Nonisothermal estimation of the activation energy by DMTA: an original approach for considering biological heterogeneity

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Among parameters used for process modelling, the activation energy (Ea) is an important thermo-physical criterion. If the differential scanning calorimetry (DSC) is generally considered as a reference to gauge Ea during both reversible and irreversible reactions, it induces a systematic misjudgement due to the local sampling, on the evidence of the biological heterogeneity

The aim of this work consists in applying an original nonisothermal approach to globally estimate Ea. The method implies cooking weatherproofed 17mm cylinders of banana plantain using the dynamic mechanical thermoanalysis (DMTA). Samples between parallel plates are submitted to a uniaxial sinusoidal stress (500 and 450 mN as static and dynamic forces, respectively) at constant 1 Hz frequency. The corresponding strain responses are recorded at different heating rates β (7.5 to 20°C min^{-1}). The loss tangent damping factor is normalised by analogy with DSC computation procedure and stated under the Arrhenius form. Prior to the resolution using the extent of reaction α, data are smoothed using a cubic spline. For comparison, a DSC dynamic approach is applied by performing multiple micro-samplings into two distinct areas corresponding to DMTA pulp specimens. Data are similarly numerically computed. Both apparatus lag temperatures at different β rates are corrected with a pure indium standard.

At α=0.05, the DSC results show a 13% maximum variation in Ea depending on sampling area. No significant differences are observed above α= 0.4. The plantain water gradient may explain such differences in regard to well-known relationship between Ea and water content. In addition, the dependence of Ea on α for both DSC and DMTA suggests the evidence of a complex multi-step process. A maximum 5% difference in Ea estimate was observed between DMTA and DSC in the 0.15-0.7 α range. Higher variations at the end of the complex reaction (ΔEa <30 KJ mol^{-1}) are probably due to some undesirable shrinkage and to the difference between temperatures of DMTA thermocouple and samples.

Then, the original DMTA approach is revealed being suitable to estimate Ea at a given conversion extent, independently of the reaction model.