

Structure Characterization of Waste-Glass Derived Silica Gel using X-Ray Diffractometer

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ABSTRACT: This study utilizes XRD analysis to unveil the structural characteristics of silica gel synthesized from waste glass. While the dominant broad halo confirms its amorphous nature, closer examination reveals the presence of minor crystalline phases contributing to peaks at 8.9° and 43.0°. The 8.9° peak is tentatively attributed to aluminosilicate impurities like mullite, based on the waste glass composition. The origin of the 43.0° peak remains ambiguous, requiring further investigation. This work emphasizes the potential of XRD in elucidating the subtle structural complexities within seemingly amorphous materials, offering valuable insights into the short-range order and potential heterogeneity of waste-derived products.

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I. INTRODUCTION

The valorization of waste glass for the production of silica gel has gained attention due to the growing emphasis on sustainability. Waste glass, with its abundance and diverse composition, presents a promising resource for the synthesis of valuable materials like silica gel, known for its exceptional adsorptive properties [1]. The structural characteristics of waste glass-derived silica gel have been explored using X-ray diffraction (XRD) analysis, revealing the presence of minor crystalline phases, which offers insights into the origin and potential influence of impurities, paving the way for optimizing silica gel for specific applications [2].

The study of waste glass dissolution and the extraction of silica for the production of silica gel is crucial for understanding the potential influence of impurities and optimizing the material for specific applications [3]. Additionally, the addition of hardeners from waste materials to the soda-lime-silica glass solution has been proposed as a viable and cleaner solution to optimize the reactivity of chemical ingredients from waste glass, thereby improving the sustainability of geopolymer cements, mortars, and concretes[4].

This research aims to investigate the structural characteristics of silica gel synthesized from waste glass using X-ray diffraction (XRD) analysis. We focus on identifying and understanding the crystalline impurities present in the material, as they might influence its properties and behavior. The findings would provide valuable insights into the potential of waste glass-derived silica gel and pave the way for further optimization and exploitation of this sustainable resource[5][6].

II. EXPERIMENTAL SETUP

A. Chemicals

Specifically, drinking trash made of clear glass bearing the Royalex brand was among the materials used in this investigation. Sulfuric acid and sodium hydroxide (NaOH), which were utilized without further purification, were supplied by Merck. The microwave heater used in the home operated at 2.45 GHz.

B. Silica Gel preparation

After being carefully cleaned and ground up, clear glass waste was combined with 4M NaOH in an Erlenmeyer flask and stirred with a magnetic agitator for three hours at 80°C. Subsequent 5-minute microwave treatment expedited the reaction. Cooled solutions were filtered to obtain sodium silicate solutions, then neutralized with H₂ SO₄ and incubated for 48 hours to form hydrogels. Drying at 80°C followed by activation at 500°C for 3 hours yielded the final silica gel products for further characterization.

III. RESULTS AND DISCUSSION

XRF ANALYSIS UNCOVERS SILICA

The XRF analysis of glass waste (fig. 1) reveals a silica content of 73.197%, making it an ideal source for gel silica production[7][8]. Glass waste also contains other oxides like aluminum oxide, phosphorus oxides,

and potassium oxides, with their content being relatively low. The XRF analysis results indicate that glass waste is a promising raw material for silica gel production, with high silica content in resulting in high-quality gel silica[9].

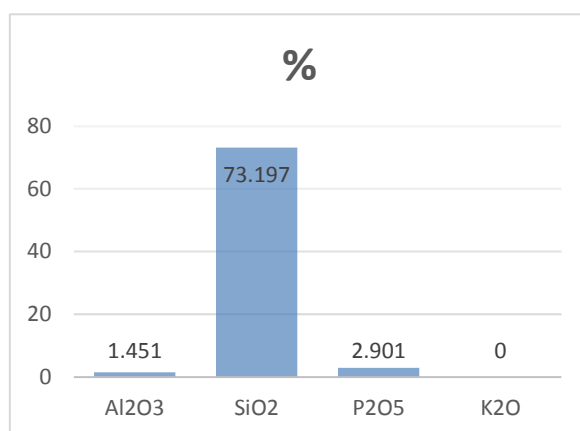


Fig. 1 Composition of Clear Glass Waste by XRF (traces elements omitted)

XRD ANALYSIS OF THE WASTE-GLASS DERIVED SILICA GEL

The result of the XRD spectrum of silica gel is found in the following Fig. 2.

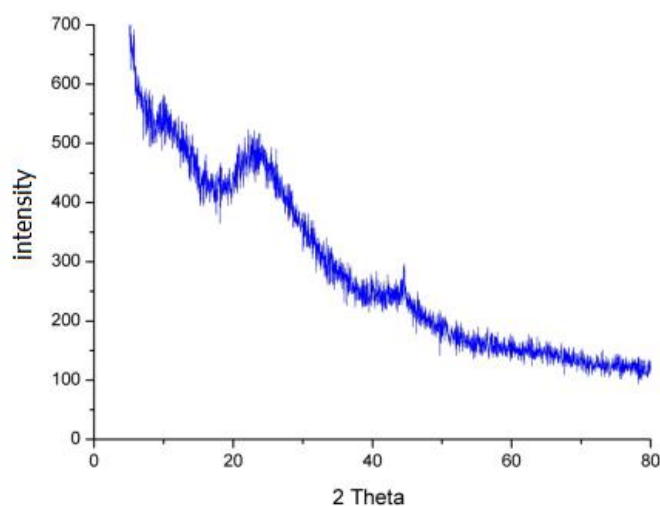


Fig. 2 XRD spectrum of silica gel synthesized

As expected for silica gel, the peak at around 22° is broad and diffuse, indicating an amorphous or poorly crystalline structure. This is consistent with the general characteristics of silica gel. The peak is relatively weak, further suggesting a low degree of crystallinity or long-range order within the material[10][11].

The peak position around 22° is characteristic of a broad halo often observed in the XRD patterns of amorphous silica gel[12]. This halo can arise due to the presence of short-range order or stacking of Si-O tetrahedra in various configurations[13].

It can be concluded that the silica gel prepared using the described method is predominantly amorphous. The broad, low-intensity peak at around 22° reflects the short-range order within the material and supports the lack of a well-defined crystal lattice[14]. While some degree of local ordering might exist, the overall structure is mainly characterized by the random arrangement of Si-O tetrahedra.

The peak present in the XRD image at 8.9°

Based on the presence of aluminum oxide (Al₂O₃) in the waste glass, the peak at 8.9° in the XRD image is likely attributed to crystalline impurities containing aluminum. Mullite (3Al₂O₃ · 2SiO₂) emerges as a potential candidate for this peak, as it exhibits a peak around 9° and is commonly found in glasses with high Al₂O₃ content [15]. Additionally, corundum (Al₂O₃) is considered a less likely candidate due to its high melting point, but it could still show a peak in this region if some alumina grains remained unreacted during

silica gel synthesis. While still possible, the presence of sodium silicate phases becomes less likely due to the presumably complete gel formation during your synthesis process.

The peak present in the XRD image at 43.0°

Determining the origin of the peak at 43.0° remains challenging. With the confirmed presence of Al₂O₃ and SiO₂ as major components, there's still a vast array of potential crystalline phases (especially aluminosilicates) that could exhibit a peak at this angle[16]. Identifying the phase at 43.0° likely requires further analysis, like Raman Spectroscopy. Raman Spectroscopy has been widely utilized for the characterization of materials, including minerals and mineral pigments, and has proven effective in phase identification based on structural order and composition[16].

IV. CONCLUSION

XRD analysis confirmed the primary amorphous nature of the synthesized silica gel, evident in the broad, diffuse peak at 22° characteristic of short-range order and random Si-O tetrahedral arrangements. Low peak intensity further underlines the limited crystallinity. Traces of crystalline impurities likely containing aluminum explain the peak at 8.9°, with mullite and corundum as probable candidates due to the waste glass composition. While sodium silicate phases are less likely due to complete gel formation, the origin of the peak at 43.0° remains elusive, potentially arising from various aluminosilicates. Identifying this phase via techniques like Raman spectroscopy is recommended for a comprehensive understanding of the material's composition.

REFERENCES

- [1] N. Valle, A. Verney-Carron, J. Sterpenich, G. Libourel, É. Deloule, and P. Jollivet, "Elemental and Isotopic (29Si and 18O) Tracing of Glass Alteration Mechanisms," *Geochim. Cosmochim. Acta*, 2010, doi: 10.1016/j.gca.2010.03.028.
- [2] L. Coppola et al., "Binders Alternative to Portland Cement and Waste Management for Sustainable Construction—part 1," *J. Appl. Biomater. Funct. Mater.*, 2018, doi: 10.1177/2280800018782845.
- [3] M. Torres-Carrasco, J. Palomo, and F. Puertas, "Sodium Silicate Solutions From Dissolution of Glasswastes. Statistical Analysis," *Mater. Construcción*, 2014, doi: 10.3989/mc.2014.05213.
- [4] S. J. K. Melele, C. Banenzoué, D. Fotio, H. K. Tchakouté, C. H. Rüscher, and C. P. N. Nansu, "Improvement of the Reactivity of Soda–lime–silica Glass Solution as a Hardener for Producing Geopolymer Materials," *Sn Appl. Sci.*, 2019, doi: 10.1007/s42452-019-1242-5.
- [5] V. A. Fabiani, "Sintesis Dan Karakterisasi Silika Gel Dari Limbah Kaca Serta Aplikasinya Pada Kromatografi Kolom," *Indones. J. Pure Appl. Chem.*, vol. 1, no. 1, p. 10, 2018, doi: 10.26418/indonesian.v1i1.26038.
- [6] R. M. Saputra, Rudiyanayah, and N. Wahyuni, "Sintesis Dan Karakterisasi Silika Gel Dari Limbah Kaca Termodifikasi Asam Stearat," *J. Kim. Khatulistiwa*, vol. 3, no. 3, pp. 36–42, 2014.
- [7] B. S. Indira and M. Khair, "Uji X-Ray Fluorescence Pada Limbah Kaca Bening Untuk Analisis Silika Dan Oksida Terkait," *CHEDS J. Chem. Educ. Sci.*, vol. 7, no. 1, pp. 92–96, 2023, doi: https://doi.org/10.30743/cheds.v7i1.7119.
- [8] M. Khair, B. S. Indira, and R. Salsabila, "Preparasi Silika Gel Dari Limbah Kaca Bening Dengan Bantuan Iradiasi Microwave," *CHEDS J. Chem. Educ. Sci.*, vol. 7, no. 1, pp. 97–102, 2023, doi: https://doi.org/10.30743/cheds.v7i1.7130.
- [9] J. Ahmad et al., "Concrete With Partial Substitution of Waste Glass and Recycled Concrete Aggregate," *Materials (Basel)*, 2022, doi: 10.3390/ma15020430.
- [10] D. Brahmi, D. Merabet, H. Belkacemi, T. A. Mostefaoui, and N. A. Ouakli, "Preparation of amorphous silica gel from Algerian siliceous by-product of kaolin and its physico chemical properties," *Ceram. Int.*, vol. 40, no. 7 PART B, pp. 10499–10503, 2014, doi: 10.1016/j.ceramint.2014.03.021.
- [11] M. N. YEASMIN, M. Sultana, A. Siddika, S. Tabassum, S. Ullah, and M. S. Bashar, "Structural, Optical, and Morphological Characterization of Silica Nanoparticles Prepared by Sol-Gel Process," *J. Turkish Chem. Soc. Sect. a Chem.*, 2022, doi: 10.18596/jotcsa.1071086.
- [12] M. F. Elkady, H. Shokry, E. E. Hafez, and A. Fouad, "Construction of Zinc Oxide Into Different Morphological Structures to Be Utilized as Antimicrobial Agent Against Multidrug Resistant Bacteria," *Bioinorg. Chem. Appl.*, 2015, doi: 10.1155/2015/536854.
- [13] N. Rangelova, L. Aleksandraov, and S. Yaneva, "Synthesis and structure of amorphous SiO₂/ZnO composites with potential application for azo dye degradation," *Mater. Today Proc.*, vol. 61, pp. 1272–1279, 2022, doi: 10.1016/j.matpr.2022.03.064.
- [14] R. Ciriminna, A. Fidalgo, V. Pandarus, F. Béland, L. M. Ilharco, and M. Pagliaro, "The Sol–Gel Route to Advanced Silica-Based Materials and Recent Applications," *Chem. Rev.*, 2013, doi: 10.1021/cr300399c.
- [15] T. F. Choo, M. A. M. Salleh, K. Y. Kok, and X. A. Матори, "A Review on Synthesis of Mullite Ceramics From Industrial Wastes," *Recycling*, 2019, doi: 10.3390/recycling4030039.
- [16] D. Bersani and P. P. Lottici, "Raman Spectroscopy of Minerals and Mineral Pigments in Archaeometry," *J. Raman Spectrosc.*, 2016, doi: 10.1002/jrs.4914.