ORLEN PIB
POLYMER INSTITUTE BRNO

Member of ORLEN Unipetrol Group

RESEARCH & DEVELOPMENT

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RESEARCH & DEVELOPMENT

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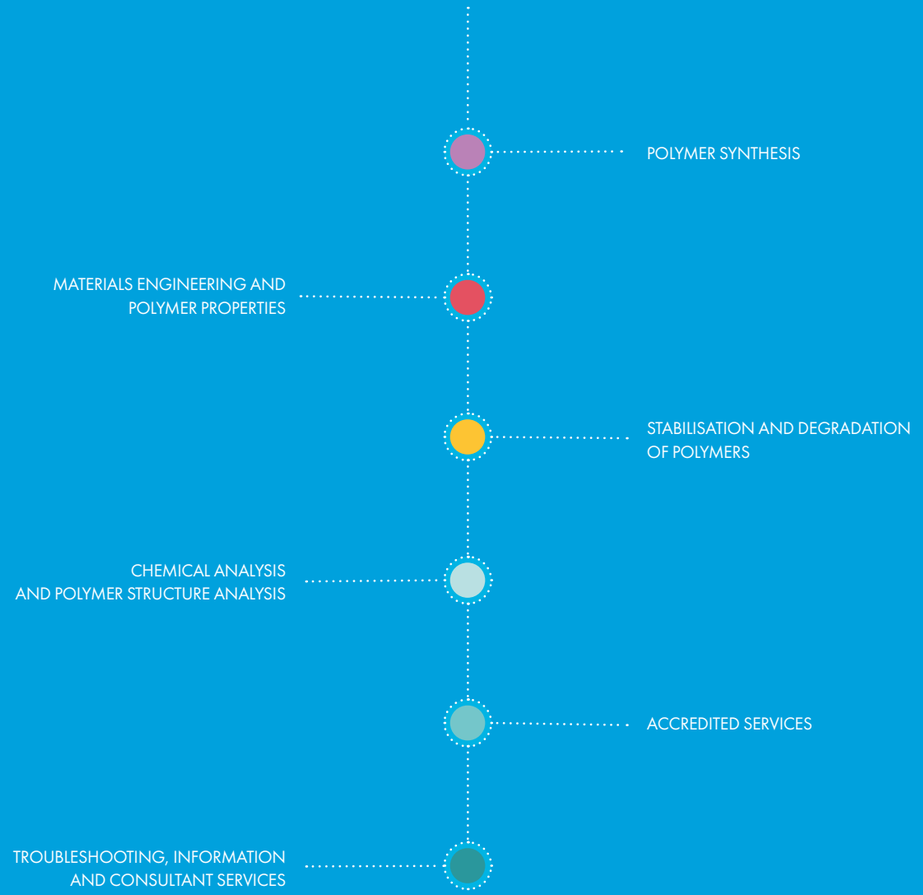
OUR SERVICES



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ABOUT
POLYMER INSTITUTE BRNO

ORLEN Unipetrol RPA s.r.o. – POLYMER INSTITUTE BRNO, o.z. (PIB) is a polymer service provider and technology partner with more than 60 years of tradition in research & development.

HISTORY

POLYMER INSTITUTE BRNO, formerly known as the Research Institute of Macromolecular Chemistry (RIMC), has long been the base of applied research in the field of polymers. The institute has been shaped by more than 60 years of evolution. For many years, the RIMC was a state-sponsored organisation, and its interests were very broad, although always directed at areas more industrial than academic. However, the institute has a long history of close cooperation with a number of universities and academic institutions at home and abroad. With a long tradition in Ziegler-Natta catalyst research and polymer stabilisation, the institute's position became very clear, particularly after building the **Chemopetrol** Litvínov polyolefin production plants in the early seventies. Since then, the institute's activities have mainly focused on polyolefin catalyst research, development of new resin grades, post-reactor modification of polymers (reactive compounding, polymer alloys, polymer reinforcement, blends, etc.), new formulations of additives (stabilisers, colour concentrates, antistatics, etc.) and troubleshooting.

In 1994, **POLYMER INSTITUTE BRNO** became a limited liability company with the status of an independent contract research organisation in which the German company Consil VBmbH and the Czech petrochemical company Chemopetrol, a.s. owned capital shares. In 2004, Unipetrol, a.s. acquired the entire share from both owners, and the institute thus became a subsidiary of the biggest petrochemical complex in the country. After the privatization of Unipetrol, a merger was set up with Unipetrol/Orlen Group. Now, after the merger (December 31, 2015), PIB is a part of **ORLEN Unipetrol RPA** as **ORLEN Unipetrol RPA s.r.o. – POLYMER INSTITUTE BRNO, o.z.**

RESEARCH & DEVELOPMENT
POLYMER SYNTHESES

POLYMER SYNTHESES

The polymer syntheses research at PIB has nearly 50 years of tradition and involves highly qualified personnel and special laboratory equipment constructed through expertise acquired over decades of experience. All polymerisation components are treated in a pure, inert atmosphere. Synthesis is done in proprietary polymerisation reactors equipped with fast and accurate systems for processing, controlling and data collection. The experimental synthesis conditions can be varied, and much effort is placed on customising them to industrial customers' needs. Some reactors are equipped with electrostatic charge sensors to monitor the static charge created in the polymer bed during the polymerisation reaction.

The experimental equipment allows us to study the effect of various additives on the polymerisation kinetics and the catalyst system's performance, including in-situ stabilisation, nucleation, electrostatic charge eliminating agents and catalyst poisons. The synthesized polymers are further tested for details of their properties, such as inner microstructure, polymer particle morphology and mechanical features.

The polyolefin research teams predominantly focus on ethene and propene polymerisation in the presence of heterogeneous Ziegler-Natta, chromium and metallocene catalyst systems.

POLYMERISATION
EQUIPMENT

STAINLESS-STEEL COMPUTER CONTROLLED BATCH REACTORS:

VOLUME	1.8 l	3.2 l	4 l	50 l
Monomer & Co-monomer continuously charged *	**ethene** **propene** butene pentene hexene	**ethene** **propene** butene pentene hexene	**ethene** **propene** **butene** pentene hexene	**ethene** propene butene pentene hexene
H_2 continuously charged	YES	YES	YES	YES
Static charge monitoring	YES	NO	NO	NO
Max. pressure	4.0 MPa	3.0 MPa	4.0 MPa	2.5 MPa
Max. temperature	120 °C	100 °C	120 °C	100 °C
Yield	(0.1–0.5) kg	(0.1–0.7) kg	(0.1– 1.0) kg	(2-8) kg
Polymerisation mode	gas, bulk HC slurry	gas HC slurry	gas, bulk HC slurry	gas
Number of reactors	4	1	6	1

* connection of the reactor to a GC apparatus ensures the replenishment of consumed comonomers and hydrogen by suitable mass flow controllers

POLYETHYLENE
SYNTHESIS

POLYETHYLENE SYNTHESIS

The PE research group has long-term experience with the synthesis of various types of catalyst systems, i.e., ZN and Cr-based. The group traditionally supports the production unit in Litvínov with modified or developmental catalysts which innovate the produced polymer grades.

DEVELOPMENT OF CATALYTIC SYSTEMS FOR PE PREPARATION

Because **ORLEN Unipetrol RPA** is licensed to produce its own catalyst for production units, a significant part of research is dedicated to PE and the development and modification of new catalyst systems. By modifying catalyst systems created at the PIB laboratories, **ORLEN Unipetrol RPA** innovates its product portfolio.

Because commercial units use our supported catalyst systems, much of our research work is on catalysts which anchor to the surfaces of carriers, i.e., mainly silica gels. The support used in these systems is a part of the catalyst which contributes to final polymerisation performance, for example, altering the kinetic profile or incorporating comonomer or transfer reactions. From that point of view, several techniques which characterize carrier properties have been developed over time.

We focus on support for commercial Ti-, Cr-, or Zr-based catalyst systems. Research of model systems based on these transition metals provide highly valuable information which can be applied to optimize of commercial catalysts or develop of new catalyst systems.

POLYETHYLENE
SYNTHESIS

The typical methods used in the process of catalyst development or characterization of silica gel properties are as follows:

- Catalyst synthesis and its handling is performed under strictly inert conditions involving the use of high vacuum lines or glove boxes together with highly purified nitrogen stands for standard operating procedures.
- A lab-scale quartz activator (approx. 15 g) and pilot-scale equipment (up to 5 kg) are used for silica or Cr-based catalyst activation processes.
- Characterization of silica gels involves a complex method which consists of several steps to attain the required quality: (i) chemical analysis, (ii) analysis related to a specific surface area (S.A.), particle size distribution (PSD) – Malvern, pore volume (P.V.), DTA, SEM and TEM microscopy, and (iii) analysis of -OH group content and their distribution using IR, NIR and DRIFT techniques.

All these studies focus on obtaining a better understanding of the relationship between catalyst composition and catalyst polymerisation performance and aim to innovate polymer properties. The development of two proprietary Cr- and Ti-based catalyst systems resulted from systematic research in this field. The Cr-based catalyst (covered by patents – CZ 288056 B6 and US 6.569.966 B1) has been used in industrial production of film and pipe grades.

PIB can perform catalyst synthesis at the customer's request.

Catalyst synthesis represents one part of PIB research and is completed with an evaluation of catalyst polymerisation performance and final polymer properties.

DEVELOPMENT AND OPTIMIZATION OF POLYMERISATION PROCESSES IN THE LABORATORY

The typical polymerisation line consists of a set-up with a reactor connected online to a GC which allow the consumed gaseous components to be continuously replenished. This system permits polymerisation tests under a constant mixture composition.

The reactor design allows simulation of the commercial fluidized bed process which requires a seed bed. Analysis of the polymer formed afterwards is thus easier and precise.

Efficient purification system for all gases (ethylene, comonomer, nitrogen and hydrogen) allows even chromium oxide or metallocene catalysts to be tested without the addition of a scavenger. The polymerisation run starts just after all parameters (temperature, composition of the polymerisation mixture) are set at the required levels in the same manner as in a commercial polymerisation process.

These test conditions allow commercial and developmental catalysts to be compared under real conditions. Kinetic profiles can also be studied, and the formation of agglomerates, deposits on the reactor walls or even the formation of by-products (oligomerisation or hydrogenation) can be observed. These can be easily compared with the structure of the obtained polymer, and a comprehensive view of the tested catalyst system can be produced.

POLYETHYLENE
SYNTHESIS

INNOVATION OF POLYMER PROPERTIES

Analysis of the polymer structure is essential in understanding the relationship between the catalyst performance and physico-mechanical properties of the resulting polymer. The techniques applied for polymer structure assessment are as follows:

- Analysis of structural parameters using GPC, FTIR, DSC
- TREF – temperature rising elution fractionation (solution method)
- SIS/DSC – stepwise isothermal segregation (melt fractionation) techniques

The common mechanical properties of polymers are evaluated using standard methods (such as tensile or flexural tests, ESCR) and many other methods related to certain application areas, for example:

- Plain Stress Impact PSI – an alternative technique used widely to estimate the polymer resistance towards Rapid Crack Propagation (RCP simulates the standard S4 test)
- Full Notch Creep Tests (FNCT test) – a technique for evaluating the material resistance against Slow Crack Growth (SCG); this technique is typically used for pipe and blow moulding grades

OTHER ACTIVITIES

- Development of cost effective chromium-based catalyst systems
- Titanium-based catalyst systems for the production of syndiospecific polystyrene
- Study of the substituent effect on a CGC catalyst complex in the formation of ethylene styrene interpolymers
- Development of laboratory techniques for the simulation of CSTR cascade mode polymerisation processes
- Study of the effect of the silica support activation process and the effect of the concentration and arrangement of hydroxyl groups on silica surfaces
- Evaluation of hydroxyl group content based on volumetric analysis via the interaction of silica support with organoaluminium compounds

POLYPROPYLENE SYNTHESIS

POLYPROPYLENE SYNTHESIS

The process involves homo- and copolymerisation (random and sequential) of propene with other 1-alkenes, specifically in the gas phase or liquid propene or hydrocarbon slurry conditions. The polymer synthesis is continuously monitored during the process, and the data obtained are used to assess the polymerisation kinetics.

The synthesised polymers are studied from the point of view of their structural and mechanical properties:

- Melt flow rate, soluble fractions, extractable fractions, density, particle size distribution, bulk density, porosity, pourability and polymer particle morphology
- Detailed structure characterisation using DSC, TGA, ^{13}C-NMR, pulsed ^{1}H-NMR, GPC, FTIR, SSA/DSC, TREF, VOC and other methods
- Tensile strength, toughness, hardness and heat resistance

POLYPROPYLENE
SYNTHESIS

PROJECT EXAMPLES

- Development of new or modified PP grades (homopolymers, random and sequential copolymers)
- Examination of the effect of specific impurities (“polymerisation poisons”) in raw materials on the kinetics of the polymerisation process and the polymer properties; analytical evaluation of raw materials used for industrial olefin polymerisation
- Fine purification of raw monomers to polymerisation grade purity (down to a level below 10 ppb of the most critical impurities)
- Development of catalyst systems based on commercial catalysts to fit specific industrial processes
- Electrostatic charge monitoring and control for specific polymerisation systems
- Development of in-situ polymer additive processes
- Cooperation with major world producers of catalysts for olefin polymerisation
- Study of polymerisation kinetics
- Assessment of polymerisation kinetics suitable for catalyst system optimization under the desired industrial conditions

SPECIAL POLYMERISATION PROCEDURES

POLYMERISATION KINETICS EVALUATION "SECOND CATALYST INJECTION"

The second catalyst injection method was developed for the purpose of improving the simulation of conditions of industrial gas-phase reactors in laboratory 2 and 4-litre reactors. The second catalyst injection is introduced into the bed of polymer powder subsequent to the initial catalyst injection. The second catalyst dose is applied at full temperature and pressure. Subsequently, an evaluation of the kinetic profile and structure of the produced polymer powder was conducted.

The second catalyst injection mode is comprised of two experiments:

- reference 100-minute run,
- run with second catalyst injection after 40 minutes of initial catalyst dose.

MOLECULAR MODELLING OF ZIEGLER-NATTA CATALYST INTERACTIONS

We can perform molecular modelling such as conformational search and energy evaluation of Ziegler-Natta catalysts interactions with various polymerisation components, including internal and external donors, cocatalysts, catalyst poisons, etc. From the results, the correlations between theoretical and experimental data can be constructed and used to predict of new structures and also visualize trends in catalyst performance and the resulting polymer properties.

SPECIAL SYNTHESES

Special polymer syntheses involve multi-step processes that are tailored to achieve unique structures and properties. In our laboratory polymerisation reactors, it is possible to perform syntheses consisting of up to four distinct steps, with the production of different materials at each stage.

Terpolymers, formed from three distinct monomers, can be produced in one step or as a multi-step process. Synthesis can be performed under conditions similar to those used in industry, or suitable conditions can be suggested according to the expected properties of the final product and the selected catalyst system.

Our state-of-the-art polymerisation reactors can produce a range of semi-crystalline and amorphous thermoplastic polyolefins (TPO) and low-viscosity 1-olefin waxes according to customer specifications. Typically, the synthesis is carried out in a suitable hydrocarbon solvent, after which the produced material is dried and, if desired, stabilised directly in the polymerisation reactor. The product can be collected in the requested packaging via the special reactor bottom valve.

MATERIALS ENGINEERING AND POLYMER PROPERTIES

MATERIALS ENGINEERING AND POLYMER PROPERTIES

The team is active in the research and development of thermoplastic materials and monitors new trends in the field of plastics processing, compounding and polymer applications. Their main areas of interest are reinforced plastics, especially polyolefins, reactive compounding, special polymer blends, thermoplastic elastomers, organosilane crosslinkable polymers, adhesives, electroconductive compounds, flame retardant polymers and many others.

The modified materials and products can be subjected to various physico-mechanical tests, flammability tests, thermal analyses, microscopy or x-ray analysis.

These activities are typically applied in the development of materials designed for automotive, electro-engineering, construction, cabling, pressure pipe, agricultural, and mining purposes, and many others.

MATERIALS ENGINEERING
AND POLYMER PROPERTIES

LABORATORY OF PLASTICS PROCESSING AND RHEOLOGY

Expertise in the structure of polymers and their rheological behaviour. Compounding and conversion are an excellent base for the following activities:

- Grafting reactions (maleic anhydride, organosilane, glycidyl methacrylate, etc.)
- Polymer blending and subsequent dynamic crosslinking
- Compatibilisation of polymer blends
- Extruder polymerisation
- Adhesives
- Controlled rheology PP
- Oxo- and bio-degradable polymers
- Modification of effects
- Flame retardant plastics (halogenated and halogen-free)

MECHANICAL TESTING LABORATORY

Standard test methods:

- Tensile properties
- Flexural properties
- Compression tests
- Tear and peel tests
- Hardness
- Impact tests Charpy, Izod
- Falling dart tests
- Vicat/HDT tests
- Density
- Coefficient of linear thermal expansion

Special testing methods:

- Full-notch creep test (FNCT)
- Crack opening creep test (PENT)
- Time to failure of plastic pipes (hydrostatic)
- Strain-hardening modulus (SHT)
- Cracked round bar test (CRB)
- Creep tests (tension, 3-point bending, 4-point bending, at laboratory temperature and higher temperatures)
- Antistatic properties
- Dynamic mechanical analysis (DMA)
- Morphology and fractography

STABILISATION AND DEGRADATION OF POLYMERS

STABILISATION AND DEGRADATION OF POLYMERS

The laboratory studies the UV, heat and long-term thermal stability of polymers, the kinetics of degradation and behaviour of additives in polymers, and develops stabilising formulations. Processing stability is evaluated using the multiple extrusion technique (polypropylene) or by measuring the torque necessary for extrusion or kneading of the molten polymer (HDPE).

Thermooxidative stability (accelerated tests) is examined in open test tubes or in an oven with forced air circulation at temperatures of 50–150 °C depending on the type of examination and polymer. To a limited extent, the oxygen absorption method may be used or the OIT can be measured.

UV stability is assessed in accelerated ageing tests or by exposure to natural weathering conditions at the PIB weathering station with the option of terrestrial light intensity monitoring.

The chemical and physical behaviour of additives in polymers may be studied by measuring the diffusion kinetics, exudation rate and solubility of additives in the polymer matrix, and by following the chemical changes of the additives in the course of the stabilisation process. The nucleation, antistatic and optical brightening effects, mutual interactions of particular additives in a package, and phenomena associated with water carry over during the production of oriented slit-tapes can also be assessed.

The group has expertise in the development of stabiliser formulations for polyolefins and additive masterbatches and also extensive experience in production plant troubleshooting.

ARTIFICIAL WEATHERING AND DEGRADATION LABORATORY

Simulation of outdoor/indoor weathering and degradation caused by UV spectra

- Xenon-light chambers (Q-SUN XE-3, Q-SUN XE-1)
- Fluorescent chambers (QUV, UVB and UVA fluorescent lamps available)

Simulation of thermo-oxidative stability caused by long-term elevated temperatures

- Aging in hot-air circulation ovens

Testing of polymer melt processing stability via multiple extrusion test

CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

The laboratory provides testing services, troubleshooting, research and quality control for a broad range of clients, markets, and industries. The testing services cover portfolio of accredited tests for automotive and food packaging industry including emission tests, technical cleanliness tests and migration tests.

Instrumentation equipment enables the examination of:

- Polymer molecular structures
- The content of non-polymeric components and their character, for example fillers, stabilizers and other additives, impurities, contaminants, residues (e.g., traces of polymerization catalyst residues, auxiliary materials, solvents)
- Input material purity (monomers, auxiliary materials and additives)
- Identification of polymers or unknown compounds present in the system

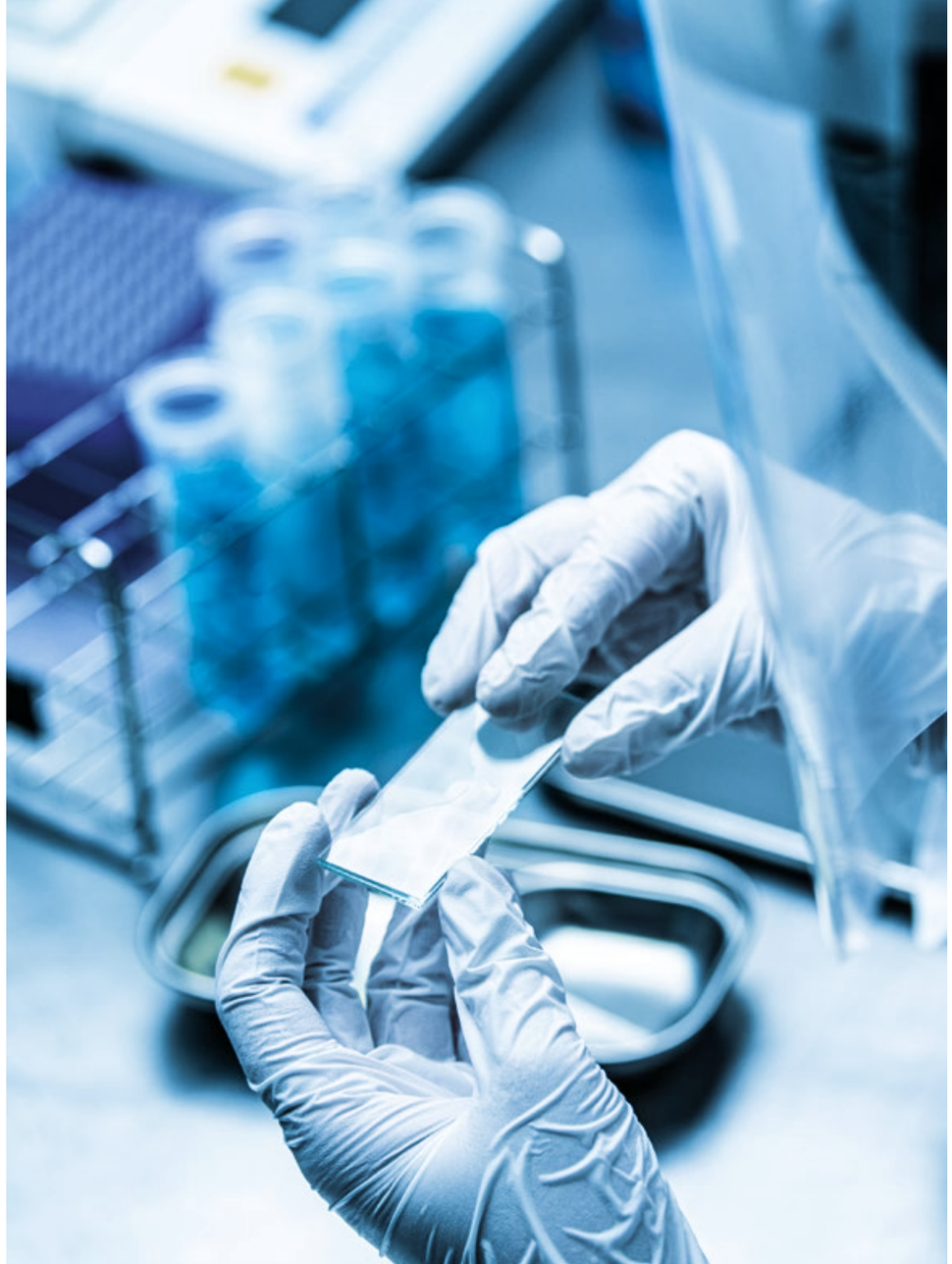
CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

LABORATORY OF SEPARATION METHODS

The laboratory utilizes a wide portfolio of chromatographic and separation methods in order to separate the components of polymeric materials using the most modern procedures and subsequently performing analyses.

Modern solution and gravimetric methods for the separation of polymer components and sample preparation:

- Speed extraction and Automatic Soxhlet extraction:
- Separation of components from masterbatches
- Separation of components from foils and packaging materials
- Recycled and waste materials
- Testing of the migration limits of polymeric materials in a wide range of simulants:
- Determination of total migration into vaporable food simulants (ČSN EN 1186-1,3)
- Determination of total migration in vegetable oils (ČSN EN 1186-1,2)
- Specific migration of additives and excipients into food simulants
- Fogging test - gravimetric determination of volatile substances according to PV 3015 (Volkswagen), DIN 75201
- Viscosity measurements Ubbelohde Viscometer and Höppler falling ball viscometer
- Determination of Intrinsic viscosity, viscosity number, relative/specific viscosity and limiting viscosity number of polymers and copolymers in a given solvent and at a certain temperature according to ISO 1628 and ISO 307
- Determination of all abovementioned viscosities of molding and extrusion materials at high temperatures according to ASTM D4020-18



Modern chromatographic methods for the analysis of individual components of polymeric materials:

- Precise separation, identification and quantification of substances contained in plastics (PP, PE, ABS, polystyrene, polycarbonates, PVC and others)
- Stabilizers, flame retardants, oligomeric UV stabilizers and other additives
- Transformation and degradation products of additives and stabilizers
- Migrating plastic components in food simulants
- Identification and determination of the content of stabilizers in operational sediments
- Determination of trace impurities and catalytic poisons in gases and monomers (CO, CO_2, AsH_3, PH_3, COS, H_2S, mercaptans, alcohols, ketones, ethylene oxide, dienes, acetylenes, hydrocarbons)

- Emission tests using the head space technique or thermal desorption in conjunction with mass detection
- Identification and determination of volatile substances and residual monomers in plastics
- Organic carbon emissions - total content according to PV 3341 (Volkswagen), VDA 277, VDA 278, ISO 16000
- Identification of substances causing smoke or odor in the production process - solving operational problems

- Non-targeted screening analysis using UPLC/Q-TOF determination of trace amounts of impurities, NIAS (Not Intentionally Added Substances and contaminants from the production process) in polymers or migration simulants, fragments of stabilizers called "Arvin substances" in the aqueous leachate of pipe materials

- Further analyses, development of methods and solutions to production problems according to individual agreement

CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

LABORATORY OF SPECTRAL METHODS

Infrared spectroscopy (FTIR) is an effective method for

- Composition identification of both the polymer matrix and additives
- Qualitative and quantitative QC check, comonomers and additives content evaluation
- Determination of raw material purity
- Determination of branching, unsaturation of polymers, degree of degradation, crystallinity and orientation of polymers

Using the **FTIR microscope**, it is possible to identify the composition of small inhomogeneities, layers and gel particles. Thanks to the motorized stage, surface mapping and the creation of chemical maps are possible.

Raman confocal microscopy allows

- Identification of small particles, inhomogeneities and layers, including depth and surface profiling
- Determination of the crystallinity of gel particles
- Orientation measurement
- Identification of carbon nanostructures

UV-VIS-NIR spectroscopy has its application in the determination of some stabilizers, metals, formaldehyde, UV barrier evaluation and other special tests.

Spectrophotometry

- Evaluation of material color coordinates using the CIEL*a*b* color space
- Determination of whiteness, brightness, yellowness, blackness, jetness, undertone of pigmented parts and articles
- Determination of haze/turbidity in transparent plastics and thin films (ASTM D 1003)
- Determination of the specular surface layer gloss level (20°, 60°, 85°) via glossmeter

NMR spectroscopy is a suitable method for

- Describing the polymer chain structure, branching and irregularity
- Identification and quantification of monomers
- Molar distribution of sequences and sequence length
- Chain end quantification
- Evaluation of polymer isomerism

X-Ray Fluorescence spectroscopy (XRF) for qualitative and quantitative elemental analysis (Z>11)

- Identification of inorganic fillers and its purity
- Screening for Pb, Cd, Hg, Br, Cr (according to RoHS)
- Screening for halogens in flame retarded polymers

LABORATORY OF INORGANIC ANALYSIS

- Determination of water content using the Karl Fischer procedure (including coulometric determination) in liquid and solid samples
- Determination of carbon-black and ash content in plastics
- Determination of active oxygen (peroxide level)
- Determination of F, Cl and Br in plastics
- Determination of conductivity and pH

ICP-OES

- Determination of Residual Catalysts
- Analysis of Additives and Stabilizers (e.g. Ca, Zn, Ba, Sr, Sb, P...)
- Contamination Control – unwanted trace elements in material
- Leaching/Migration Studies - important for food contact material, toys, medical devices.
- Characterization of Fillers and Pigments

LA-ICP-MS

- Trace Metal Analysis – detection of ultra-trace amounts of metals (e.g. Pb, Cd, Hg, As, Sb, Cr, Ni...) – critical for compliance with regulations like RoHS, REACH, FDA or ISO 10993.
- Fast semi-quantitative analysis – determination of metal content directly in polymer films or pellets "as is"
- 2D Elemental Mapping and Depth profiling
- Micro-contamination studies (e.g. metal particles or inclusions)

CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

LABORATORY OF THERMAL METHODS

Differential scanning calorimetry (DSC) is used especially for:

- Determination of temperature and enthalpy of melting and crystallization
- Determination of glass transition temperature
- Determination of the degree of crystallinity of semicrystalline polymers
- Identification of polymers, copolymers, polymer blends and polymer impurities
- Isothermal and non-isothermal crystallization and determination of crystallization kinetics of polymers
- Determination of specific heat capacity
- Determination of thermooxidative stability of polymers (OIT method)
- Measurement at low temperatures (from -85 °C)

Thermogravimetric Analysis (TGA) of Plastic

- Provides valuable insights into thermal stability, decomposition and composition of plastics by measuring changes in mass as a function of temperature
- Supports material identification, quality control and material troubleshooting
- In our laboratory, TGA is routinely used for: determination of carbon black and calcium carbonate content, quantification of inorganic fillers, analysis of UV stabilizers and others additives

FLAMMABILITY TESTING

Determination of oxygen index LOI (ISO 4589)

UL-94 procedure:

- Horizontal test (HB)
- Vertical test 50 W (V-0, V-1, V-2)
- Vertical test 500 W (5VA, 5VB)
- Cellular, foam and lightweight materials (HBF, HF-1, HF-2)
- Thin films (VTM-0, VTM-1, VTM-2)

Glow wire testing:

- Determination of glow wire ignition temperature GWIT (EN 60695-2-13)
- Determination of glow wire flammability index GWFI (EN 60695-2-12)
- Determination of glow wire for end-products GWEPT (EN 60695-2-11)
- Needle-flame test method (EN 60695-11-5)
- Fire hazard testing - Abnormal heat - Ball pressure test method (EN 60695-10-2)
- Reaction to fire tests — Ignitability of products subjected to direct impingement of flame (ISO 11925-2)
- Classification of building materials (EU class E acc. to ISO 13501-1)
- Determination of building and construction parts flammability and burning behaviour (B2 according to DIN 4102-1)
- Determination of horizontal burning rate and flame spread of interior automotive materials (ISO 3795, DIN 75200)
- Hot-wire based test methods - Hot-wire coil ignitability test on materials (IEC 60695-2-20, UL 746A)
- Smoke generation, Part 2: Determination of optical density by a single-chamber test (ISO 5659)

CHEMICAL ANALYSIS AND POLYMER STRUCTURE ANALYSES

LABORATORY OF MORPHOLOGY AND PHYSICAL PROPERTIES

Optical Microscopy

- Initial inspection of materials
- Identification of fractures, deformations and surface defects
- Troubleshooting of material failures and production issues
- Detection of defects by microscopic evaluation of microtome sections
- Determination of technical cleanliness

SEM/EDX analysis

- High-resolution imaging of surface structure, deformations and fracture zones
- Detailed examination of micro-cracks, delamination and material interfaces
- Elemental composition analysis using EDX (Energy Dispersive X-ray Spectroscopy)
- Identification of contaminants and unknown particles
- Troubleshooting of material failures and production issues

Evaluation of powdered materials

- Measuring the particle size and particle size distribution of materials by laser diffraction
- Wide particle size measuring range from 10 nm to 3.5 mm and by DLS measurement 0.1 nm –10 µm
- Measuring by Wet, Dry dispersion and small sample unit suitable for catalysts
- Measuring zeta potential (surface charge of a particle)

LABORATORY OF ADVANCED FRACTIONATION TECHNIQUES

Polymer microstructure is analyzed by fractionation based on molecular size and crystallizability.

Size-based fractionation method – HT-GPC (High-Temperature Gel Permeation Chromatography)

- Characterizes polymers insoluble at conventional temperatures.
- With triple detection, provides molar mass distribution, intrinsic viscosity, and long-chain branching.
- Coupled with FTIR, enables analysis of comonomer content and short-chain branching.

Crystallization- and solubility-based fractionation methods

- Temperature rising elution fractionation **(TREF) in analytical or preparative mode**
- Crystallization elution fractionation **(CEF)**
- Successive multistep isothermal crystallization **(SIC)**
- Successive self-nucleation annealing **(SSA, SNA)**
- Combination of methods, e.g. **TREF-SIC, TREF-SNA**

ACCREDITED
SERVICES

The Czech Accreditation Institute has granted PIB a Certificate of Accreditation as an accredited testing site for the mechanical, analytical and special properties of materials. A list of valid Accreditation Certificates and Accredited Tests is available directly on the website of the Czech Accreditation Institute **(https://www.cai.cz/?subjekt=1380-unipetrol-rpa-s-r-o-polymer-institute-brno-odstepny-zavod&lang=en)**.

Chemical analysis tests:

- Determination of melting and crystallization temperature and enthalpy, glass transition temperature and including kinetics of crystallization by differential scanning calorimetry (DSC) acc. to ČSN EN ISO 11357-1; -2; -3; -7
- Determination of ash content (content of glass and mineral filler) by gravimetry acc. to ČSN EN ISO 3451-1; -4; ČSN EN ISO 1172
- Determination of water content (moisture) - Karl Fischer method acc. to ČSN EN ISO 15512, method B2; ČSN ISO 760
- Determination of the sum of emissions of organic compounds by gas chromatography (GC/FID/MS) acc. to PV 3341; VDA 277:1995
- Determination of formaldehyde emissions by spectrophotometry acc. to PV 3925; VDA 275
- Determination of viscosity number by capillary viscometer acc. to ČSN EN ISO 1628-1; 5; ISO 1628-4; ČSN EN ISO 307
- Determination of total migration of substances from plastics in evaporable food simulants - total immersion method acc. to ČSN EN 1186-1; -3
- Determination of total migration of substances from plastics in vegetable oils - method of total immersion in olive oil acc. to ČSN EN 1186-1; -2
- Determination of oxidation induction time (OIT) by differential scanning calorimetry acc. to ČSN EN ISO 11357-1; -6
- Determination of the content of glass fibres, soot and inorganic fillers by thermogravimetry acc. to ČSN EN ISO 11358-1
- Detection of defects by microscopic evaluation of microtome sections acc. to the internal method
- Fogging test acc. to PV 3015; DIN 75201
- Identification by FTIR method acc. to the internal method
- Determination of technical cleanliness by gravimetry acc. to ISO 16232, VDA 19.1
- Determination of technical cleanliness by microscopy acc. to ISO 16232, VDA 19.1
- Determination of elements (Ba, Co, Cu, Fe, Li, Mn, Zn) by ICP-OES acc. to the internal method (ČSN EN ISO 11885)

Mechanical and Physical tests:

- Determination of tensile properties acc. to ČSN EN ISO 527-1; -2; -3; ČSN EN ISO 6259-1
- Determination of flexural properties acc. to ČSN EN ISO 178
- Determination of impact strength using the Charpy method acc. to ČSN EN ISO 179-1; -2
- Determination of impact strength using the Izod method acc. to ČSN EN ISO 180
- Determination of properties at multiaxial impact loading acc. to ČSN EN ISO 6603-1; -2
- Determination of indentation hardness by means of a durometer (Shore hardness) acc. to ČSN EN ISO 868
- Determination of hardness – ball indentation method acc. to ČSN EN ISO 2039-1
- Determination of temperature of deflection under load acc. to ČSN EN ISO 75-1; -2
- Determination of Vicat softening temperature (VST) acc. to ČSN EN ISO 306
- Determination of density using the immersion method acc. to ČSN EN ISO 1183-1, cl. 5.1, method A
- Determination of resistance to slow propagation of crack (PENT) acc. to ISO 16241
- Determination of environmental stress cracking using the full-notch creep test (FNCT) acc. to ISO 16770; ASTM D5397
- Determination of hardness – Rockwell hardness acc. to ČSN EN ISO 2039-2
- Determination of odour of components acc. to PV 3900; VDA 270
- Determination of component damage at elevated or reduced temperatures without load acc. to the internal method (DIN 53497, cl. 4.2, Method B)
- Determination of strain hardening modulus (SHT) acc. to ČSN ISO 18488
- Determination of resistance to slow crack growth under cyclic loading acc. to ČSN ISO 18489
- Determination of burning behaviour acc. to ČSN ISO 3795; DIN 75200
- Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) acc. to ČSN EN ISO 1133-1; -2

RESEARCH
& DEVELOPMENT

TROUBLESHOOTING AND CONSULTANT SERVICES

Even during reliable processes of polymer manufacturing and conversions, problems can occur and expert troubleshooting is needed for rapid and efficient solutions.

We offer a unique multidisciplinary approach to problem solving, starting from chemical analysis of raw material purity and polymerisation catalyst activity, examination of the chemical engineering principles in production units, and finally evaluation of the processing conditions and additive package performance. We can suggest and in most cases supply suitable polymer raw materials and additive and colour concentrates according to the customer's specific needs. The experience of our staff and the analytical and testing facilities at the institute have an important role in our services.

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