GROUP14: SILICA RECYCLING

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BACKGROUND

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

Group14, an advanced materials manufacturing company, produces a silicon-carbon powder (SC55), 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): metal/metalloid 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 material specifications.

RESULTS

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

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.
2. XRD designated the compound as amorphous, and suggested the presence of amorphous or crystalline silicon in the sample (Figure 1).
3. 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.

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.

We recommend four possible courses of action for Group14 moving forward:

1. WE RECOMMEND FOUR POSSIBLE COURSES OF ACTION FOR GROUP14 MOVING FORWARD:
   - REQUIRED FURTHER CHARACTERIZATIONS
     - WE RECOMMEND PERFORMANCE OF ADDITIONAL CHARACTERIZATION TECHNIQUES:
       - SIMS/Glow Discharge Emission Spectroscopy
       - XPS + Sputtering
   - 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, along with the benefits 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 minimal 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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ACKNOWLEDGEMENTS

Table 1: Material Specifications for Each Batch

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<tr>
<th>Batch</th>
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<th>XRF</th>
<th>SEM</th>
<th>XPS</th>
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Figure 1: XRD Spectra for Batch 1 silica sample

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

Figure 3: Image of Silica Powder, Batch 1