

Separation Of DR Synthesized Ti-Mg Alloy By Dissolution Of Waste By-Products In Acid Media.

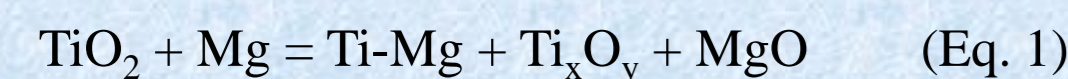
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INTRODUCTION

Ambient temperature direct reduction (DR) of TiO₂ powder with Mg can be used to synthesise Ti-Mg alloy powders in Mechanochemical Processing (MCP) (1). Ti-Mg alloys have high strength and low density.



The objective of this work is to isolate synthesized Ti-Mg alloy powder through leaching out of MgO and any unreduced oxides of Titanium.

Leaching media investigated are H₂SO₄, HCl (2) and organic acids ascorbic acid/oxalic and L-cysteine/ascorbic acids in equi-molar concentrations (3). The use of the test leaching media will be counter balanced by the need not to dissolve the Ti-Mg alloy formed during milling.

METHODS

The dissolution experiments were done in a Julabo SW23 waterbath shaker, with 300 ml flasks. Acid solution of predetermined molarity was charged into a flask and heated to the required temperature before initiating a leach run.

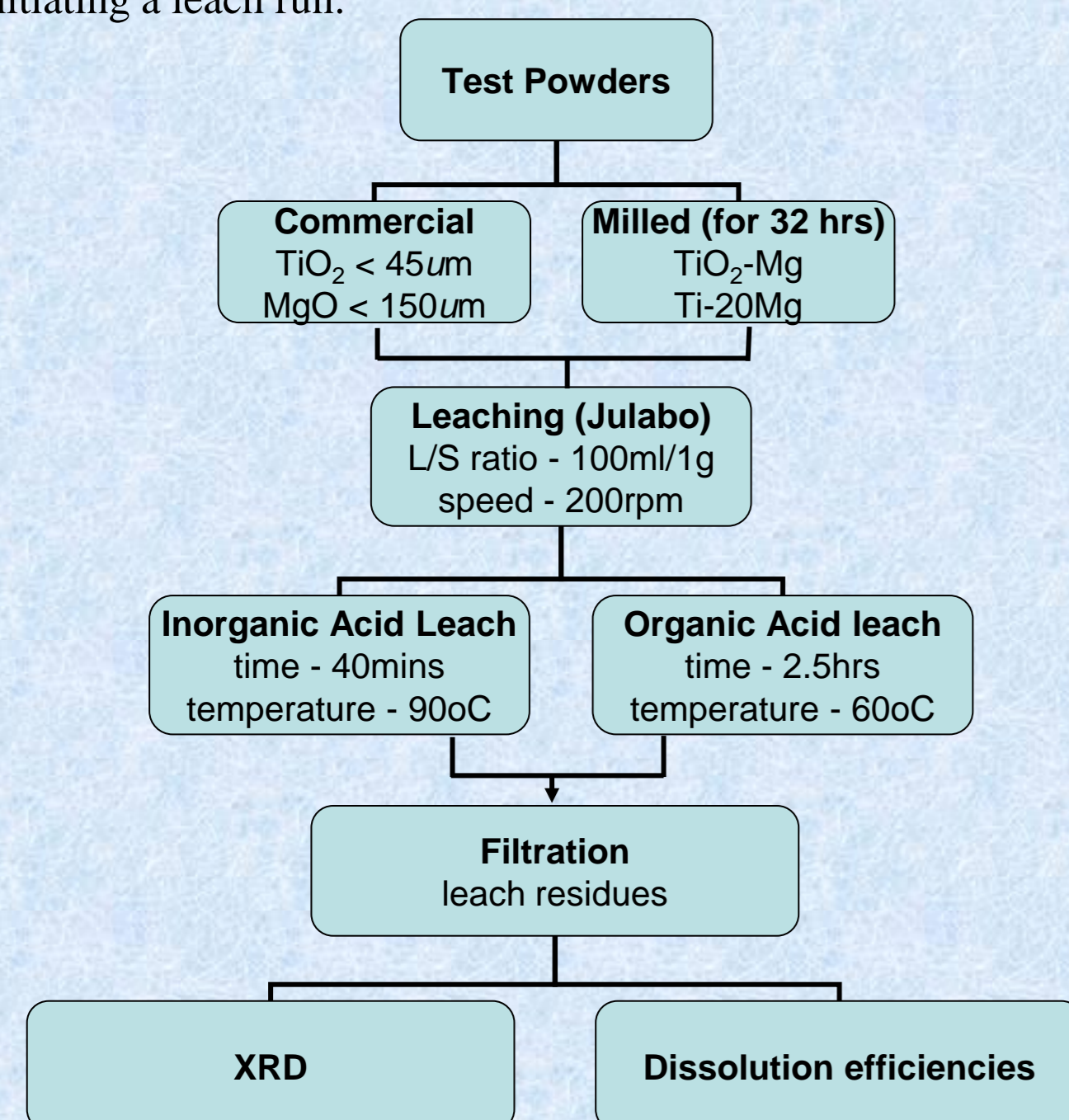


Figure 1: Flowchart of the leaching processes

- Separate TiO₂, Ti-20Mg alloy and MgO powders were leached in the same conditions as TiO₂-Mg to:-
- investigate how they would behave in the leaching reagents reviewed.
- note how well the Ti-Mg separation process could be achieved by each reagent.

Proportion of Ti-Mg synthesized.

The extent of the reduction of TiO₂ by Mg was evaluated from the following equation:

$$D = \left(\frac{I(\text{TiO}_2)}{I(\text{TiO}_2) + I(\text{MgO})} \right) \times 100 \quad (\text{Eq. 2})$$

where I is the intensity of the diffraction peaks of Mg and TiO₂. The reduction of TiO₂ at 32hrs of milling was evaluated from Equation 6.2 to be 90.4% (4). Calculated proportions of products, and the respective masses from a 2g sample, are shown in Table 1.

Table 1: Calculated compositions of the milled powder at 32 hrs of milling.

Constituent	TiO ₂	Ti-Mg	MgO
Weight %	5.7	40.7	53.7
mass from 2g samples	0.114	0.814	1.07

RESULTS

Leaching in different acid media

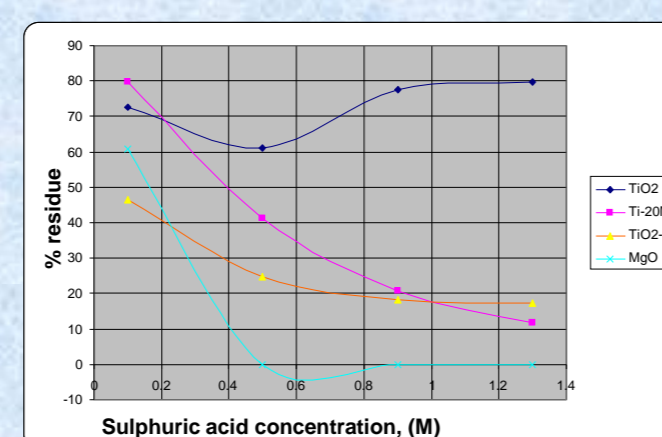


Figure 2: Effect of H₂SO₄ concentration

Figures 2 and 3 above show the variation in solubility of powders with acid concentration and they show that:-

- The residue percentages for powders other than TiO₂ are generally indirectly proportional to the acid strength, with H₂SO₄ being more aggressive.
- Same pattern occurs with TiO₂ in HCL but with less amounts of residue.
- For TiO₂ in H₂SO₄ acid, trend is broken at higher molarities where there were more residue proportions.

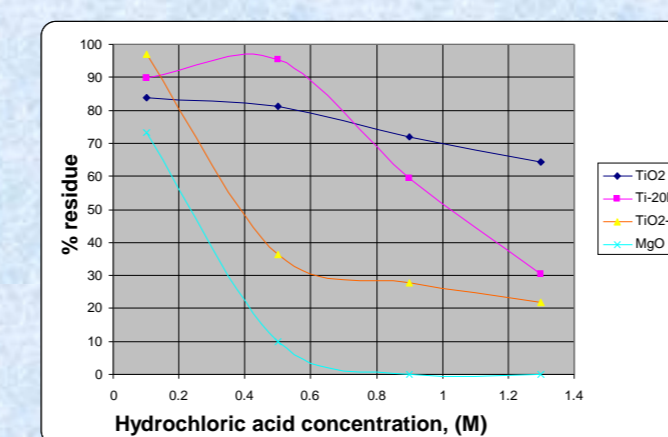


Figure 3: Effect of HCL concentration

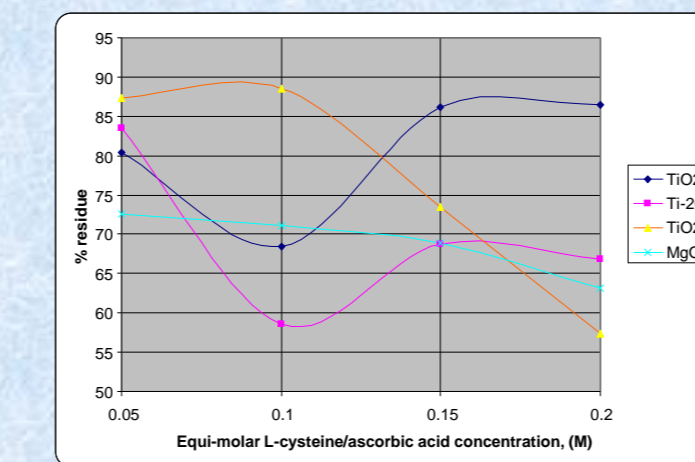


Figure 4: Effect of L-cysteine/ascorbic acids concentration

Figures 4 and 5 represent the dissolution behaviour in equimolar L-cysteine/ascorbic acids, and in combined HCL and equi-molar L-cysteine/ascorbic acids, respectively

- TiO₂-Mg and MgO dissolution proportional to concentration.
- Dissolution of TiO₂-Mg increased on either side of 0.1M with L-cysteine/ascorbic acids.
- more TiO₂ and Ti-20Mg leached at lower molarity in cysteine/ascorbic acids, trend reversed with increase in acid strength
- TiO₂ and Ti-20Mg residues fluctuate around 80% in combined organic/inorganic acids.

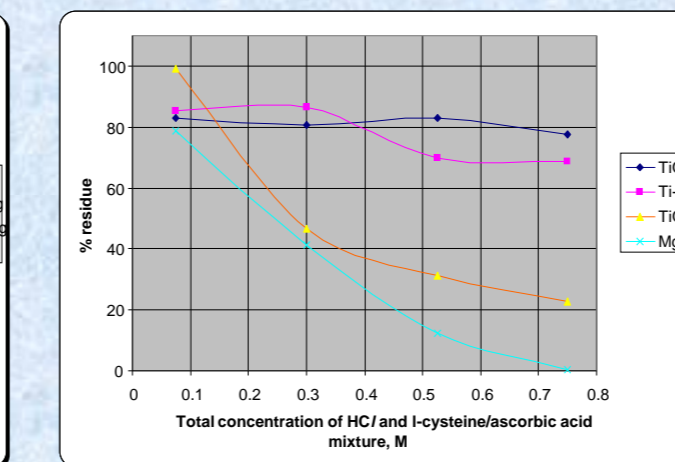


Figure 5: Effect of combined HCL and equi-molar L-cysteine/ascorbic acids

DISCUSSION

Leaching experiments were conducted using high L/S ratios (100ml/1g) to decrease mass transfer resistance in the acid-powder particle interfaces (5) and to achieve maximum acid exposure to each constituent in a given powder.

A.Sulphuric acid, and hydrochloric acid

TiO₂ showed marked resistance to dissolution (at higher molarity for H₂SO₄) in both acids. This was probably due to hydrolysis of Ti ions back to TiO₂ (6).

Other powder species (MgO, T-20Mg and TiO₂-Mg) show steady exponential dissolution to critically low levels. Thus H₂SO₄, HCL acid isolation of the Ti-Mg alloy powder is not viable.

B.L-cysteine/ascorbic acids, Combined HCL and equi-molar L-cysteine/ascorbic acids.

Slightly over 30% TiO₂ dissolved in equi-molar L-cysteine/ascorbic at low acid concentration. However, at this point, MgO and TiO₂ are still substantially present. Thus it is difficult to isolate a clean Ti-Mg alloy from the milled TiO₂-Mg mixture with the acid mixtures above.

CONCLUSIONS

MCP synthesized Ti-Mg alloy can not be effectively separated from the waste powders with the acids and acid concentration ranges utilised as TiO₂ proved difficult to dissolve.

Acid aggressiveness (molarity and temperature) can not be manipulated further as the investigated acid strengths have shown that Ti-Mg alloy is dissolving at levels higher than TiO₂ already. Increased acid strengths will only intensify this trend.

RECOMMENDATIONS

- Further work is recommended on the optimisation of the MCP process to ensure all TiO₂ is reduced in the first place.
- It is also recommended to explore and investigate other leach media combinations beyond the inorganic and organic acids.

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FURTHER INFORMATION

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