From Moon Rock to Cement:

Extraction of silica from serpentine and the use of waste acid to produce magnesium chloride cement

Nan Yang[1,2]. Allan Scott[1]. Matt Watson[3], Vineet Shah[1], Christopher Oze [4].

[1] *Department of Civil and Natural Resources Engineering, University of Canterbury, Christchurch, New Zealand*
[2] *Advance Material Group, Callaghan Innovation, Wellington, New Zealand*
[3] *Department of Chemical and Processing Engineering, University of Canterbury, Christchurch, New Zealand*
[4] *Department of Geology, Occidental College, Los Angeles, California, United States*

1. QUICK FACTS & QUESTION

- Olivine are typically seen in the lunar mantle [1] and occurs all over Mars [2]; Olivine can form serpentine when interact with water[3].
- Serpentine were one of the most feasible minerals for producing pozzolana by chemical treatment[4], due to its high content of SiO_2 and simplicity in structure.
- Acid was commonly used in serpentine leaching[5] for the extraction of nickel and magnesium.
- **Can the dissolved magnesium salt (such as $MgCl_2$) and the insoluble residual (mainly SiO_2) be tailored for applications in cement?**

2. MATERIALS & METHODS

Sample powder **Hydrochloric acid** **Acid digestion** **Vacuum Filtration**

- Powder particle mean size: 12µm.
- Chemical composition:

	SiO_2	MgO	$Fe_2O_3^T$	L.O.I
%	40.7	37.27	7.84	12.01

- Hydrochloric acid (2mol/L), pH= -0.3

- Simplified reaction: $Mg_3Si_2O_5(OH)_4 + 6HCl = 3MgCl_2 + 2SiO_2 + 5H_2O$
- Stirring speed= 120 rpm
- Solid /Liquid ratio=1:20.
- 1atm, 20°C for 24 hours.

- Filter size=0.45µm
- The filtration separated the solution to permeate and retentate.
- The permeate is collected for analysis.
- The retentate was then washed and dried.

Permeate: (liquid: waste acid)

Strength test

- 40ml permeate/water was mixed with MgO, respectively.
- Sample size: 20*20mm

Retentate: (solids: digested serpentine)

Conductivity test:

- 4g solid sample +80ml $Ca(OH)_2$ solution (0.8g/L)

Strength activity test

- 20% replacement of the PC.
- Water/cement=0.48, Sand/cement ratio=2.75

3.1 PRODUCING SOREL CEMENT FROM WASTE ACID

1. What is in the solution?

- ICP-MS results showed it was mainly consist of Mg^{2+}, Fe^{3+} and Cl^-.

	Mg	*Fe*	*Ca*	*Si*	*Cl**
Ion Concentration (mg/L)	*996.5*	*128.5*	*62.0*	*57.5*	*70900**

- After evaporating, $MgCl_2 \cdot 6H_2O$ was the main phase left in the solution(*Fig.1*).

2. How it behaves in strength?

- Sample with the waste acid demonstrated higher compressive strength than MgO mixed with water(*Fig.2*).

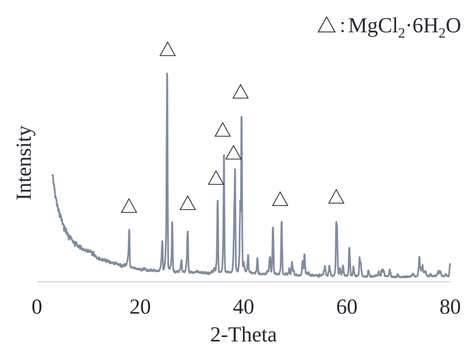

Fig.1 XRD of the products recovered from the solution

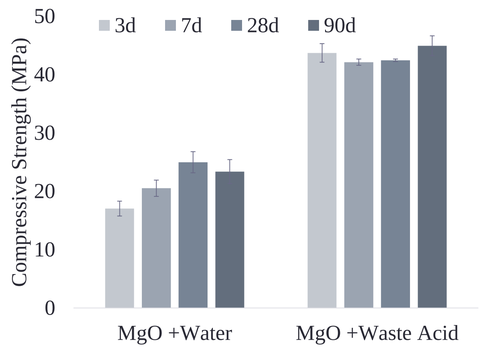

Fig.2 Strength of the samples with different mixture

3. What does it look like?

SEM pictures shows the differences between the hydration products:

- Fig.3(a) showed the formation of the brucite in the mixture of MgO+water;
- The hydration products formed in Fig.3(b) was very similar to the hydrate phases found in magnesium oxychloride cement[6] (Sorel cement: MgO-$MgCl_2$-H_2O).

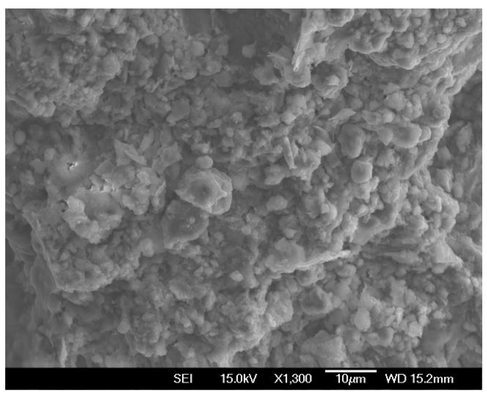

Fig.3(a) SEM of MgO + water at 3 days

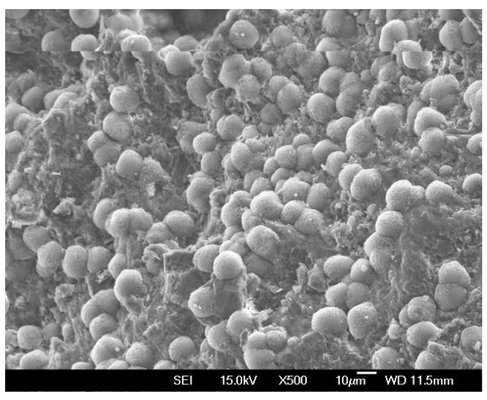

Fig.3(b) SEM of MgO +waste acid at 3 days

3.2 POZZOLAN REACTIVITY OF THE SOLIDS

1. Conductivity and BET surface area test.

- The digested serpentine demonstrated highest/fastest $Ca(OH)_2$ consumption compared to the unprocessed sample and silica fume (*Fig.4*).
- The reduction of the conductivity can be attributed to the formation of the C-S-H.
- It also showed highest surface area due to the dissolution of Mg^{2+}, Fe^{3+} (*Fig.5*).
- These results suggested a high potential silica reactivity of the digested sample.

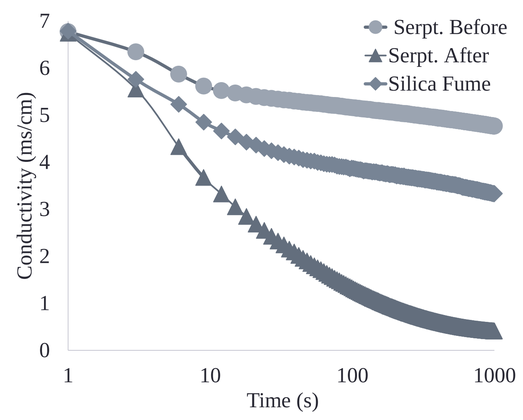

Fig.4 Conductivity change of the CH mixed with samples

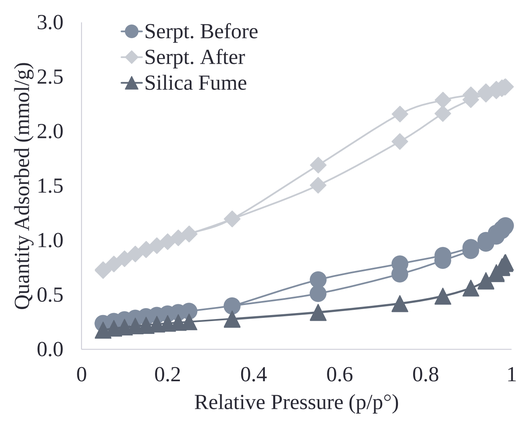

Fig.5 Absorption and desorption isotherms

2. XRD analysis and strength activity test

- XRD identified unreacted serpentine and the board peaks 20~30° suggested the presence of amorphous silica in the digested serpentine sample (*Fig.6*).
- However, the digested sample didn't show significant strength improvement and the value was lower than silica fume reference (*Fig.7*).

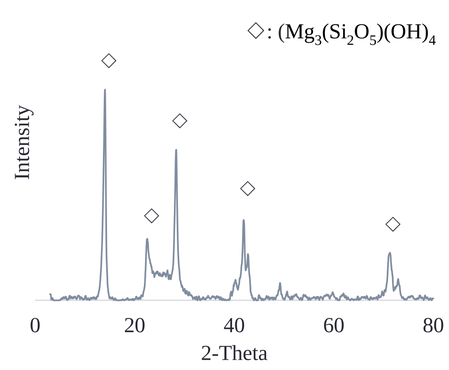

Fig.6 XRD pattern of the digested sample

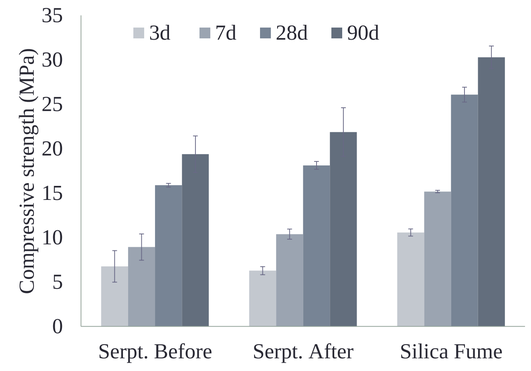

Fig.7 Strength activity test

4. CONCLUSIONS

This is a feasibility study of producing cementitious materials from serpentine, which can be obtained from the reaction of olivine with water.

- After the serpentine was digested in HCl, Mg^{2+} and Cl^- were the main ions in the solution and $MgCl_2 \cdot 6H_2O$ was the product left after evaporation.
- The mixture of MgO+ waste acid showed higher strength compared to MgO+ water, and the hydration products with waste acid were similar to Sorel cement.
- The conductivity test indicated the higher $Ca(OH)_2$ consumption of the serpentine solid residual while it also demonstrated greater surface area.
- Amorphous silica was found in the solid residual by XRD, however the strength activity test didn't prove its high silica reactivity which need further investigation.

5. REFERENCES

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