29]. In other research, show molasses improved extraction rates of Li, Co, Ni and Mn and explicitly tested adding molasses at the start and after 1 hour-demonstrating that timing of molasses addition affects kinetics and final recoveries. It is a clear demonstration that molasses speeds up dissolution in acetic-acid systems [30].

In contrast, a decrease in manganese recovery was observed at the 150-minute leaching duration. This reduction is attributed to prolonged evaporation over time, which resulted in a smaller volume of filtrate and subsequently lowered the recovery yield. According to Hafni [31], the decline in recovery is caused by extended dissolution time, which increases the likelihood of solvent evaporation, especially when combined with agitation—both of which significantly influence the evaporation process. Another study [32] also reported a decrease in recovery, which was attributed to the formation of lead sulfate compounds. These compounds create a barrier, requiring the metal ions to diffuse through the lead sulfate layer before dissolving into the sulfuric acid solution. Leaching time strongly controls manganese recovery. The other research show extraction rises quickly at short times, then the curve flattens once easily accessible phases are exhausted, diffusion/product-layer or reagent-depletion limits set in. Optimal reported in the literature are typically on the order of tens of minutes to 2 hours depending on ore, temperature, acid and molasses dosages [24]. The Mn recovery obtained in this study does not align with previous research findings that suggested longer leaching times would lead to increased manganese recovery.

4. CONCLUSION

Based on the results of the study, it can be concluded that the dissolved Mn recovery at leaching times of 90, 120, 150, and 180 minutes were 14.404590%, 14.4221077%, 14.4173441%, and 14.4244638%, respectively. Multiple reductive-leaching studies report a steep increase in Mn extraction early in the experiment and then a levelling-off (plateau) as time increases. This pattern is consistent with surface reaction control followed by diffusion/product-layer or reagent-limit control. These findings indicate that the optimum leaching time for achieving the highest dissolved Mn recovery is 120 minutes.

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