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BPA-FREE EPOXY RESINS FOR STONE CONSERVATION: SYNTHESIS AND NEW ANALYTICAL APPROACH TO OPTIMIZE THEIR CURING

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The development of stone conservation products is an active field of research because the achievement of environment-friendly multifunctional products able to ensure, at the same time, long-term key properties such as consolidating and hydrophobic effects, is a hard task. In this sense, novel products derived from Bisphenol A (BPA)-free epoxy resins have become increasingly appealing due to the easy tailoring of their physical, thermal and chemical properties coming from their combination with compatible inorganic precursors and/or their nano-reinforcement [1].
The aim of this work was double: on one side the synthesis of a BPA-free epoxy resin, based on a diglycidyl ether of a substituted cycloaliphatic diol, with lower health and environmental concerns with respect the widespread phenolic-based epoxies; on the other hand, the development of a new spectroscopic methodology to follow and optimize the synthesis and curing processes.
Accordingly, the synthesis of 2,2,4,4-tetramethyl-1,3-cyclobutanediol diglycidyl ether (CBDO-DGE) was carried out from the reaction of 2,2,4,4-tetramethyl-1,3-cyclobutanediol (CBDO), 2-(chloromethyl)oxirane (EHC) and sodium hydroxide (NaOH) in the presence of a phase transfer catalyst, tetra-n-butylammonium bromide (TBAB) (Figure 1) [2].

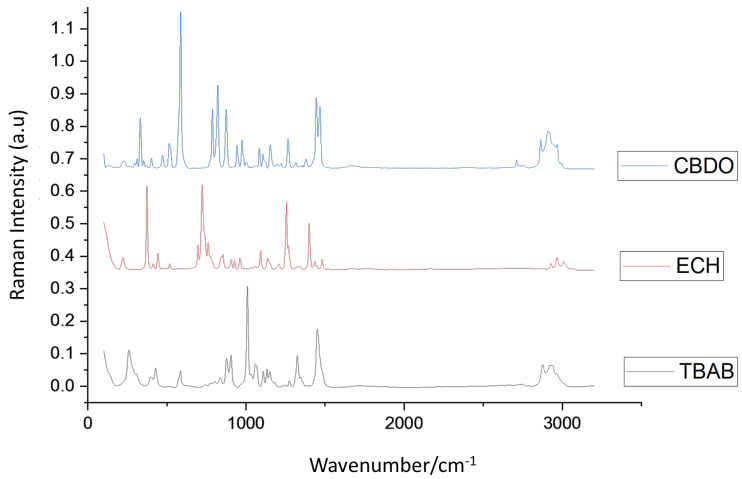

Figure 1. Raman spectra of reactants employed for the epoxy synthesis.

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The crude purification method was followed by FT-IR and Raman spectroscopies, whereas the characterization of the expected compound was carried out by ^{1}H-NMR and ^{13}C-NMR investigations. Then, the ratio epoxy-amine used for its thermal cure was optimized with the help of attenuated total reflection (ATR) measurements and chemometric treatment of the obtained data with The Unscrambler software, using multivariate analysis methods such as Partial Least Squares (PLS) or Multivariate Curve Resolution (MCR) [3]. The thermal behavior of the most promising product, to be exploited as stone conservation material, was studied by means thermogravimetric (TGA) and differential scanning calorimetry (DSC) investigations. Preliminary results coming from the application of such product on selected stones are also discussed.

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References

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