

## ABSTRACT

### **Debranched Cassava Starch Crystallinity Determination by Raman Spectroscopy: Correlation of Features in Raman Spectra with X-Ray Diffraction And <sup>13</sup>C CP/MAS NMR Spectroscopy**

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Because starch crystallinity influences the physical, mechanical, and technological aspects of numerous starch-based products during production and storage, rapid techniques for its assessment are vital. Samples of different levels of crystallinity were obtained by debranching gelatinized cassava starch, followed by subjection to various hydrothermal treatments. The recrystallized products were further subjected to partial hydrolysis with a mixture of  $\alpha$ -amylase and glucoamylase prior to freeze-drying. Crystallinities were determined using X-ray diffraction (XRD) and <sup>13</sup>C CP/MAS NMR spectroscopy, and correlated with FT-Raman spectra features. XRD crystallinities ranged between 0 and 58%, and agreed with crystalline-phase fractions ( $R^2 = 0.99$ ) derived from the respective <sup>13</sup>C CP/MAS NMR spectra. A strong linear correlation was found between crystallinities and integrated areas of the skeletal mode Raman band at 480  $\text{cm}^{-1}$  ( $R^2 = 0.99$ ). With appropriate calibration, FT-Raman spectroscopy is a promising tool for rapid determination of starch crystallinity.

**Key words:** Debranched Cassava starch, crystallinity, X-ray diffraction, <sup>13</sup>C CP/MAS NMR spectroscopy