Electronic supplementary materials *Mendeleev Commun.*, 2021, **31**, 76-78

Thermal decomposition of β-cyclodextrin and its inclusion complex with vitamin E

Meiram Burkeev, Serik Fazylov, Ryszhan Bakirova, Ainara Iskineyeva, Akmaral Sarsenbekova, Erkeblan Tazhbaev and Santai Davrenbekov

The following reagents were used in the experiments: β-CD (99.5%, Fluka), VE (α-tocopherol acetate, Aldrich), a clear oil of light yellow color. The preparation of β-CD inclusion complexes with VE was carried out in a water–alcohol medium under ultrasonic treatment at 60 ^{o}C for 60 min. The β-CD–VE inclusion complexes were obtained in two ratios: 2 : 1 and 4 : 1. The resulting complexes were freeze-dried. They were stored in hermetically sealed bottles, in a desiccator with $CaCl_2$. Thermal properties were studied using a DTA/DTS differential scanning calorimeter (Labsys Evalution). The measurements were carried out in the dynamic mode in the temperature range of 30–500°C, the heating rate of the samples was 10 °C min^{-1}, the atmosphere was nitrogen, the weight of the samples was 12–16 mg, and the Al_2O_3 crucible.

Using TG/DTG data, we estimated the activation energies of chemical transformations during the destruction of β-CD and the β-CD–VE inclusion complexes. Figure S1 shows the calculation results of the activation energy of the thermo-oxidative destruction of β-CD and the inclusion complexes β-CD–VE according to the Freeman–Carroll method (a), Sharp–Wentworth (b), Achar (c) and Coats–Redfern (d) one. The choice of these methods is due to the possibility of comparing the activation energy obtained by differential and integral methods that allow us to evaluate the validity of assumptions made in the derivation of these equations.

The construction of kinetic curves in the coordinates $ln(Rt) = f(1/T)$ made it possible to determine the activation energy (E_a/kJ mol^{-1}) and pre-exponential factor (A/s^{-1}) of the process of thermo-oxidative destruction of β-CD and the inclusion complexes β-CD–VE. The values of the kinetic parameters of thermal degradation for single-stage processes are determined, for example, by the rate of change in the mass of the sample: $-\frac{dw}{dt} = Aexp\left(-\frac{E}{RT}\right)$. Since it is analytically impossible to solve the right side of the equation, various approximate methods are used in practice.

As shown in Figure S1, the graphs for all three samples are very close to a linear function, so they can be approximated by a straight line. The approximation in this case is satisfactory and has a correlation coefficient of 0.9000.

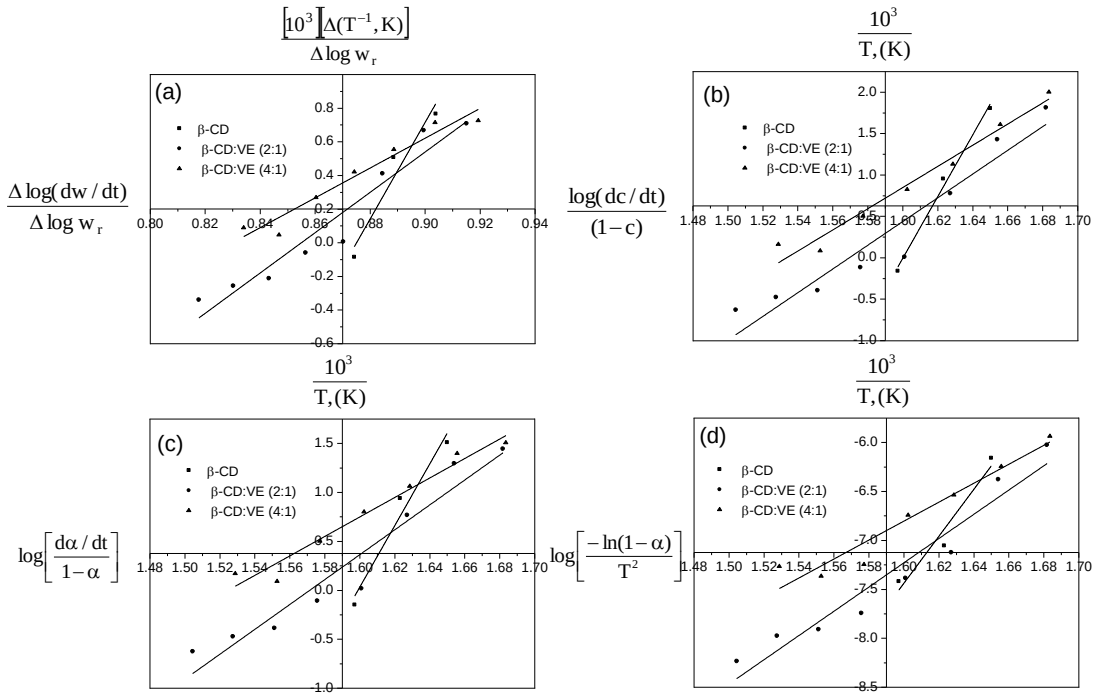

Figure S1 Processing of TGA β-cyclodextrin data and β-CD–VE inclusion complexes by the method of Freeman–Carroll (a), Sharp–Wentworth (b), Achar (c) and Coats–Redfern (d).

In general, mechanisms proposed for solid state reactions can be written as the following: $d\alpha/dt = k\,f(\alpha)$, where α is the conversion at time t, and k is the rate constant. The form of function $f(\alpha)$ depends on the reaction mechanism. The function $g(\alpha)$ represents an integral form of $f(\alpha)$.

Graphic dependences in coordinates from $log\left[\frac{-ln\,g(\alpha)}{T^2}\right]$ is $\frac{10^3}{T}$, expressed by functions $g(\alpha)$ for various kinetic models are shown in Figure S2. The values of the function $g(\alpha)$ selected in this way reflect the most likely reaction mechanism.

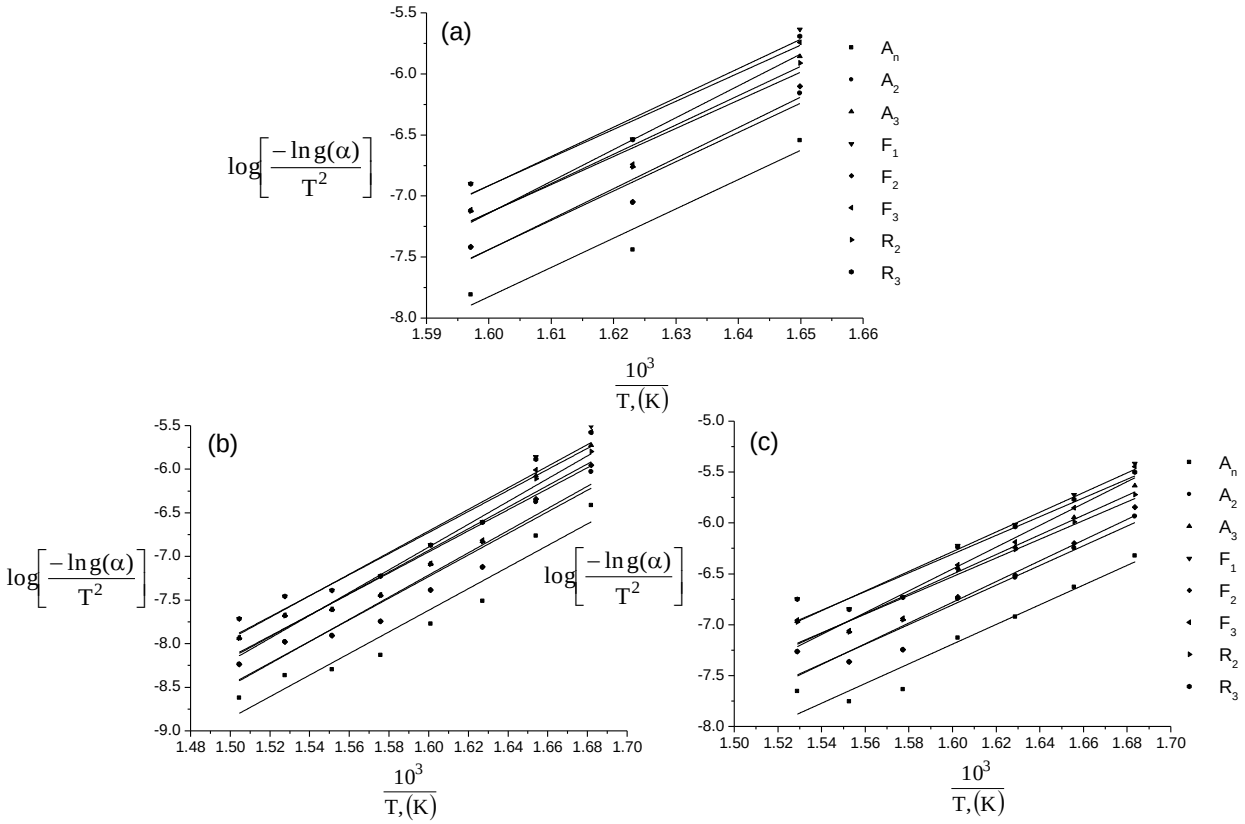

Figure S2 Linearization of thermogravimetry of the objects analyzed: (a) β-CD; (b) β-CD–VE (2 : 1) and (c) β-CD–VE (4 : 1) using the Coats–Redfern method.