DOI: http://doi.org/10.21698/simi.2025.ab27

XRF ANALYSIS OF STYRENE ISOPRENE STYRENE-ISOPRENE/BUTADIENE-STYRENE POLYMERS FILLED WITH CALCIUM CARBONATE AND SEASHELL POWDER: SURFACE DISTRIBUTION AND HOMOGENIZATION CHALLENGES

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Keywords*: innorganic polymer fillers, seashell powder, styrene butadiene compo, XRF analysis*

Introduction

Inorganic fillers, such as calcium carbonate ($CaCO_3$), are commonly added to styrene-butadiene copolymers (SBCs) to enhance mechanical properties like strength, hardness, and wear resistance, while also reducing costs. The effectiveness of these fillers depends on factors such as surface area, shape, and the use of coupling agents to improve filler-polymer interactions. Alternative fillers, including talc and barium sulfate, are also utilized, while carbon black, despite its widespread use, poses environmental concerns. In SIBS (styrene-isoprene/butadiene-styrene) polymers, renowned for their excellent mechanical properties and chemical resistance, fillers such as $CaCO_3$ and natural materials like seashell powder have been incorporated to further enhance functionality by reinforcing the polymer matrix.

Materials and methods

In this study, a composite of SIBS and seashell powder from the *Cardiidae* family was developed. Despite efforts to maximize the powder content, the addition was limited to 15% due to agglomeration issues within the material. The materials were prepared following specific formulations and initially homogenized using a heated blade homogenizer (Nanjing Haisi Extrusion Equipment Co. Ltd., China) to ensure uniform mixing and dispersion of solid and liquid powders. The raw material was then processed through a Nanjing Haisi TSE 65 L/D 44 twin-screw extruder for further homogenization. Finally, the material was pelletized using an underwater cutting system (water ring). For comparison, SIBS samples were also prepared with

the same powder content, substituting cockle shell powder with calcium carbonate (5 VO - Omyacarb®, Omya International AG), a polymer filler with an average particle size of 50 µm. The materials were injected in a molding machine to obtain the discs needed for XRF analysis. This study focuses on the surface distribution of fillers, as the XRF technique probes a depth of ~30–40 micrometers, providing insights into how the fillers are dispersed during the molding process. The findings contribute to understanding filler behavior in polymer composites.

Results and conclusions

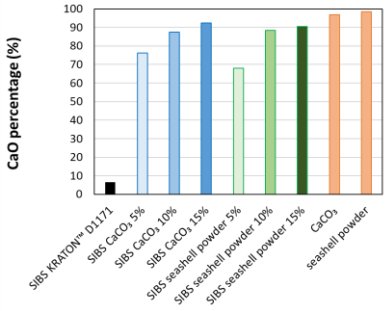

Figure 1. CaO content (%) as indicated by the XRF analysis

The findings of the XRF analysis for Styrene-Isoprene/Butadiene-Styrene (SIBS) compounds filled with calcium carbonate ($CaCO_3$) and seashell powder reveal significant differences in the distribution and migration of calcium oxide (CaO) within the polymer matrix. The pure SIBS polymer exhibits a very low CaO content, approximately 6 ± 0.5%, compared to the polymers filled with $CaCO_3$ and seashell powder. Notably, the filled compounds demonstrate a substantially higher percentage of CaO in the analyzed samples than the initial loading percentage of the fillers.

For instance, a 5% loading of $CaCO_3$ or seashell powder results in CaO content exceeding 65%, while a 15% loading yields CaO content above 85%. This suggests that calcium carbonate migrates to the surface of the material during the extrusion and molding processes, as indicated by the XRF results (considering the 30-40 µm analysis depth).

Additionally, the results highlight differences in the behavior of seashell powder and $CaCO_3$ as fillers. A 5% loading of seashell powder produces 68% CaO in the filled polymer samples, whereas the same loading of $CaCO_3$ results in 76% CaO. This indicates that seashell powder is better accommodated within the polymer matrix than $CaCO_3$, leading to a more uniform distribution. However, at higher filler loadings of 10% or 15%, the XRF analysis shows CaO levels between 88% and 92%, suggesting a saturation limit for the absorption of $CaCO_3$ in the polymer matrix, regardless of the filler quantity added. These findings provide valuable insights into the interaction between the fillers and the polymer matrix, as well as the migration behavior of calcium carbonate during processing.

Acknowledgment

This work was supported by the Ministry of Education and Research – UEFISCDI, project PN-IV-P7.1-PTE-2024-0653 – contract no: 38PTE/2025, and by project CNFIS-FDI-2025-F-0421.