

QUANTIFICATION OF INDOMETHACIN-SACCHARIN COCRYSTALS BY POWDER X-RAY DIFFRACTION

L. Padrela², E. G. Azevedo², S. P. Velaga^{1*}

^{1*} Department of Health Sciences, Luleå University of Technology, Luleå, S-971 87, Sweden, Tel: +46-920-493924, Fax: +45-920-493850, email: sitaram.velaga@ltu.se

² Department of Chemical and Biological Engineering, Instituto Superior Técnico, Av. Rovisco Pais, 1049-001, Lisboa, Portugal

Aim

To develop a method for the quantification of indomethacin-saccharin cocrystals in the mixture of its components by using powder x-ray diffraction.

Methods

INDSAC cocrystals were physically mixed in various ratios with indomethacin and saccharin (particles slightly ground and passed through a 125µm sieve) by geometric mixing in a controlled environment and 550 mg of these powder mixtures were loaded on the sample holder prior being measured by PXRD (scan between 5° and 40° 2θ, step size of 0.025° and step time 6 s). The most intense characteristic peak of INDSAC at 14.4° 2θ was used for the generation and validation of the calibration curve. A graph was prepared plotting the average area of the selected INDSAC characteristic peak as a function of the weight percentage (% w/w) of INDSAC in each sample mixture. Samples with different concentrations of INDSAC cocrystals in the mixture (15, 30, 50, 70 and 90%) were analyzed in triplicate to determine the linearity of the generated calibration curve.

Results and Conclusions

The calibration curve covering a wide range (0–100%, w/w) of INDSAC showed good linearity over the entire concentration range and follows the equation $y = 1.5562x + 0.9227$ with an excellent correlation coefficient ($R^2 = 0.9996$). The validation results showed excellent correlation between actual and predicted concentrations following the equation $y = 1.0644x - 0.332$ ($R^2 = 0.9981$).

A new powder X-ray quantification method for INDSAC cocrystals was successfully generated in this work.