GROUP14: SILICA RECYCLING

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BACKGROUND

AT GROUP 14, EXCESS SILICA POWDER IS A COMMON MANUFACTURING BYPRODUCT OF THE TRADEMARKED SILIGENESIS PROCESS.

Group14, an advanced materials manufacturing company, produces a silicon-carbon powder (SCC55), an anode material that improves energy density and stability in lithium-ion batteries. The production process generates amorphous silica powder as a byproduct. Currently, Group14 disposes of the silica as waste, carrying negative economic and environmental consequences. Group14 has requested that the capstone team characterize the material, identify an application for the silica, and design a feasible processing procedure to meet required material specifications.

METHODS

THE TEAM IDENTIFIED TARGET MARKETS FOR THE SILICA AND USED INDUSTRY STANDARDS TO INFORM CHARACTERIZATION WORK.

Characterization Techniques:

> **X-Ray Diffraction (XRD):** crystalline structure, contaminant identification
> **Brunauer–Emmett–Teller (BET):** porosity & specific surface area
> **X-Ray Fluorescence (XRF):** metallic/inorganic contaminants
> **Total Reflection X-Ray Fluorescence Spectroscopy (TXRF):** trace contaminants
> **Scanning Electron Microscopy (SEM):** surface morphology
> **Energy-dispersive X-ray spectroscopy (EDS):** chemical composition

Based on initial industry research, target markets were narrowed down to the most potentially profitable applications. The team then contacted manufacturers to obtain details of in-house silica to compare to characterization results. Despite initial attempts to design a silica purification system, it was found to be highly energy intensive and would bring insignificant profits to Group 14 during the research. Instead, from these results, a decision tree was used to propose multiple approaches to recycle the silica based on the variety of the powder's composition and properties.

RESULTS

THE TEAM CHARACTERIZED THE MATERIAL PROPERTIES OF THE SILICA AND COMPARED THEM TO TARGET MARKETS (TABLE 1).

> Elemental analysis conducted through XRF, TXRF, and EDS indicated a slight calcium contamination and trace amounts of heavy metal contaminants. It should be noted that the EDS results may be compromised due to charging of the sample.

> XRD designated the compound as amorphous, and suggested the presence of amorphous or crystalline silicon in the sample (Figure 1).

> The specific surface area obtained from BET remained consistent within each batch, but the third batch exhibited a much larger surface area than the previous two. This indicates an inconsistency in a material property that affects potential applications.

#	Phase ID	Chemical Formula	PDF-#	RIR	Wt%
1	Cristobalite	O_2Si	98-900-8226	4.91	83.9
2	Silicon	Si	98-901-3108	4.74	16.1

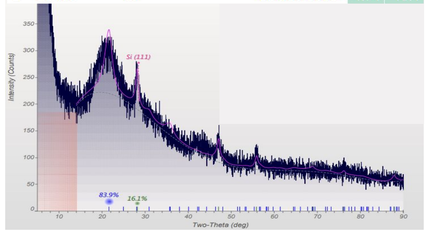

Figure 1: XRD Spectra for Batch 1 silica sample

Table 1: Material Specifications for Each Batch

Batch Number	Characterization Method								
	BET	XRF		EDS		DLS	XRD		Density
	Surface Area (m^2/g)	Element	Mass %	Element	Atomic %	Weighted Avg. Particle size (nm)	Compound	Wt. %	g/mm^3
Batch 1	71.46 ± 1.74	Si	44.66	Si	35.94 ± 3.58	544.90	SiO_2	83.9	0.752
		Ca	0.41	O	64.06 ± 3.58		Si	16.1	
Batch 2	74.61 ± 1.57	Si	42.36	Si	31.18 ± 0.99	481.88	SiO_2	53.2	0.649
		Ca	1.22	O	68.82 ± 0.99		Si	46.8	
Batch 3	102.67 ± 1.61	Si	24.75	Si	35.57 ± 2.07	458.49	SiO_2	100	0.992
		Ca	0.16	O	64.43 ± 2.07		N/A	N/A	
Total Avg.	82.91 ± 1.64	Si	37.26	Si	34.23 ± 2.22	495.09	SiO_2	79.03	0.798 ± 0.176
		Ca	0.60	O	65.77 ± 2.22		Si	20.97	

DISCUSSION

THE PROVIDED SILICA APPEARS TO HAVE UNEXPECTED SILICON, WHICH LIMITS SOME PROPOSED USES.

> A few key results led to the conclusion of unexpected elements in the silica powder:
1. The unique brownish-grey colors of all three batches
2. XRD indicating traces of silicon (111) peak (Figure 1)
3. XRF analysis picked up traces of calcium

> Our current theory is that the byproduct has a core shell structure with crystalline silicon in the center and silica powder coating the exterior.

> Contamination in the silica powder can potentially disqualify it from multiple applications that require >99% purity, such as rubber filler and paint filler. This would require either removing the contaminant or finding new applications.

> Each batch had varying purity from characterization, preventing consistent use of the silica for certain applications.

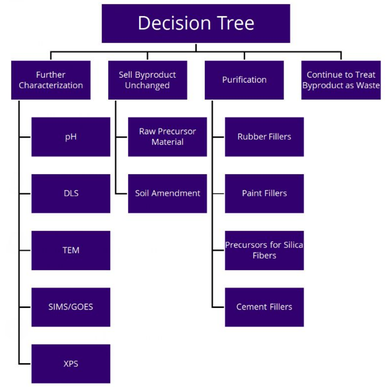

Figure 2: Decision tree depicting potential silica recycling options considered in this project

Figure 3: Image of Silica Powder, Batch 1

NEXT STEPS

WE RECOMMEND FOUR POSSIBLE COURSES OF ACTION FOR GROUP14 MOVING FORWARD:

REQUIRED FURTHER CHARACTERIZATIONS

For commercial recycling, these final two characterization methods are required before selecting an application:
> pH
> Repeat DLS due uncertainty regarding measurement

SELL AS SOIL AMENDMENT

At present, the raw byproduct can be sold as a soil amendment for hydroponic and fertigation applications. The low purity requirements and benefit of metal oxides make this our most recommended application. Companies such as Miracle-Gro work with suppliers to source raw material and continue processing in-house. This process would require Group14 to receive supplier certification, which may be difficult due to batch-to-batch variation.

SELL AS RAW PRECURSOR

We also recommend the silica to be sold as a raw precursor for use in rubbers, concrete, and paint. Companies such as 3m or Evonik utilize an interface called Coupa Cloud to interact with potential suppliers. This may require minor processing for Group14 (such as washing to remove impurities) but we anticipate little in-house purification. While less environmentally favorable as soil amendment, this application would bring additional profit to Group14 and improve waste stream recycling overall.

OPTIONAL FURTHER CHARACTERIZATIONS

If desired, to confirm the possible silicon crystal structure Group14 should utilize the following characterization techniques:
> TEM
> Run standardized silica sample to validate XRD method
> SIMS/Glow Discharge Emission Spectroscopy
> XPS + Sputtering

REFERENCES

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