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OPTIMIZATION POLYMER BINDER BURNOUT PROCESS

18th March 2025, Dr. Mohammed Bouzbib, Chemist, NETZSCH Analyzing & Testing

Agenda

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1. Introduction to NETZSCH Group
2. Kinetics in Solids
3. Thermal Debinding for Binder Burnout in Powder Metallurgy and Ceramic Processing
4. Kinetics Neo Software
5. Review Example Study: Optimization Polymer Binder Burnout Process with Kinetics Neo Software

The NETZSCH Group

and its three globally operating divisions

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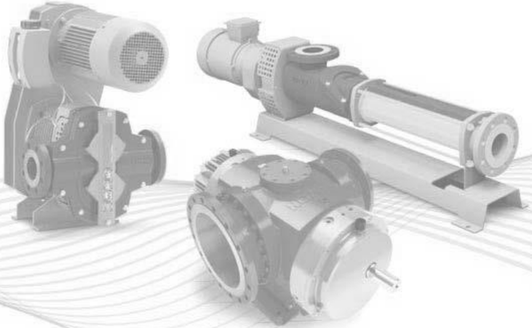
Erich NETZSCH GmbH & Co. Holding KG



Analyzing & Testing

Devices for thermal analysis, determining thermophysical properties and rheology as well as fire testing systems

Grinding & Dispersing

Extensive range of machines for wet and dry grinding, mixing, dispersing, homogenizing and separating

Pumps & Systems

Always the right positive displacement pump for your application

Business areas Analyzing & Testing

Products and services for applications in the
low and high temperature range from -260°C to 2800°C

Thermal Analysis

Determination of dimensional and mass changes, phase transitions and enthalpies as a function of temperature

Thermophysical Properties

Determination of thermal diffusivity and thermal conductivity, specific heat capacity and thermal expansion coefficients

Adiabatic Calorimetry

Analysis of decomposition processes and reaction processes with regard to temperatures, released heat quantities and pressure curves

Rheology

Measurement of rheological properties of non-Newtonian fluids and soft solids - from formulation to product use

Fire Testing Devices

Determination of the fire behavior of products in the automotive, construction, electronics and polymer sectors; classification into "European fire classes"

The Business Unit Analyzing & Testing

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>150M €
Sales

>700
Employees worldwide

>10
Laboratories worldwide

6
International production sites

> 50
Sales and service locations worldwide

NETZSCH Analyzing & Testing - worldwide presence

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Europe

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France
Lyon

Great Britain
Wolverhampton

Italy
Verona

Austria
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Poland
Krakow

Spain
Barcelona

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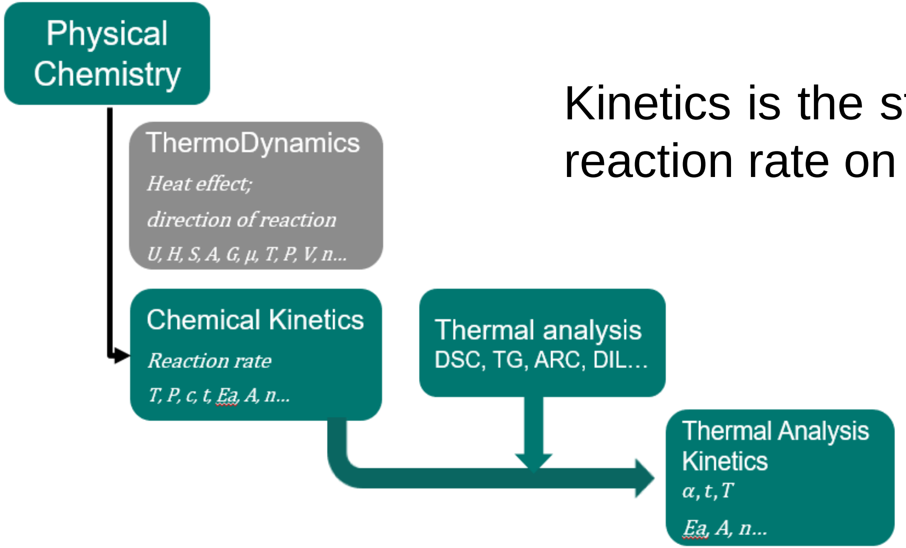
Stockholm

Czech Republic
Prague

Production site   Branch office

1 Kinetics in Solids

Kinetics in Solids



Kinetics is the study of the dependents of a chemical reaction rate on time and temperature.

Dependence of reaction rate on temperature: Arrhenius equation

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General equation

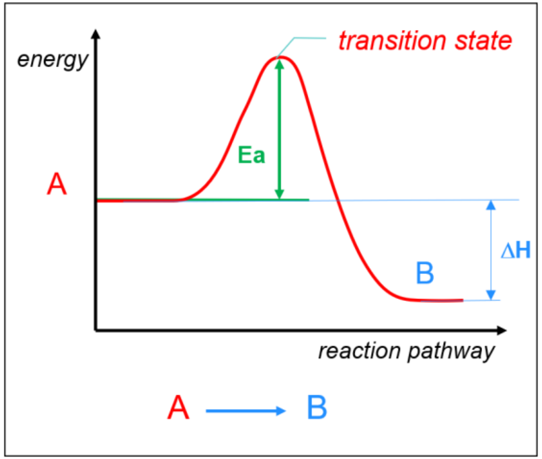
$$\frac{d\alpha}{dt} = k(T) \cdot f(\alpha)$$

Arrhenius dependence

$$k(T) = A \cdot exp\left(\frac{-Ea}{RT}\right)$$

Arrhenius equation

$$\frac{d\alpha}{dt} = A \cdot exp\left(\frac{-Ea}{RT}\right) \cdot f(\alpha)$$



α : conversion
A: pre-exponential factor [1/s]
Ea: activation energy [kJ/mol]
R: gas constant 8.31 [J/(mol.K)]
T: absolute temperature [K] T[K]=T[°C]+273.15

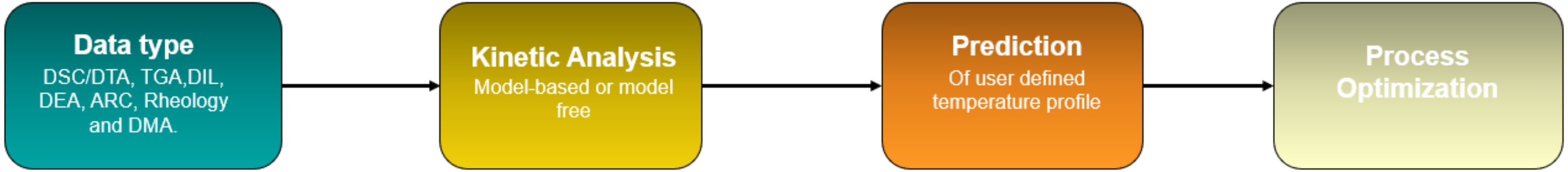
2 Kinetics Neo Software

Kinetics Neo Software

- **Fitting Experimental Data by using Mathematical models:** Mathematical models enable the fitting of experimental data to theoretical equations, which helps in determining kinetic parameters like **Activation energy**, **Pre-exponential factor**, **f(α)** and **Coefficient of determination R^2**.

- **Kinetics Neo software** can simulate and **predict** the behavior of chemical processes under various temperature conditions, such as isothermal, dynamic, and adiabatic profiles.

- **Kinetics Neo Software** allows users to **optimize** temperature programs, ensuring the highest product quality while minimizing processing time.



Supported Measurement Types

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ARC / MMC

Rheology

DMA

DIL

KINETICS NEO

- NETZSCH and non-NETZSCH data
- ASCII text files like TXT or CSV

DSC

DEA

TGA

Kinetics Neo Software

Model design

Curve fit

Data import

Single-point model-free

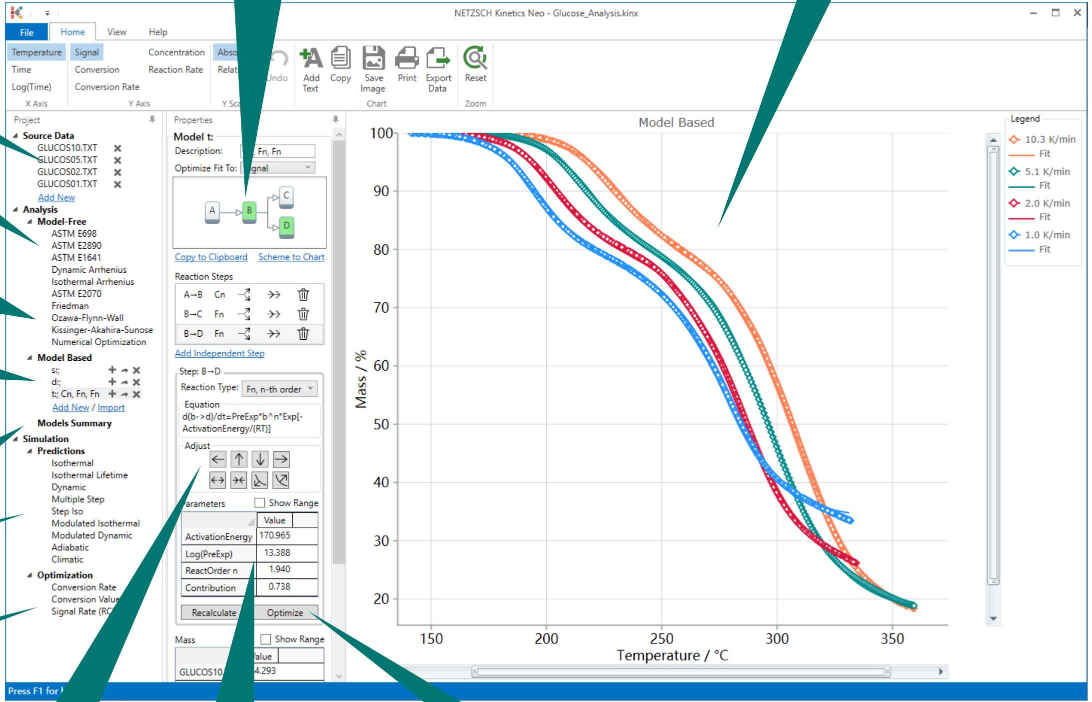
Iso-conversional model-free

Model-based kinetics

Statistical comparison

Prediction

Rate control

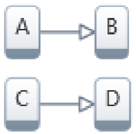


Manual optimization

Kinetics parameters

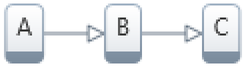
Automatic optimization

Multi-step Model-fitting: Connection Between Steps



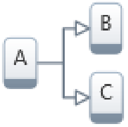
Independent

Mixture of non-interacting components



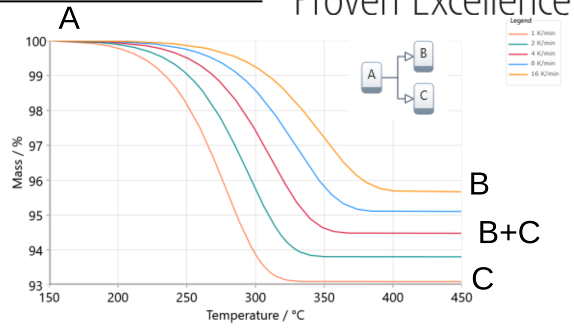
Consecutive

Reaction in the single component



Competing

Product depends on heating rate

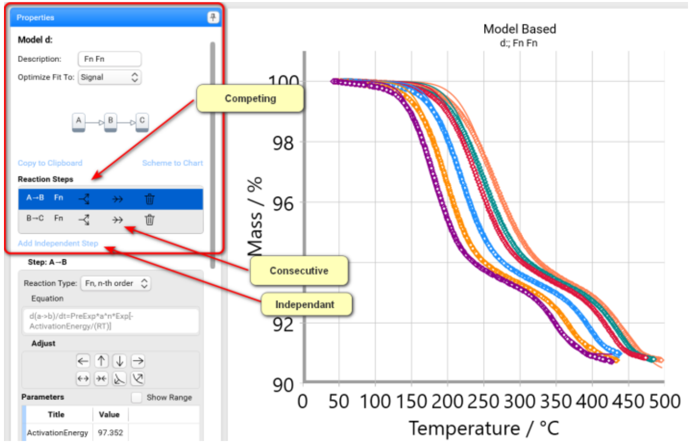


Competing

Independent: reactant and product are not involved in other steps

Consecutive: product of one step is a reactant of another step

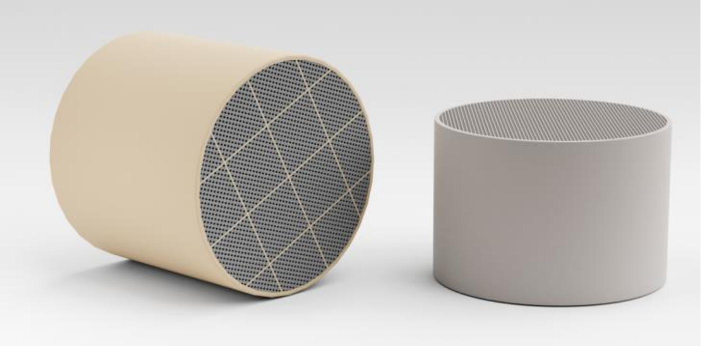
Competing: involve the same reactant



3 Step-by-Step Guide to the Process Optimization

- **Ceramics and Powder Metallurgy**
  1. Used in ceramic processing and Metal Injection Molding (MIM)
  2. Polymer binders help hold fine powders together in a green body

Workflow:
1. Shaping
2. **Debinding**
3. Sintering



Measurements for kinetic analysis: Thermogravimetry

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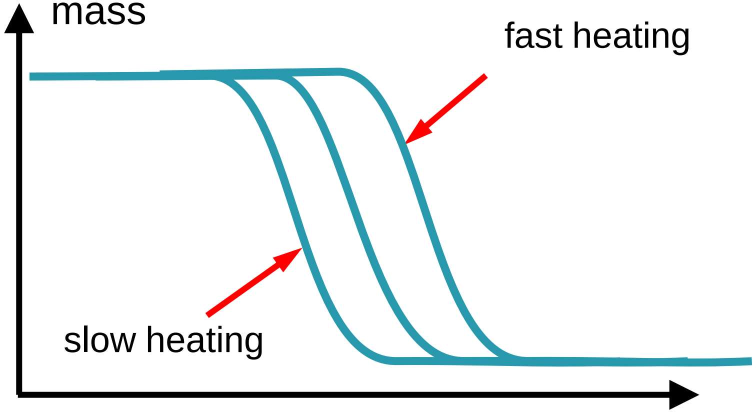
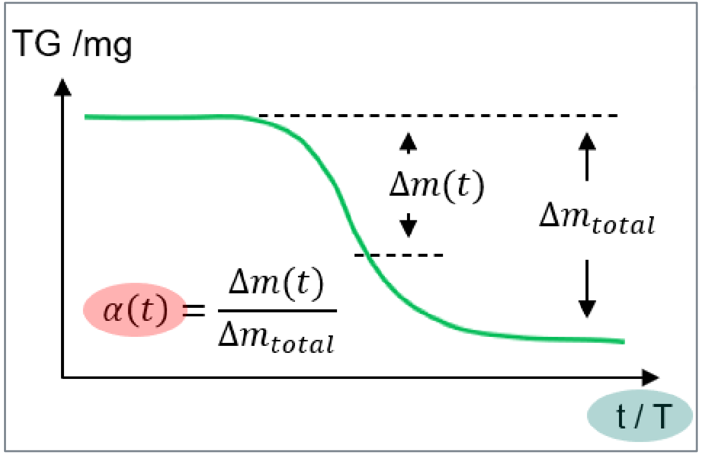
- At least 3 measurements at **different heating rates with R^2 value greater than 0.995**
- Example: 0.25, 0.5, 1, 2, 5, 10, 20 K/min
- **Reproducibility**

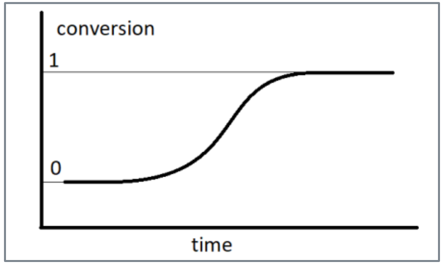
TG 309 Libra Classic



Thermogravimetry: mass change is measured during heating

Conversion α(t) for TGA data





$$\alpha(t) = \frac{\Delta m(t)}{\Delta m_{total}}$$

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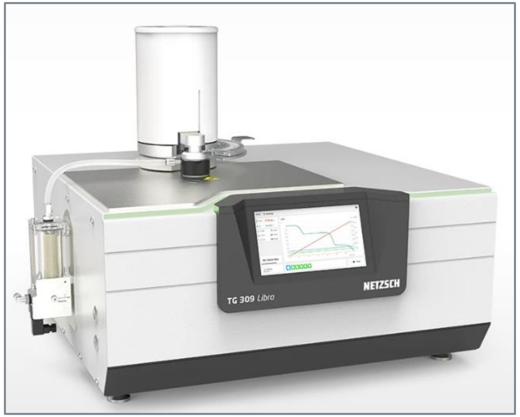

A —(t, T; $\alpha: 0..1$)→ **B**

$$c_A \rightarrow 1 - \alpha$$
$$c_B \rightarrow \alpha$$

- α(t) : Conversion is the ratio of the partial mass loss at given time point to the total mass loss at the final time point

Decomposition Temperature With TGA

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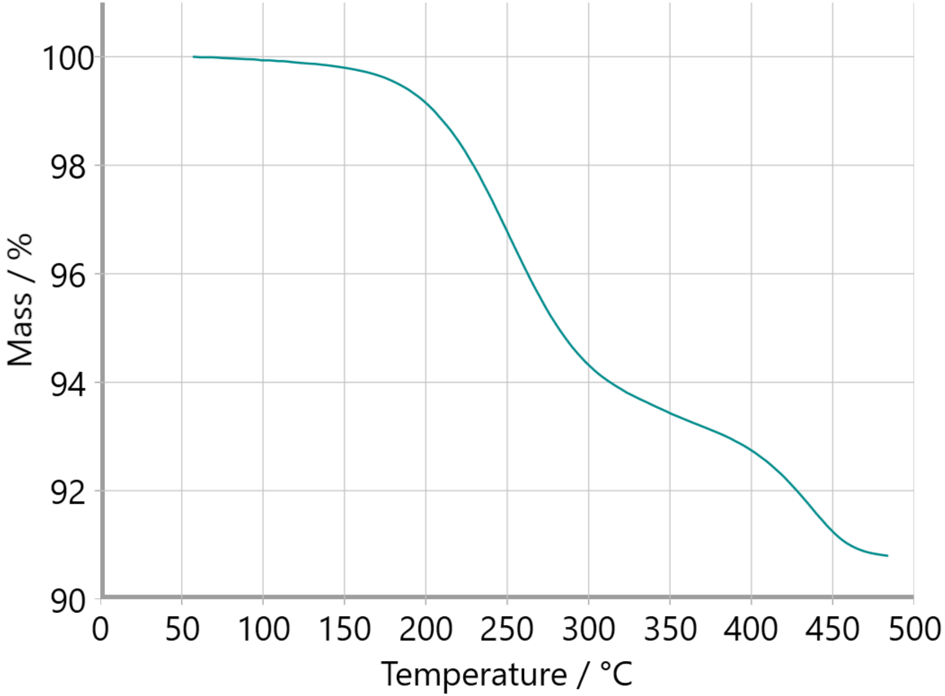

Mass loss versus temperature (TG) curve of **polymer binder** for heating rate 5 K/min in N_2

More Than Decomposition Temperature?

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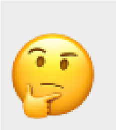
TGA measurement

→ decomposition temperature



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Measurements at Different Heating Rates

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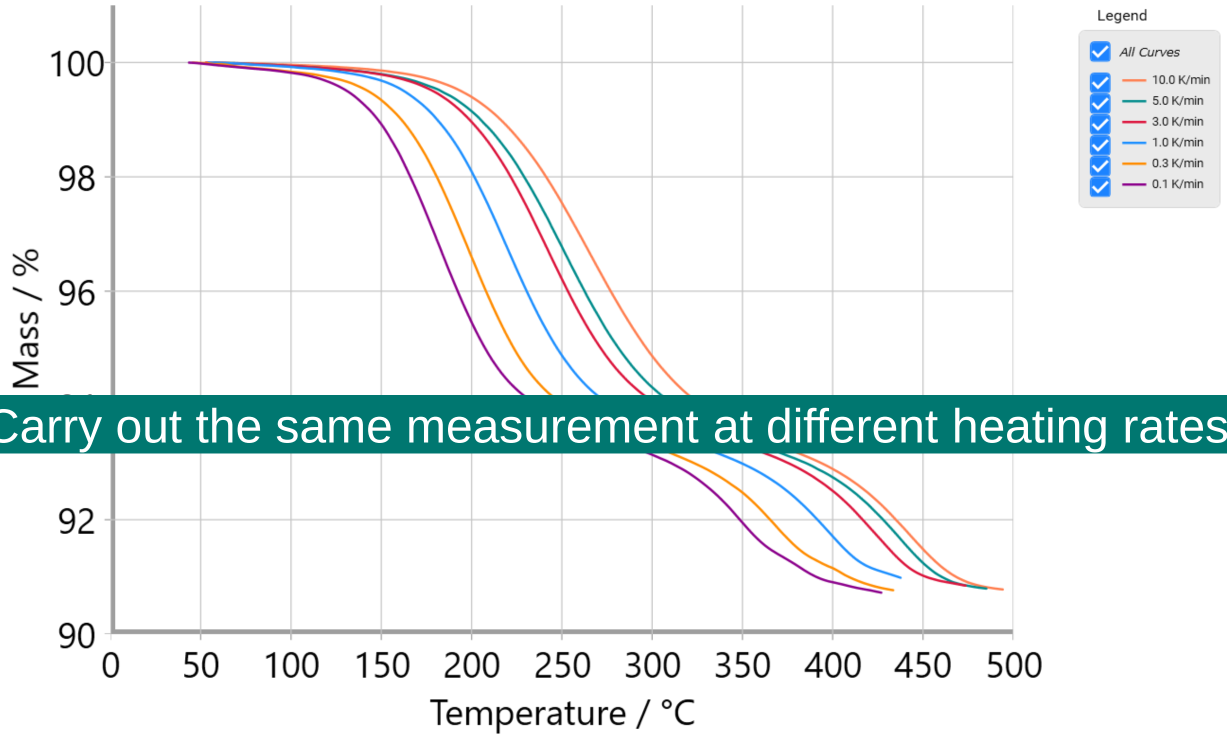
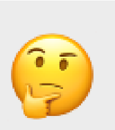


1. Carry out the same measurement at different heating rates

Mass loss versus temperature (TG) curve of **polymer binder at different** heating rates under N_2

How to Determine the number of steps?



Decomposition Steps…

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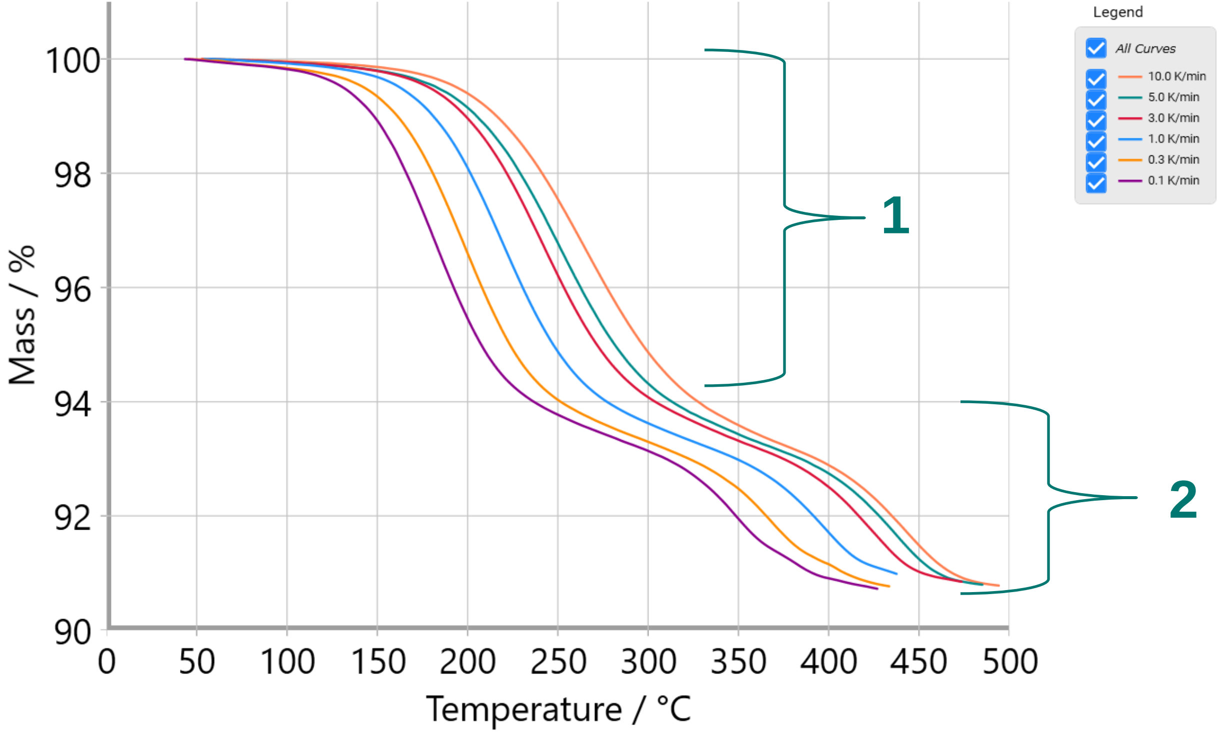
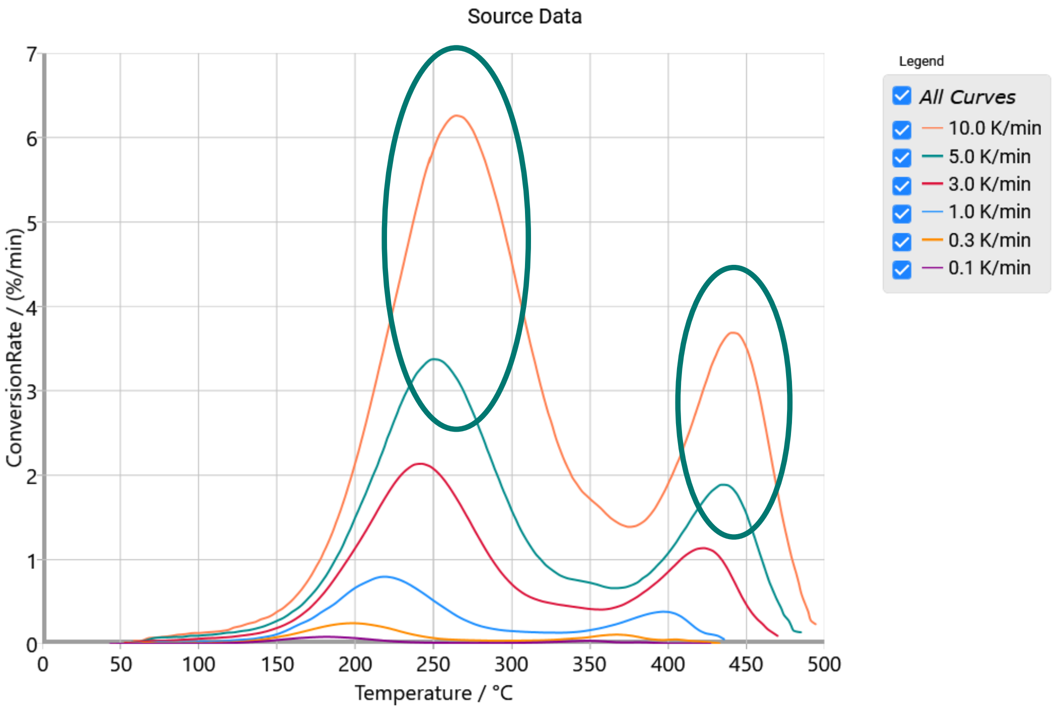


… Or More????



Source Data
ConversionRate / (%/min)
Temperature / °C
0
1
2
3
4
5
6
7
0
50
100
150
200
250
300
350
400
450
500
Legend
All Curves
10.0 K/min
5.0 K/min
3.0 K/min
1.0 K/min
0.3 K/min
0.1 K/min

Kinetics Analysis of the Decomposition of Polymer Binder

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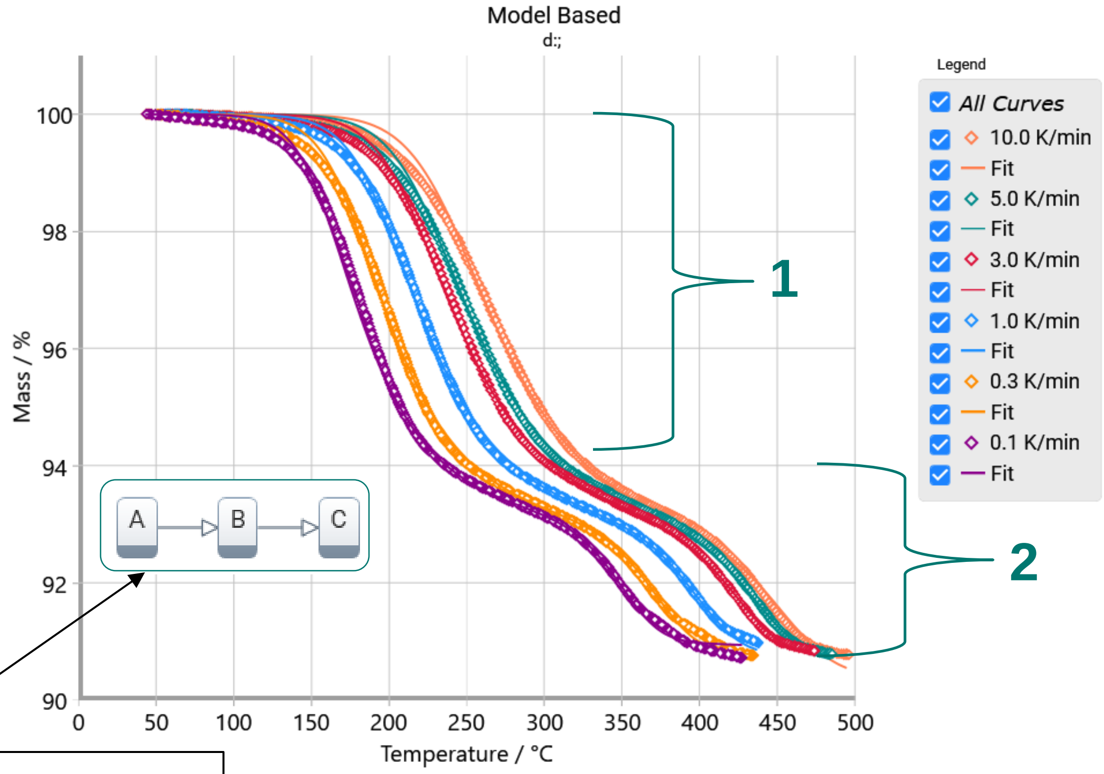
- $R^2 = 0.999$



Kinetic Model for the process
Simulated curves must fit experimental data

Kinetics Analysis of the Decomposition of Polymer Binder

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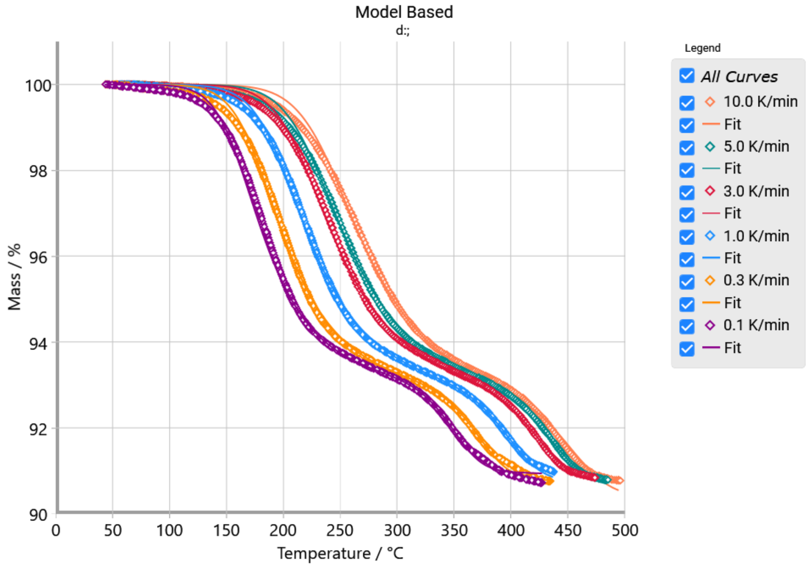
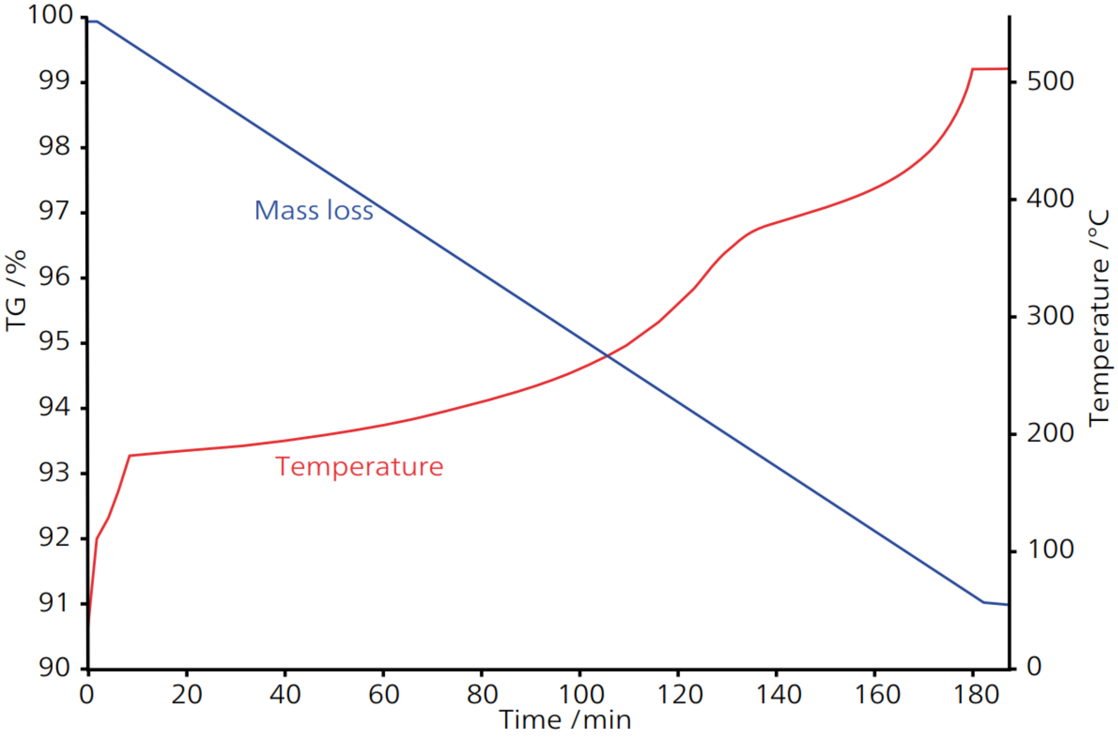


	A → B	B → C
Activation energy (kJ/mol)	97.4	152.3
Log (PreExp) (Log (1/s)	7.4	8.7
Reaction Order n	3.2	1.2
Contribution	0.77	0.23
Coefficient of determination (R^2)	0.999	

The Optimum Temperature Profile for the Polymer Burnout

For the best material quality, a constant mass-loss rate of 1 %/min should be maintained over the entire process.



Temperature program for a Tunnel kiln

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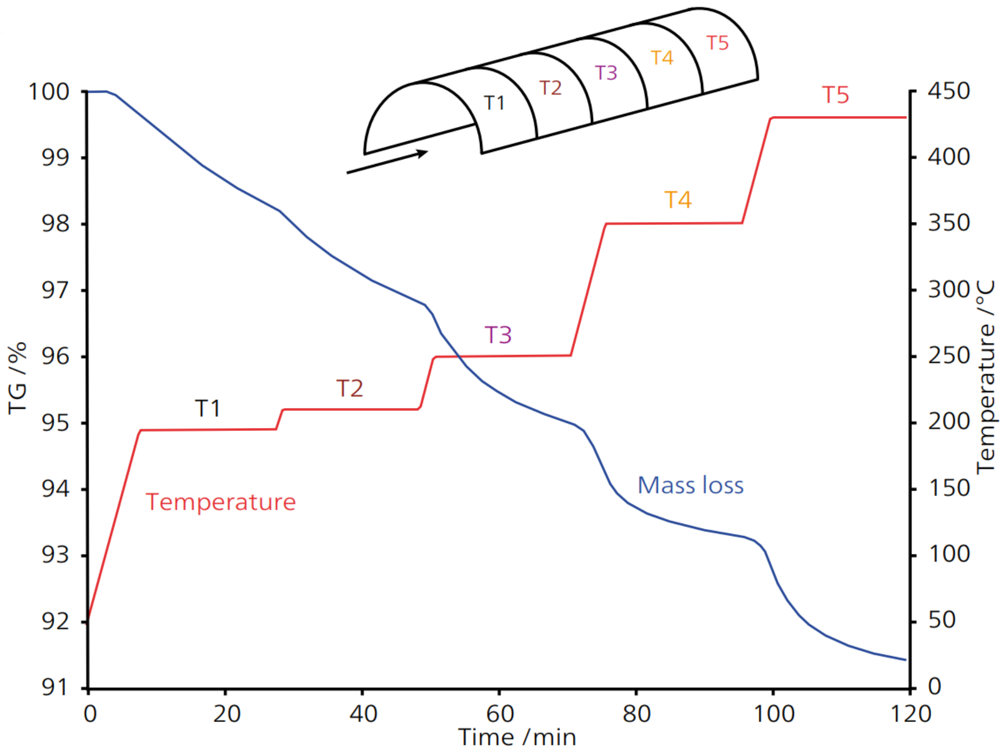
The mass-loss prediction in a 5-zone tunnel kiln for a temperature program corresponding to the optimum zone temperatures for the polymer burnout during the production process.



4 Live Demo

Step-by-Step Recap

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Polymer binder

Measured data TGA

At least **3** measurements at different heating rates.

Kinetics Analysis

- Import data
- Create the most suitable model
  - Reaction type for each step
    - nth order
    - Autocatalysis

Kinetics Triplet, Prediction and Process Optimization

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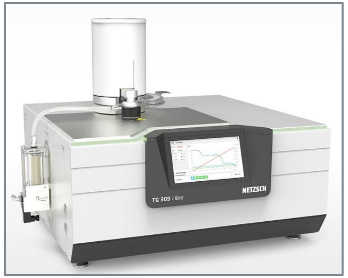

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Key Benefits of Kinetics Analysis

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- ✓ **Improved Product Quality:** Prevents defects like cracking or distorting.
- ✓ **Reduced Processing Time:** Optimizes temperature profiles.
- ✓ **Energy and Resource Efficiency:** Minimizes energy waste.
- ✓ **Reduced Experimental Time :** Eliminates the need for long-term tests by avoiding trial-and-error experimentation.

Kinetics Analysis must fulfil ICTAC kinetics recommendations

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ICTAC: International Confederation for Thermal Analysis and Calorimetry

- Model free analysis
- Multi-step model-fitting (model based)

KINETICS NEO

ELSEVIER

Thermochimica Acta
Volume 689, July 2020, 178597

Review

ICTAC Kinetics Committee recommendations for analysis of multi-step kinetics

Sergey Vyazovkin [a], Alan K. Burnham [b], Loic Favergeon [c], Nobuyoshi Koga [d], Elena Moukhina [e], Luis A. Pérez-Maqueda [f], Nicolas Sbirrazzuoli [g]

[a] Department of Chemistry, University of Alabama at Birmingham, 901 S. 14th Street, Birmingham, AL, 35294, USA
[b] Alan Burnham Consultant, 4221 Findlay Way, Livermore, CA, 94550, USA
[c] Mines Saint-Etienne, University of Lyon, CNRS, UMR 5307 LGF, Centre SPIN, F-42023 Saint-Etienne, France
[d] Department of Science Education, Graduate School of Education, Hiroshima University, 1-1-1 Kagamiyama, Higashi-Hiroshima 739-8524, Japan
[e] NETZSCH-Gerätebau GmbH, Wittelsbacherstrasse 42, Selb 95100, Germany
[f] Instituto de Ciencia de Materiales de Sevilla, C.S.I.C–Universidad de Sevilla, C. Américo Vespucio No. 49, 41092 Sevilla, Spain
[g] University Côte d'Azur, Institute of Chemistry of Nice, UMR CNRS 7272, 06100 Nice, France

Received 18 March 2020, Accepted 19 March 2020, Available online 16 May 2020, Version of Record 5 June 2020.

NETZSCH Kinetics Neo Web Site: How to get a trail version

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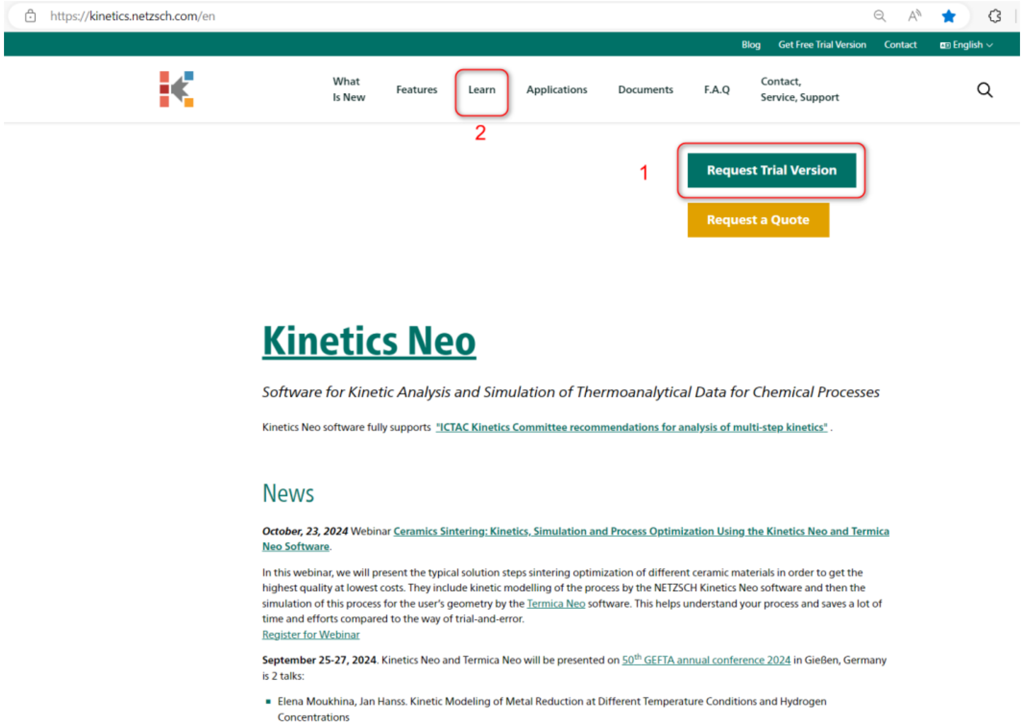
- Go to: https://kinetics.netzsch.com
- Trial Version **30** days

kinetics.neo@netzsch.com



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Thank you for your attention!

Dr. Mohammed Bouzbib
Chemist

For further questions please contact

kinetics.neo@netzsch.com
webinar_ngb@netzsch.com

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