Space Resources Engineering: Ilmenite Deposits for Oxygen Production on the Moon

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Abstract This decade is an incredible stage in the history of humanity. Man will return to the Moon to stay there. To successfully fulfill this enormous challenge, scientific staff will use the in-situ resources or ISRU, and terrestrial mining industry knowledge to produce prime matters. Oxygen and hydrogen are the crucial resources to obtain water, rocket fuel, and establish commercial activities between the Earth and the Moon. One alternative to produce oxygen is using metallurgical processing of oxide minerals as ilmenite. Also, iron and titanium could be produced from ilmenite to supply a future lunar aerospace industry, making attractive the exploration of this mineral. This paper discusses the ilmenite deposit features in the equator zone's Marias, especially in Mare Tranquillitatis. The author reviews the feasibility of producing oxygen from the ilmenite in a pyrometallurgical process, reviewing the reactions proposed through thermodynamic calculation with the Software HSC Chemistry 6.0 and the regolith sample’s geological data Apollo 11 to verify ore features that can affect the metallurgical behavior. Besides, there is a summary of the challenges to processing ilmenite associated with the degree of liberation. This research aims to clarify the industrial opportunities and challenges to produce oxygen on the Moon as a framework for future process architecture and equipment design.

Keywords: ISRU, space resources, space mining, lunar ilmenite

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1. Introduction

Several nations plan to return to the Moon this decade, including United States, China, Russia, the European Union; also, private companies such as SpaceX, Ispace, among others, want to establish commercial activities in the cis-lunar space [1]. However, the future of lunar colonization will depend directly on our species’ ability to produce commodities far from the Earth [2]. Space mining begins with the prospection stage, which is the mapping and characterization of resources. The target resources explored could be used in the future for Moon exploration, scientific endeavors, and economic activities outside of Earth (i.e., fuel production, life support consumables, construction, agriculture, fertilizers, etc.). Finally, collected materials could be transferred to Earth as commercial products [3]. The first step for colonization is accessing oxygen and hydrogen resources from water for life support and fuel production for spacecraft to establish continuous transport between the Moon and Earth. Oxygen could be produced in equatorial zones by processing lunar minerals. One of these minerals is the Ilmenite, and Mare Tranquillitatis is where remote sensing instruments found the highest ilmenite signs. [3]. This region has 873 km in diameter [4]; it is about 300,000 Km² [5]. The lunar TiO₂ highest values were found on its surface, as shown in Figure 1 and Figure 2. Most of its signs belong to Ilmenite because other titanium oxides, such as rutile, spinel, and armacolite [6], were found in Apollo samples everywhere in tiny quantities.

Figure 1. TiO₂ global map, nearside, and farside of the Moon [7]

Cameron [5] argues that an enormous impact formed Mare Tranquillitatis during the Pre-Nectarian lunar geological stage (4 billion years ago). At the time of Early Imbriam (3 billion years ago), the region was flooded by basaltic lava flows and pyroclastic glass. In the beginning, the lunar minerals were as single particles, but the surface suffered meteorite impacts for millions of years; impacts caused a deformation effect called shock metamorphism
on rocks and minerals, producing fusion between particles, glasses production, and modifying the regolith mineralogical composition. This effect grounded the lunar rocks.

**2. Materials and Methods**

Table 1. Characterization of the regolith. Data summarized from [9-16]

| Element  | Regolith | Rock 10085 | Minerals | Regolith | Rock 10085 |
|----------|----------|------------|----------|----------|------------|
| SiO₂     | 42,1     | 40,2       | Ilmenite | 6,7      | 12,8       |
| TiO₂     | 7,7      | 11,9       | Pyroxene | 33,3     | 49,3       |
| Al₂O₃    | 13,5     | 7,5        | Plagioclase | 16,2    | 32,3       |
| FeO      | 15,2     | 20,4       | Olivine  | 1,3      | 0,0        |
| MgO      | 8,0      | 8,1        | Cristobalite | 0,4    | 2,8        |
| CaO      | 12,0     | 10,2       | Glass(Al rich) | 27,6   | -          |
| Na₂O     | 0,5      | 0,5        | Glass(Ti rich) | 6,2    | -          |
| Others   | 1,1      | 1,3        | Glass(Fe-Mg rich) | 5,5   | -          |
| Helium-3 | 50-80 ppb/m³ | Others | 2,8      | 2,8       |

**3. Results and Discussion**

**3.1. Thermodynamic Evaluation**

Taylor et al. in [8] present three processes to produce water and oxygen, based on reducing the Ilmenite by hydrogen, methane, or carbon monoxide, showed in the stoichiometric equations 1 - 7.

- **Reduction by Hydrogen**
  \[ \text{FeTiO}_3(s) + \text{H}_2(g) = \text{Fe}^0(s) + \text{TiO}_2(s) + \text{H}_2\text{O} \]  

- **Reduction by Carbon Monoxide**
  \[ \text{FeTiO}_3(s) + \text{CO}(g) = \text{Fe}^0(s) + \text{TiO}_2(s) + \text{CO}_2(g) \]

- **Reduction with Methane:**
  \[ \text{FeTiO}_3(s) + \text{CH}_4(g) = \text{Fe}^0(s) + \text{TiO}_2(s) + \text{CO}(g) + 2\text{H}_2(g) \]

Using the software HSC Chemistry Version 6.0, the author calculated the required temperature for the Ilmenite reduction when the Gibbs energy is negative [20], which means the reaction is able to occur. All these reactions are not spontaneous and need external power to increase the temperature between 500 - 900°C. It would be feasible during lunar days using sunlight and nuclear power system. Figure 3 and Figure 4 shows that the process with Carbon Monoxide (CO) requires less temperature of the three gases (over 500°C), which means less power for the system. Nonetheless, the sequential reaction to recover the oxygen from CO₂ needs more energy of the three. The second gas that requires less temperature is the Ilmenite - methane that can start from 600°C. However, the sequential reaction to producing water from the CO produced with the methane requires a freezing stage below 500°C and finally a third sequential reaction to recover the oxygen from water. Finally, and easier to manipulate is the Reduction Ilmenite - hydrogen that requires temperatures up to 900°C. Also, the regolith in the equator Marias has hydrogen in significant quantities that can be recovered during heating [3]. This is the process more developed.

Besides, for reaction to happen, Ilmenite requires increasing their content from 10% to 90%, as Gibson and Knudsen explained in [21]. The metallurgical techniques have not succeeded in this task. To solve this challenge, we use geometallurgical analysis to interpret initial metallurgical results and the degree of liberation, a critical feature mentioned by Lishchuk et al. in [22] and commonly used on concentrator plants, according to Manzaneda in [23]. The chemical composition data is analyzed to predict Ilmenite's behavior with “atomic inclusions oxides” on pyrometallurgical processes.
3.2. Regolith Size Distribution

Concentrating an ore requires adequate liberation of the ore from the gangue (not economically minerals). For that reason, terrestrial mining applies various comminution operations like crushing, milling, and classification of previous physicochemical or extractive processes. To measure if these natural processes have produced enough liberation for concentration, it needs first to measure the regolith's size grains. Figure 5 summarizes 15 granulometric tests applied to the sample N° 10084 of Mare Tranquillitatis.

On average, the size distribution has a K65 (65% undersize) of 101 micrometers [20]. Regolith has a similar distribution to the overflow in a secondary milling-classification stage of a concentrator plant on the Earth. This feature's advantage is that the regolith would not require drilling activities in the extraction stage of comminution previous to the concentration, reducing the operatives' processing costs [20]. The disadvantage is that the regolith is so small that neither process could be feasible to free the mixed particles; even if we designed a cheap strategy for milling the regolith, many ilmenite particles already appear mixed in a tinny size, impossible to liberate (about 10 micrometers), as shown in Figure 6 (Particle e, g, and f).
3.3. Degree of Liberation

Table 2 shows the degree of liberation (DL) and its influence on metallurgical behavior. Previous authors applied a magnetic separation test using two samples: A) basalt rock crushed and screened to a regolith size, and B) regolith from Mare Tranquillitatis. Sample A (DL = 78) produced a concentrate with an acceptable grade (62.2%) with a low recovery (39%); in contrast, the concentrate of Sample B (DL = 37) has a low grade (23.5%) with tinny recovery (9%). The details are in Table 2. It seems like the magnetic effect cannot discriminate between the Ilmenite and regolith because the ore is in mixed particles (as can see in Figure 6), and by the magnetic FeO content in the glasses and agglutinates, for that reason, about 91% of the ore was lost in the tail, and the concentrate was dirty.

Sample A could choose some options to improve their recovery, increasing the residence time, but sample B does not have many alternatives to improve the grade or recovery through magnetic separation significantly. Another technique for concentration is electrostatic separation. Test C used this technique in a simulant with Ilmenite pure (DL = 100). The recovery is higher than other conditions (68%), with an ilmenite concentrate near pure (grade of 95%). A similar process was applied to a real regolith (test D), and the results dropped (Recovery: 24%, Grade: 45%). The considerable difference between the results makes regolith simulants impractical to predict metallurgical results in the processing stage. On the other hand, if we combine both techniques (magnetic + electrostatic), the recovery can increase up to 60% (Test E) with a low-grade concentrate (37%). We can increase the quality up to 51% if we use a vacuum environment instead of nitrogen (Test F), but with a recovery of 48%.

Table 2. Summary of concentration test for ilmenite \[16,32,33\]

| Sample     | Technique                  | Size  | Feed %w Ilmenite | Grade %w Ilmenite | Recovery (%) | DL |
|------------|----------------------------|-------|------------------|-------------------|--------------|----|
| A.Basalt 10058 | Magnetic                  | 45-90 | 18.5             | 62                | 39.0         | 78 |
| B.Regolith 10084 | Magnetic                | 45-74 | 10.2             | 95                | 8.8          | 37 |
| C.Simulant | Electrostatic              | 90-150| 10               | 45                | 24.0         | No data |
| D.Regolith 10084 | Electrostatic            | 90-150| 7                | 37                | 60.0         | No data |
| E.Regolith 10084 | Magnetic + Electrostatic | 90-150| 7.3              | 51                | 48.0         | No data |
| F.Regolith 10084 | Magnetic + Electrostatic | 90-150| 7.3              | 51                | 48.0         | No data |

Figure 5. The author calculated the size distribution of the regolith from 15 previous granulometric tests in \[3,25-31\]

Figure 6. Microscopic specimens of particles in the regolith. A: Ilmenite; B: Olivine; C: pyroxene ; D: plagioclase; E: basalt; F: agglutinate; G: breccia; H: glass. Picture from \[18\]
The metallurgical test results are comparing with the microscopy photographs in Figure 7. It can confirm that the Ilmenite in the basalt is freer than the Ilmenite in the regolith. Furthermore, ilmenite deposits should be classified according to their DL to predict their real availability. For instance, in a sample with 10% of Ilmenite but with a DL of 10%, only 1% of ore is available. Instead, for a sample with 5% of Ilmenite but a DL of 80%, 4% of ore is available. Apply the degree of liberation will make more accurate the evaluation of the deposit.

Chambers et al. in [16] propose using the soil maturity index (Is/FeO) to predict the degree of liberation and Is/FeO in the Apollo 11 and 17 samples. Nevertheless, previously it needs to make many microscopy analyses in different places to correlate both variables and predict future mining exploitation zones.

4. Conclusion

In the last century the metallurgical process had advanced using new technologies from the optimization of the process as hydrometallurgy, pyrometallurgy to the use of bacteria to process minerals [34,35]. On the other hand, many medical technologies in the next years can be prepare to the humanity to travel and colonize other worlds permanently [36,37]. Now, it is the time to apply all this knowledge in develop process in the space for the future of the humanity. Mare Tranquillitatis as an excellent soil to be processed since it is a fine grain size material. This material would also be easy to mine, transport, and also, it does not need crushing stages or grinding. The Ilmenite is a mineral feasible to process for obtain oxygen. However, there is a need to evaluate the deposit, including a geometallurgy approach to identify the real challenges. One of them is that the fine Ilmenite is hard to concentrate from the regolith up to an acceptable grade because other minerals lock its particles. The degree of liberation is intrinsic to the regolith; therefore, although it will test many processing techniques, the results will be directly influenced by the degree of liberation, and it would not be feasible to apply current grinding technologies to increase it. For that reason, further studies are required with a focus on the degree of liberation and obtain information to select the best range size of particles. This knowledge will assist in selecting the correct location in the deposit to mine, the right size range to process, and an accurate metallurgical architecture to maximize the operative results and, therefore, the profitability.

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