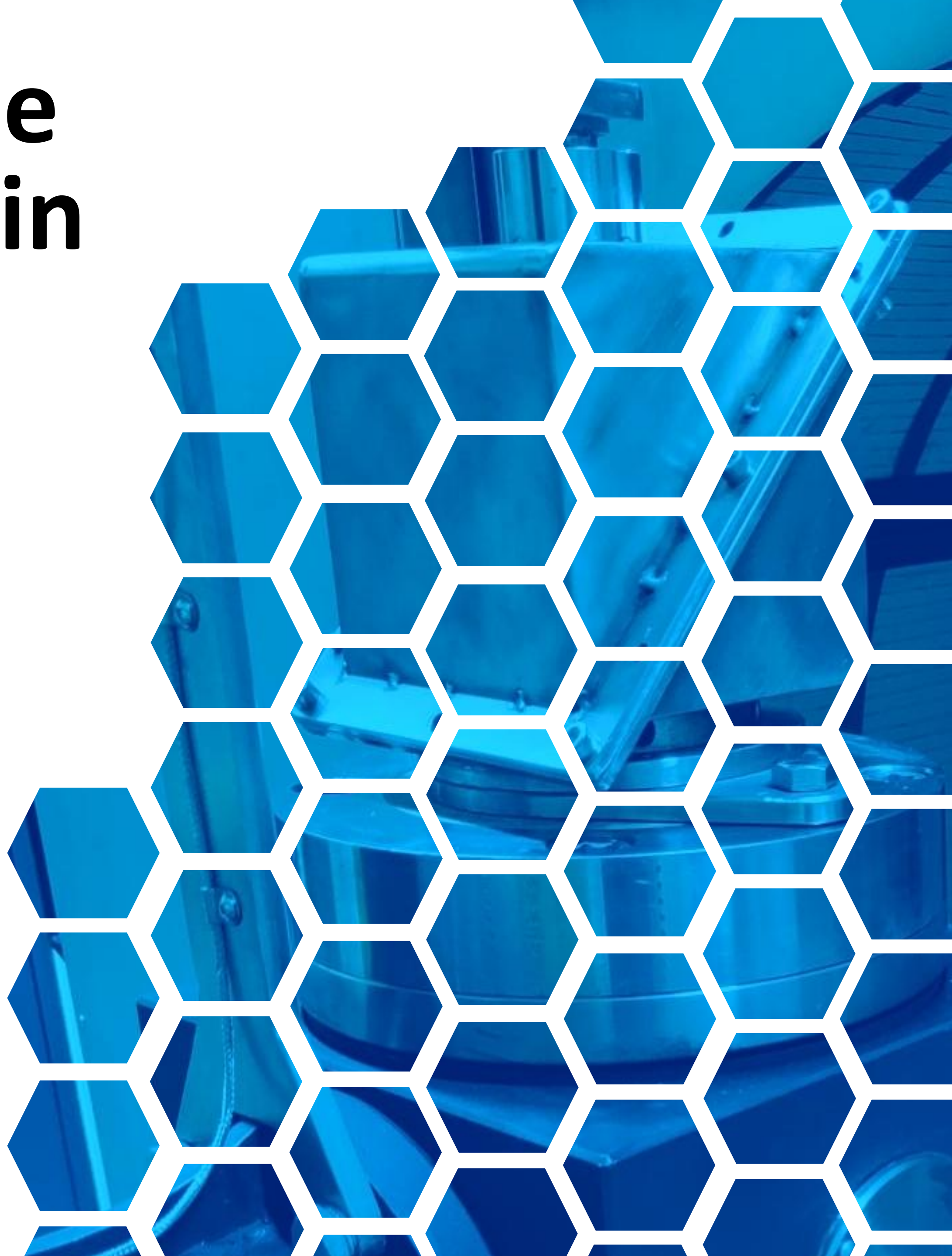
New perspectives in the use of biomass as active fillers in rotational molding technology

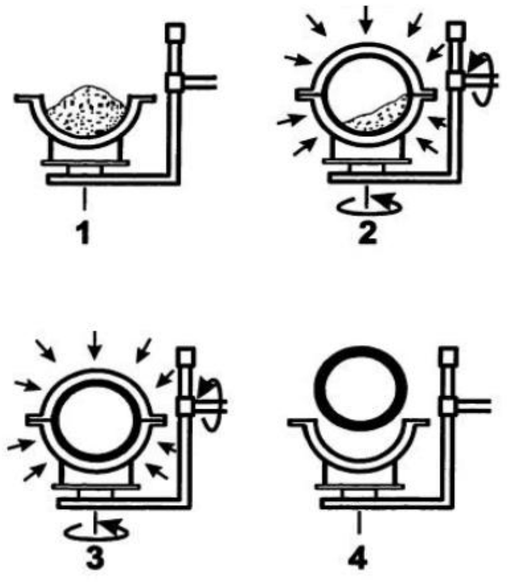
Mateusz Barczewski

mateusz.barczewski@put.poznan.pl

Poznan University of Technology
Faculty of Mechanical Engineering
Institute of Mechanical Technology
Division of Polymer Processing
Piotrowo 3, 61-138 Poznan, Poland



Principles and basics of the process



1. Introduction of polymer powder in the mold
2. Mold heating + rotation
3. Mold cooling + rotation
4. Product demolding + mold cleaning

Transport mechanisms of polymer powders during rotational molding processing *

* K. O. Ogila, M. Shao, W. Yang, J. Tan, eXPRESS Polymer Letters, 2017, 11, 10, 778-798.

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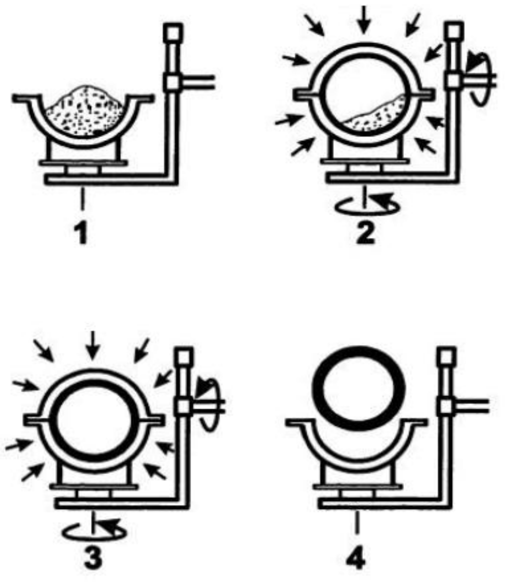

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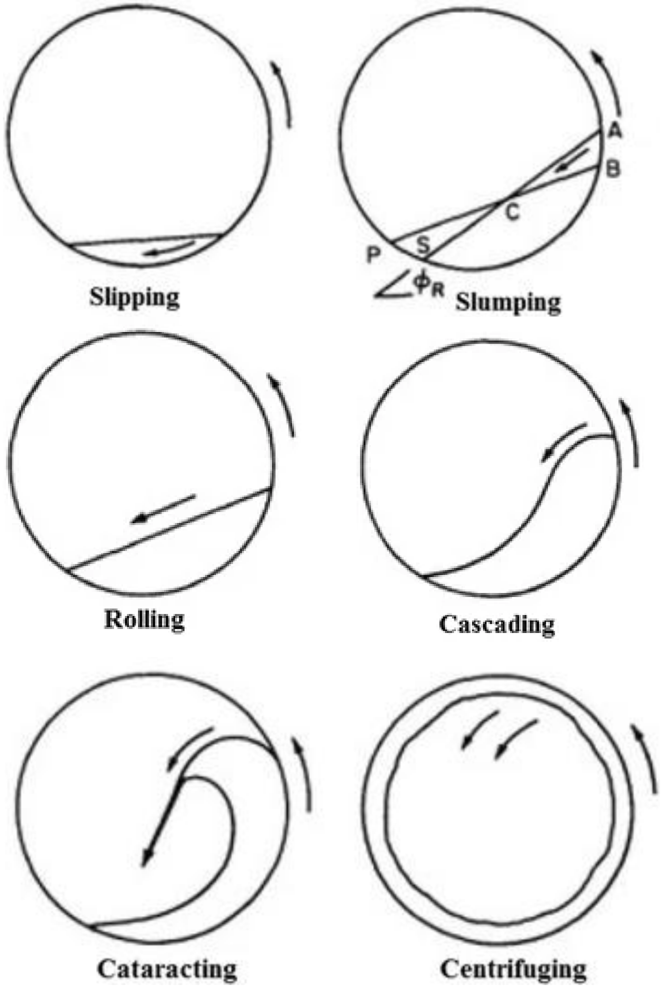

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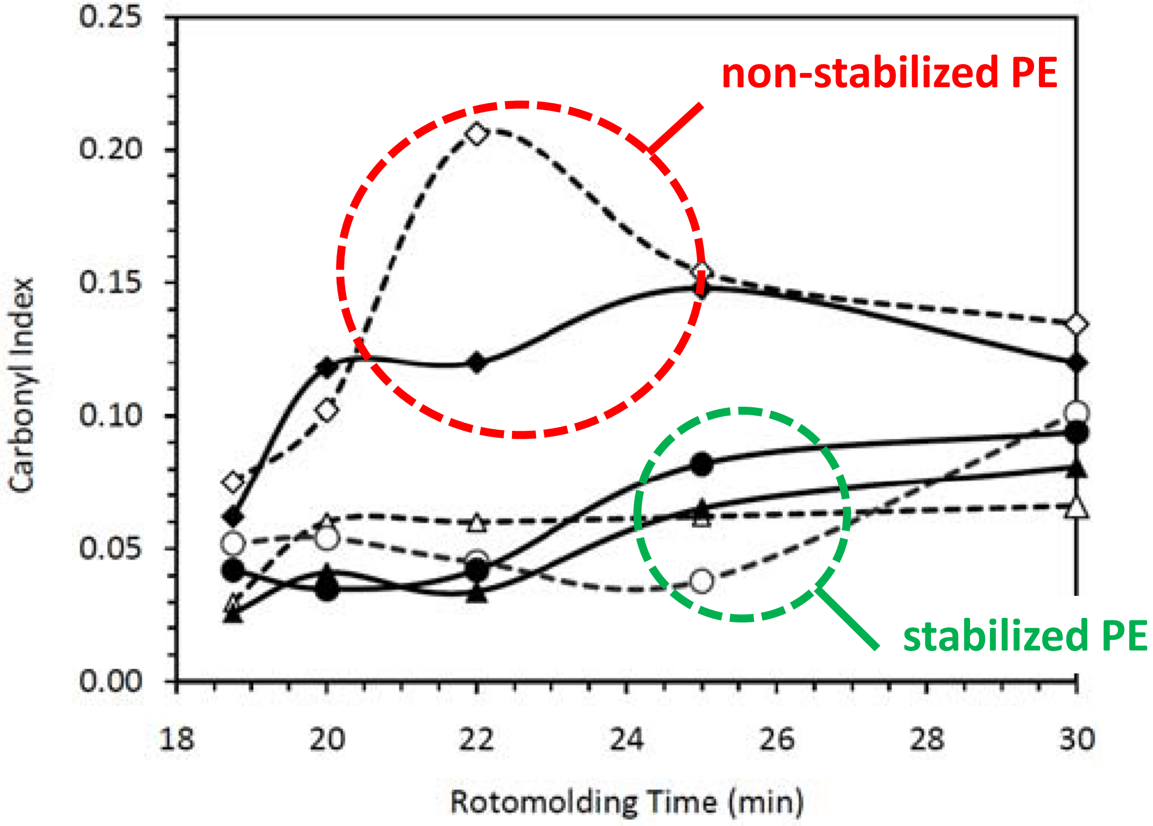
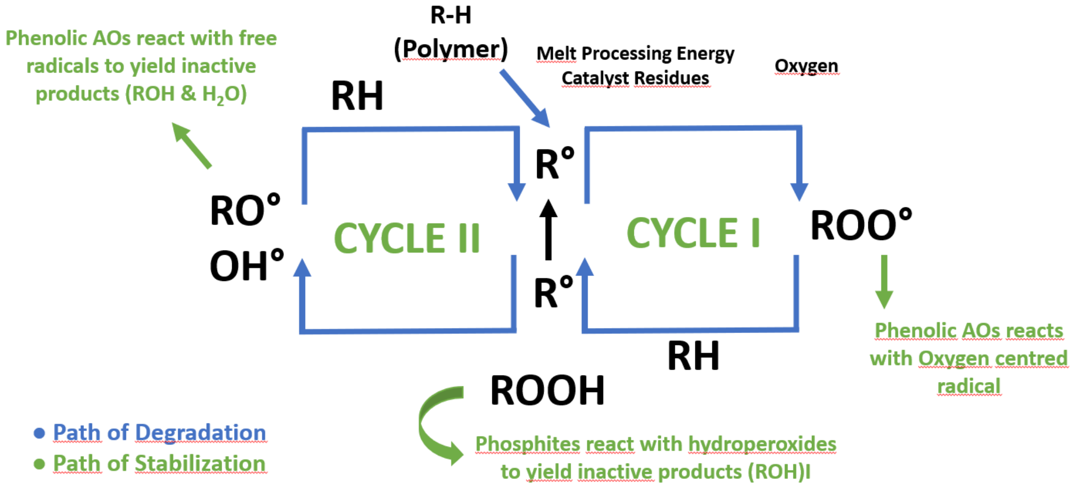
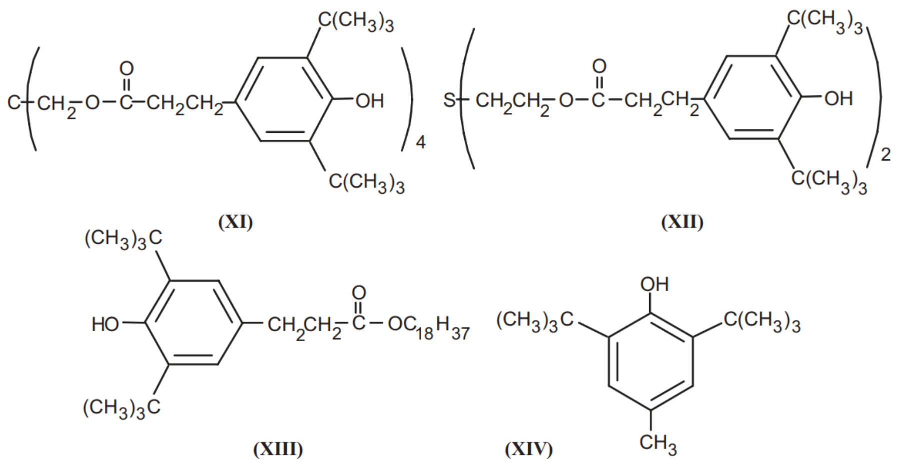
Thermooxidative degradation, stabilization and migration of stabilizers



Carbonyl index (**CI**) of rotational moldings versus molding time for the stabilizer different combinations.
Open symbols: pure PE / Solid symbol: PE/silica composites.



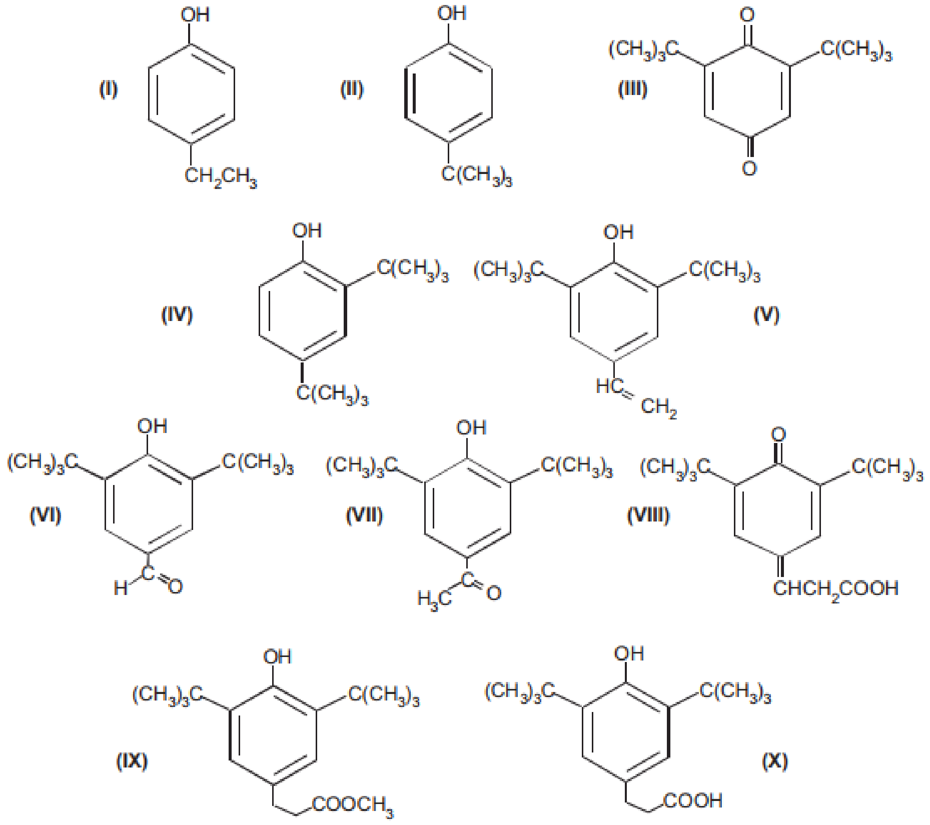
Autooxidation cycle for polymers.



Structures and names of some common polymer additives: (XI) Irganox 1010 (XII) Irganox 1035 (XIII) Irganox 1076 (XIV) 2,6-di-tert-butyl-4-methyl phenol (BHT)

*D. Brocca, et al., Water Res. 2002, 36, 3675; **E. Feyz, et al., J. Appl. Polym. Sci. 2008, 110, 1590.

Thermooxidative degradation, stabilization and migration of stabilizers



(I) 4-ethyl phenol
(II) 4-*tert*-butyl phenol
(III) 2,6-di- *tert*-butyl-p-benzoquinone
(IV) 2,4-di- *tert*-butyl phenol
(V) 3,5-di- *tert*-butyl-4-hydroxy styrene
(VI) 3,5-di- *tert*-butyl-4-hydroxy benzaldehyde
(VII) 3,5-di- *tert*-butyl-4-hydroxy aceto phenone
(VIII) Cyclo hexa 1,4 dien, 1,5-bis (tert-butyl), 6-on,4-(2-carboxy-ethylidene)
(IX) 3-(3,5-di- *tert*-butyl-4-hydroxyphenyl) methyl propanoate
(X) 3-(3,5-di- *tert*-butyl-4-hydroxyphenyl) propanoic acid

No.	PEX	MDPE I	MDPE II	LDPE
I				X
II		X	X	X
III			X	X
IV		X	X	
V		X	X	X
VI	X	X	X	X
VII	X	X	X	X
VIII	X	X	X	X
IX		X	X	X
X		x	X	X

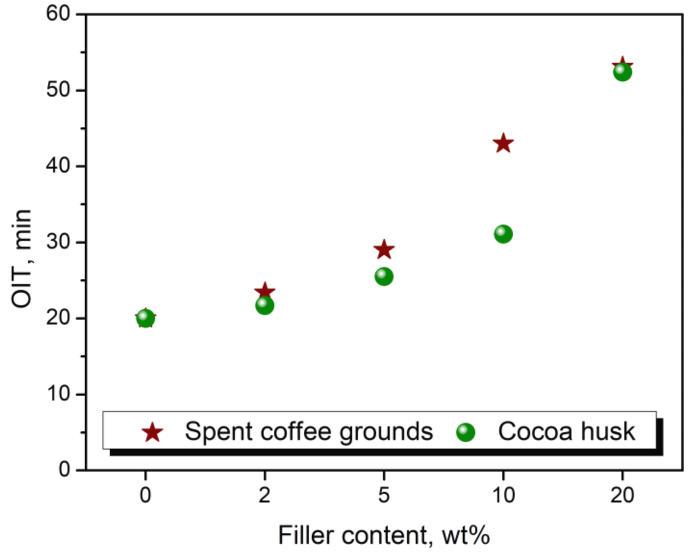
List of the most abundant organic compounds found in water which was in contact with various polyethylene samples.

*D. Brocca, et al., Water Res. 2002, 36, 3675.

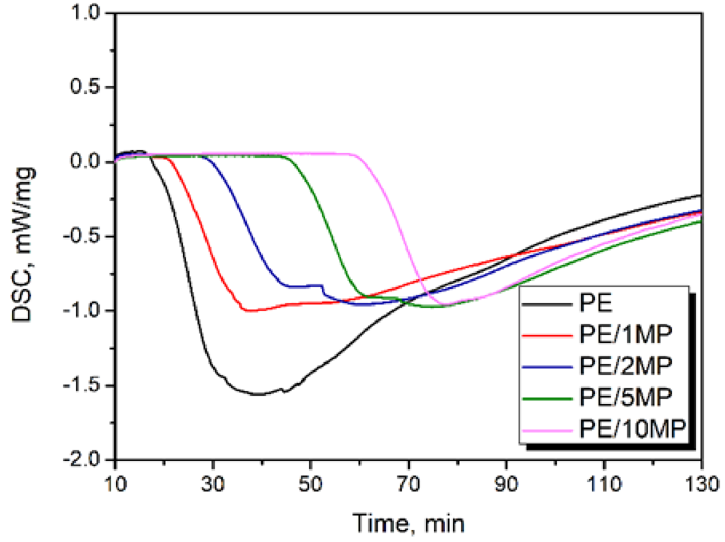
Application of polymer composites in RM

The use of waste fillers of plant origin with functional properties for the production of self-stabilizing wood polymer composites (WPC)

Migration of natural antioxidants to polyethylene



OIT for PE composites - coffee grounds/cocoa husks



DSC-OIT thermograms for composites PE-tangerine peel

Research on the influence of the polymer composites processing conditions on the stabilizing effect of functional plant-derived fillers SONATA-17 2021/43/D/ST8/01491
Implementation period : **11.07.2022 – 10.07.2025**

Principal Investigator: dr hab. inż. Mateusz Barczewski, prof. PP

NATIONAL SCIENCE CENTRE POLAND

Process and thermal stability of plant-based fillers

How fears of structural deterioration caused by lignocellulosic filler degradation led to a new path in WPC processing

Rotational molding of natural composites

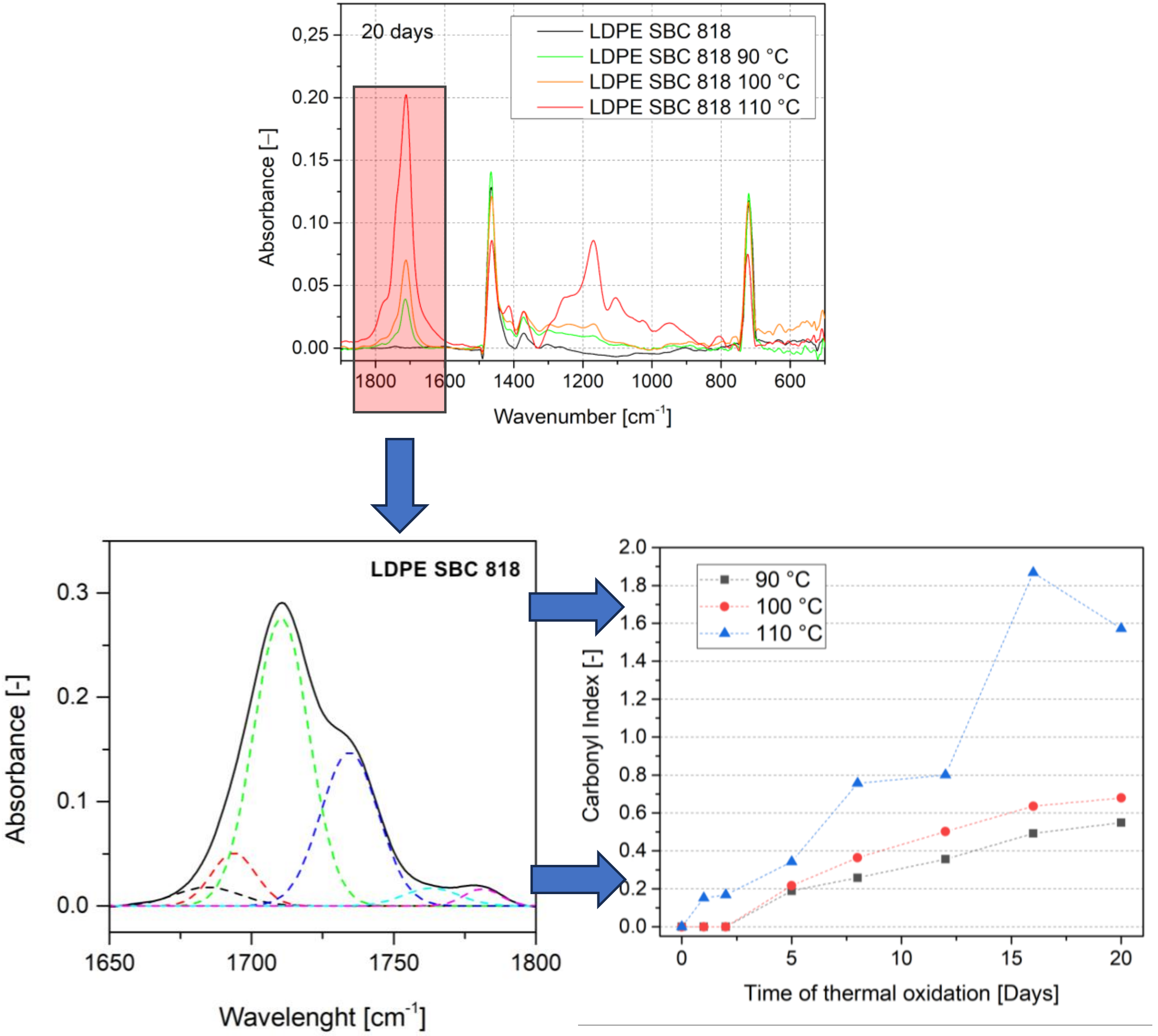
Polymers

Bio-based high-density polyethylene (**HDPE**) SHC 7260 I'm Green® (Braskem, Brazil); melt flow rate (MFR) 7.2 g/10 min (190°C/2.16 kg), density 0.959 g/cm³; content of ingredients of biological origin 94% (ASTM D6866).

Petrochemical high-density polyethylene (**HDPE**) KT 1000 UE (Dow, USA); melt flow rate (MFR) 8.0 g/10 min (190°C/2.16 kg), density 0.964 g/cm³.

Petrochemical high-density polyethylene (**HDPE**) GC 7260 (Basell Orlen Polyolefins, USA); melt flow rate (MFR) 8.0 g/10 min (190°C/2.16 kg), density 0.960 g/cm³.

Bio-based low-density polyethylene (**LDPE**) SBC 818 I’m Green Green® (Braskem, Brazil); melt flow rate (MFR) 8.3 g/10 min (190°C/2.16 kg), density 0.918 g/cm³; content of ingredients of biological origin 95% (ASTM D6866).



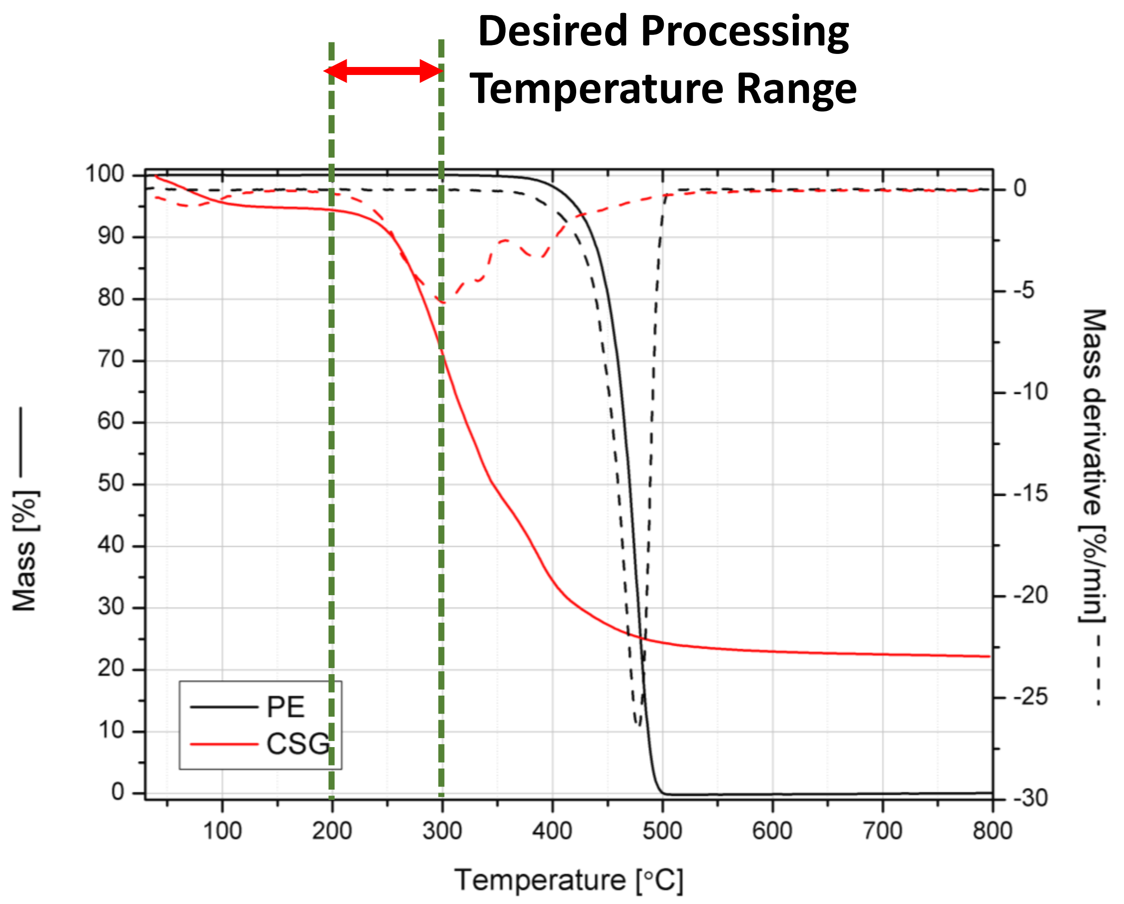
Filler

ASSAM TGFOP **black tea** (tippy golden flowery orange pekoe) country of origin: India; supplier: Zdrowie Natura (Poland). Brewing process: water:tea (150 g:1500 ml), temperature 90°C; brewing time 10 min.

Coffee-spent grounds (**CSG**); local franchise café – Poznań (Poland)

Nushells - **pistachio (PS), walnut (WS), and pecan (PES)**. The countries of origin of PS, WS, and PES were the United States of America, Poland (Roztocze Area), and Mexico, respectively. The harvest year of all nuts was 2022. Biomass was ground and sieved below 800 µm.

Beech wood flour (**WF**), country of origin: Poland. Filler was mechanically crushed and sieved below 400 µm.



TG and **DTG** curves of base materials: PE and CSG.

Rotational molding of natural composites

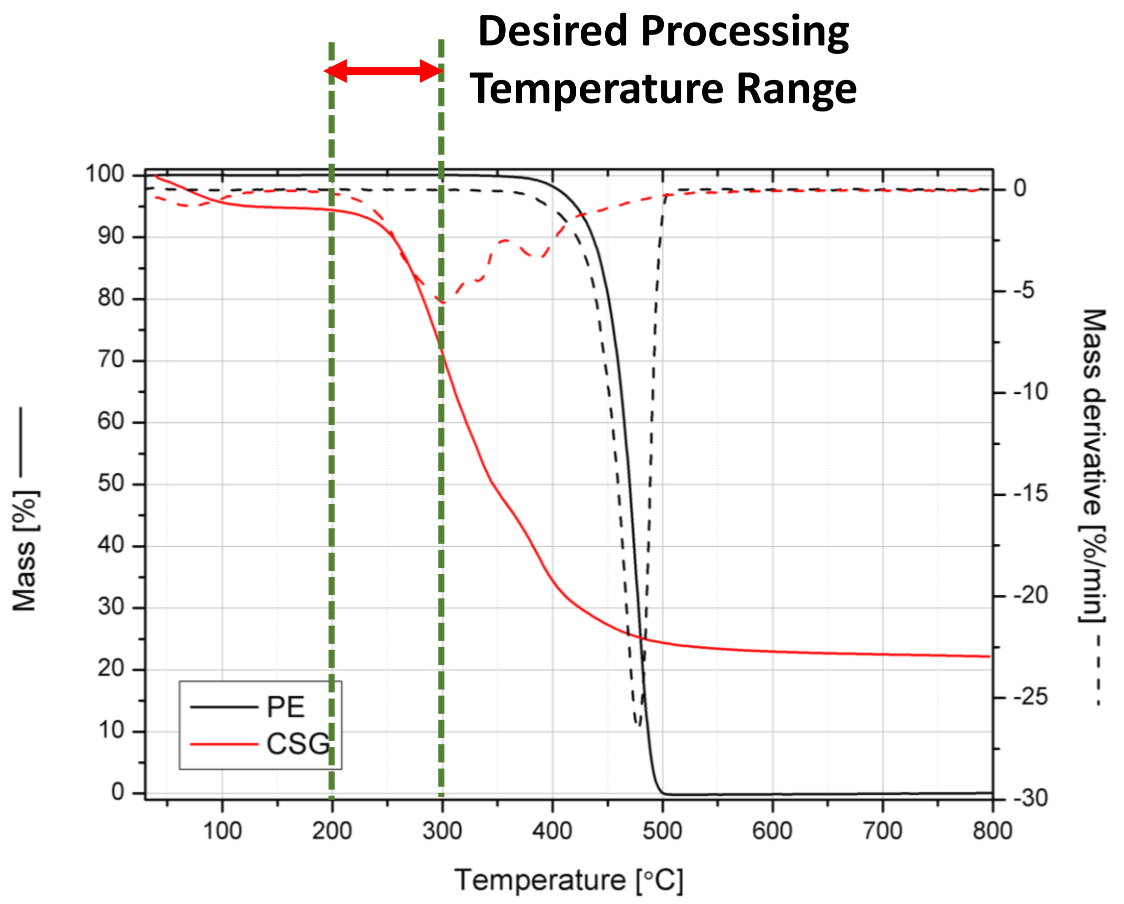
Main process- and material-oriented research questions:

- How much will the degradation of the lignocellulosic filler affect the deterioration of the RM structure of composites and their porosity?

- Will degradation in the sintering and densification process conditions limit the stabilizing effectiveness of the phytochemicals contained in the fillers?

- What manufacturing procedure (dry-blending/melt mixing) should be used for fillers with low thermal stability for RM purposes?

- How can the degradation processes of fillers be limited and controlled?



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Case study: The use of tea waste

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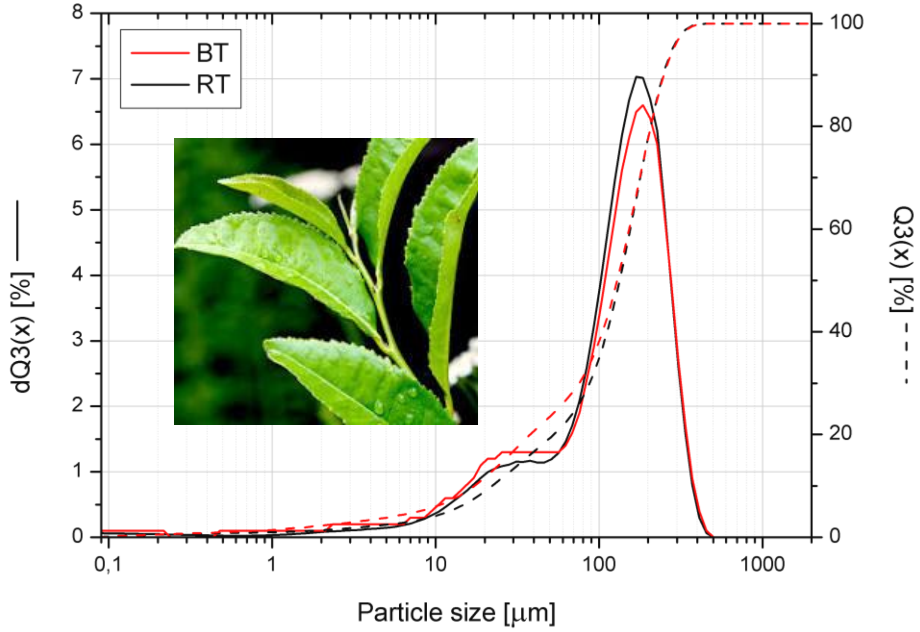
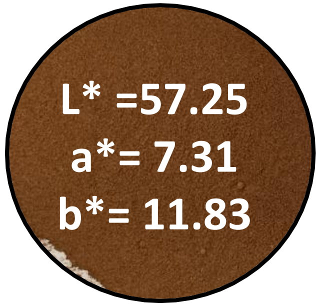
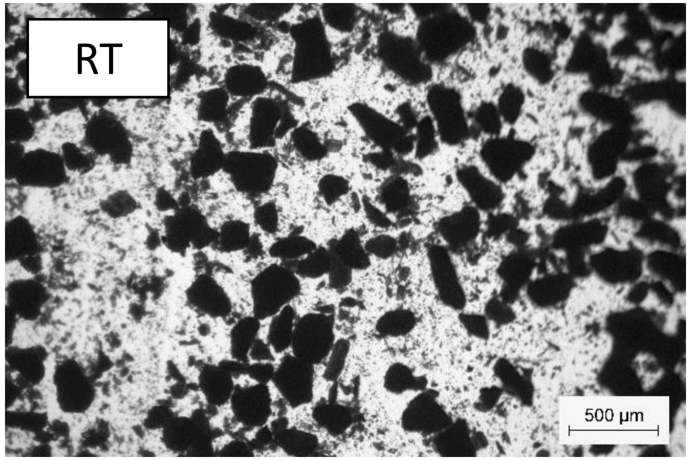


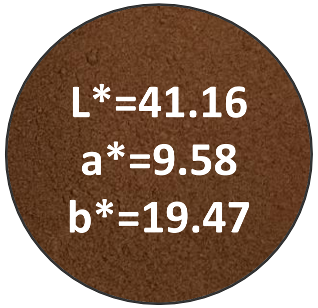
Particle size distribution of RT i BT.

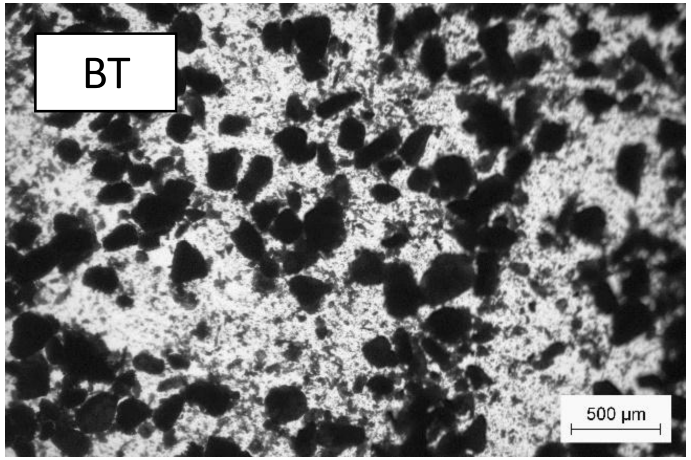
Raw tea (RT)





Tea after brewing (BT)





The external appearance of fractionated filler portions with the color determined in the CIE L*a*b* space and microscopic photographs of the fillers.

Case study: The use of tea waste

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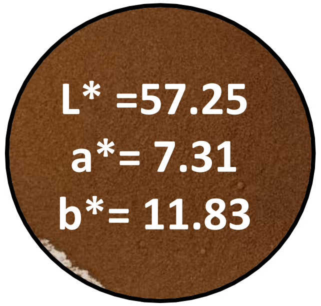
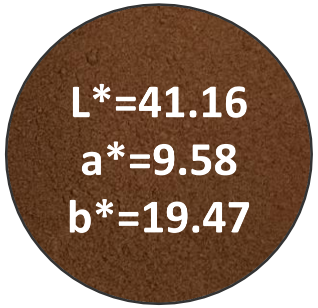
Sample (extract)	Concentration [g/l]	Antioxidant activity [%]	DRSC* [mg/g dry mass]
Raw tea (RT)	1	**50,11**	**79,00**
Brewed tea (BT)	1	**39,75**	**60,25**

Antioxidant capacity determined using UV-Vis spectroscopy using the DPPH method; **DPPH radical scavenging capacity*.

Raw tea (RT)



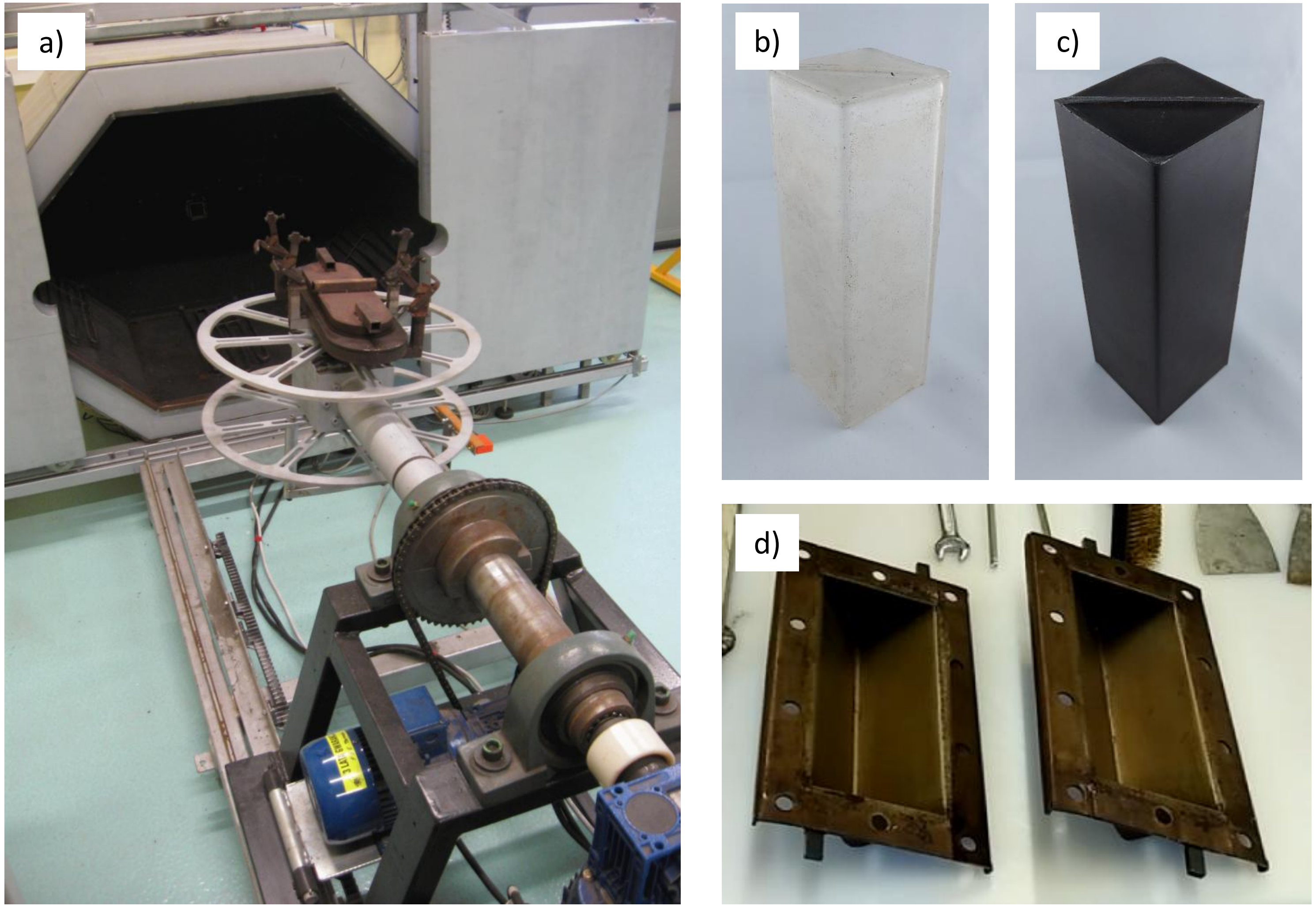
Tea after brewing (BT)



Component	% dry mass
Theogallin	1,0
Gallic acid	0,5
Quinic acid	2,0
Theaflavins (TF)	**5,6**
Thearubigins (TR)	**18**
β-carotene	0,006
Lutein	0,007
Violaxanthin	0,001
Neoxanthin	0,003
Theanine	3,1
Caffeine	3,2
Pectin	2,9
Proteins	7,2
Amino acids	6,1
Ash	5,0
Cellulose	6,2
Carbohydrates	12,1
Lignin	5,1
Lipids/acids	4,2
VOCs	0,01

External appearance of fractionated filler portions with color marking in the CIE L*a*b* space and chemical composition of the fillers.

Case study: The use of tea waste

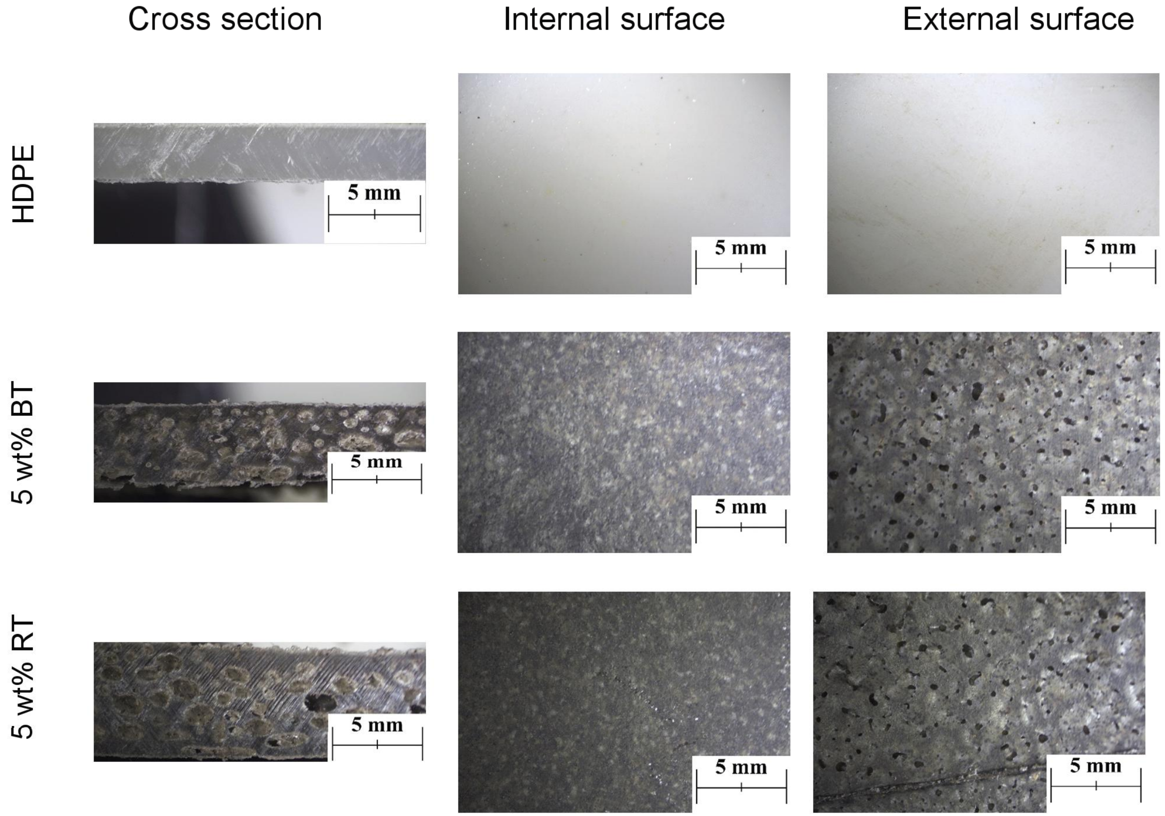


REMO GRAF single-arm shuttle rotational molding machine (a), products made of HDPE and HDPE-BC composite (b,c), steel mold (d).

Rotational molding (RM)

REMO GRAF single-arm shuttle machine
Rotational speed: 8 / 5 rpm
Temperature: 230°C (200°C)
Heating phase time: 20 min

The use of waste fillers of plant origin with functional properties for the production of self-stabilizing wood polymer composites (WPC)



Optical microscope images of the HDPE and composite rotomolded samples' cross-section, internal and external surface.

Properties	Physical mixing			Melt mixing		
	HDPE	5 wt% BT	5 wt% RT	HDPE	5 wt% BT	5 wt% RT
Elastic modulus, MPa	1240 ± 62	468 ± 67	367 ± 46	1370 ± 52	677 ± 74	647 ± 71
Tensile strength, MPa	22.8 ± 2.3	5.9 ± 1.9	4.8 ± 1.6	24.9 ±1.8	7.7 ± 1.2	7.6 ± 2.1
Elongation at break, %	8.8 ± 2.8	3.9 ± 1.9	3.6 ± 1.1	11.0 ± 1.7	3.2 ± 0.9	3.6 ± 0.8
Hardness, MPa	29.7 ± 3.1	19.0 ± 3.9	12.7 ± 4.2	34.1 ± 6.3	12.1 ± 2.8	15.1 ± 3.2

Results of mechanical properties of HDPE and HDPE-BT/RT composites.

M. Barczewski, et al. Composites Part C: Open Access 2024, 13, 100437.

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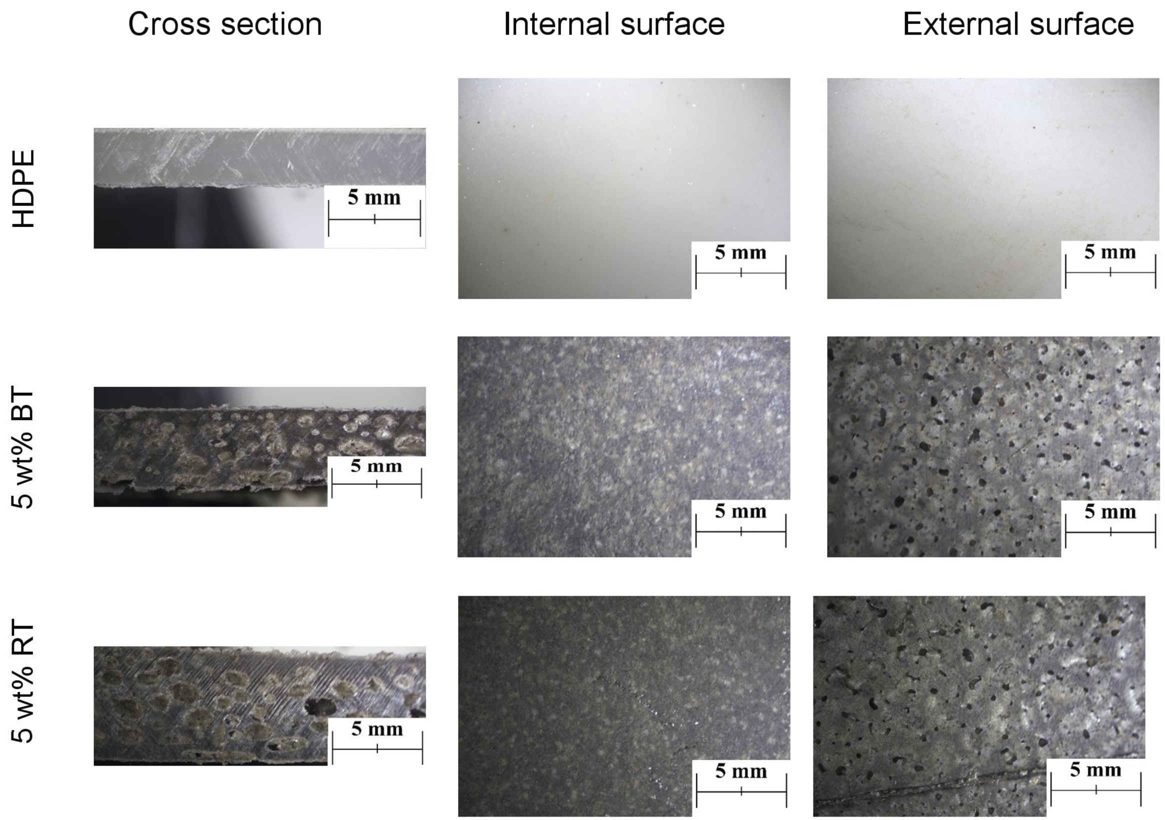

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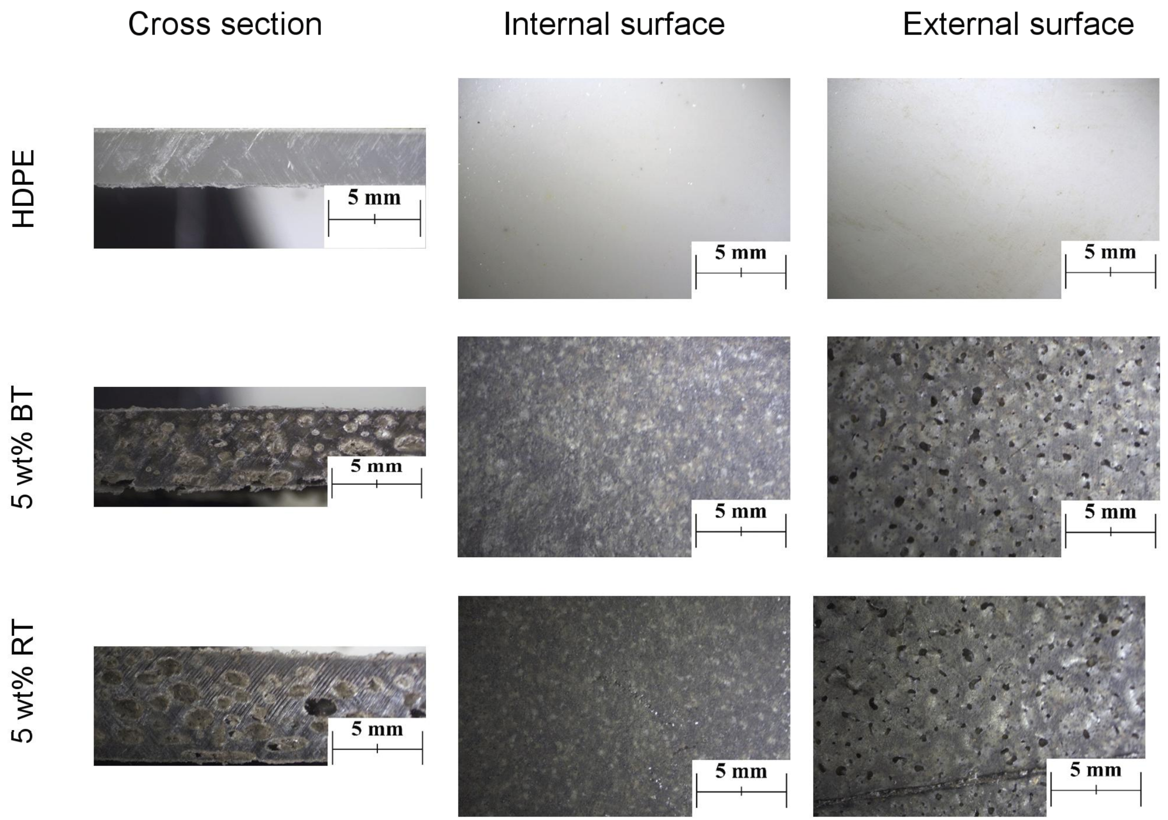
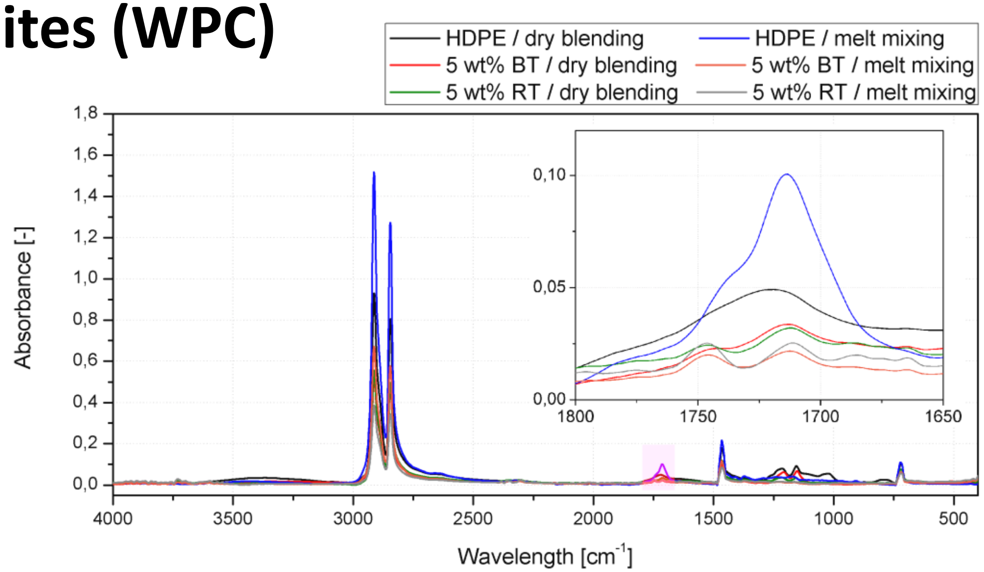
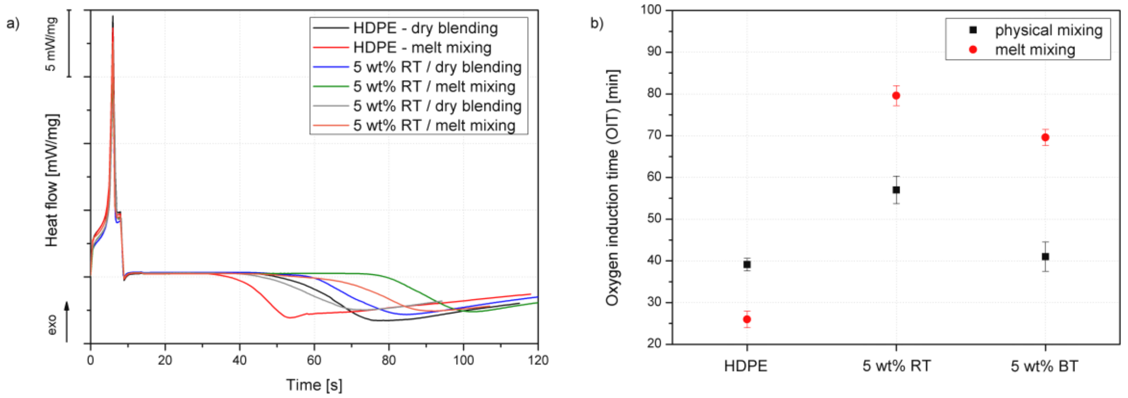


Optical microscope images of the HDPE and composite rotomolded samples' cross-section, internal and external surface.



FTIR spectra of rotomolded PE – black tea composites



DSC-OIT test results; heat flow curves (a); average OIT values (b)

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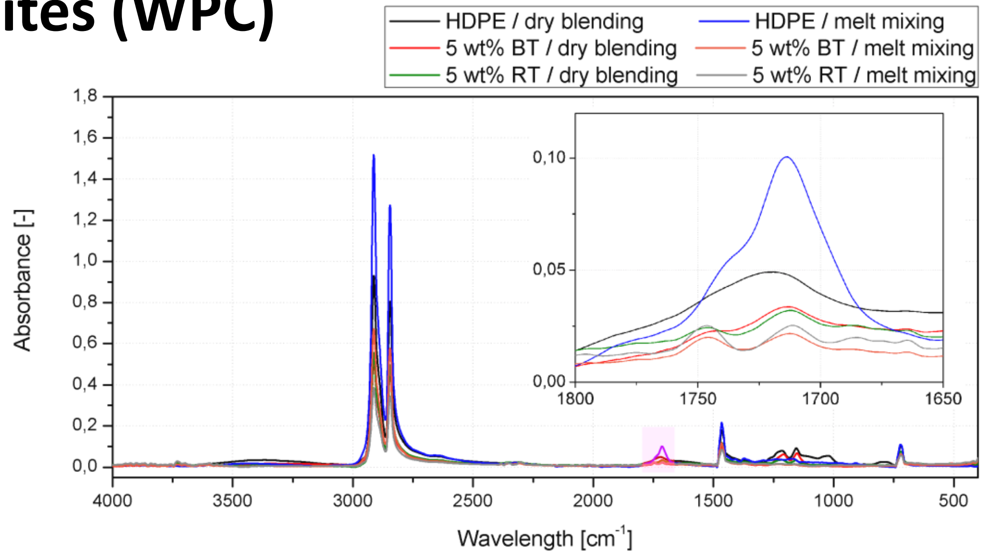
Case study: The use of tea waste

The use of waste fillers of plant origin with functional properties for the production of self-stabilizing wood polymer composites (WPC)

Thermal degradation of lignocellulosic filler is not synonymous with degradation of antioxidants!

Despite the structural defects of rotomolded parts, the polymer did not undergo thermo-oxidative degradation!

Optical microscope images of the HDPE and composite rotomolded samples' cross-section, internal and external surface.



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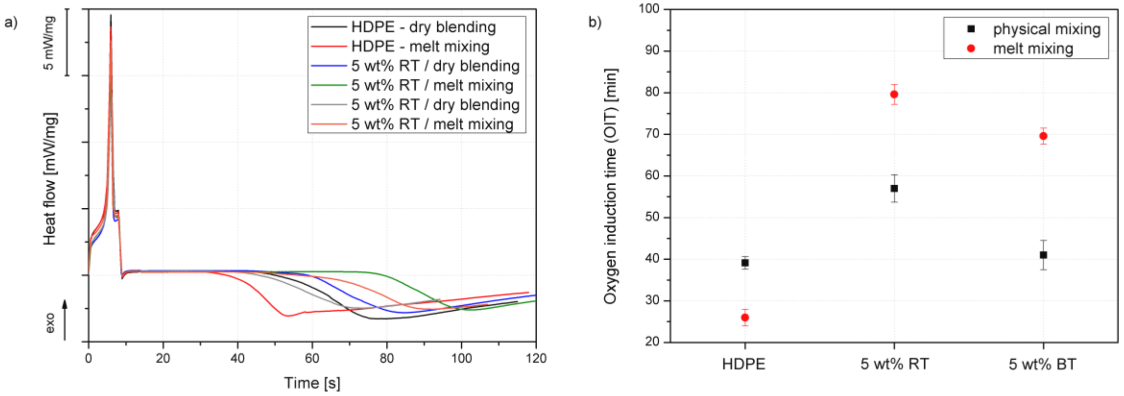

DSC-OIT test results; heat flow curves (a); average OIT values (b)

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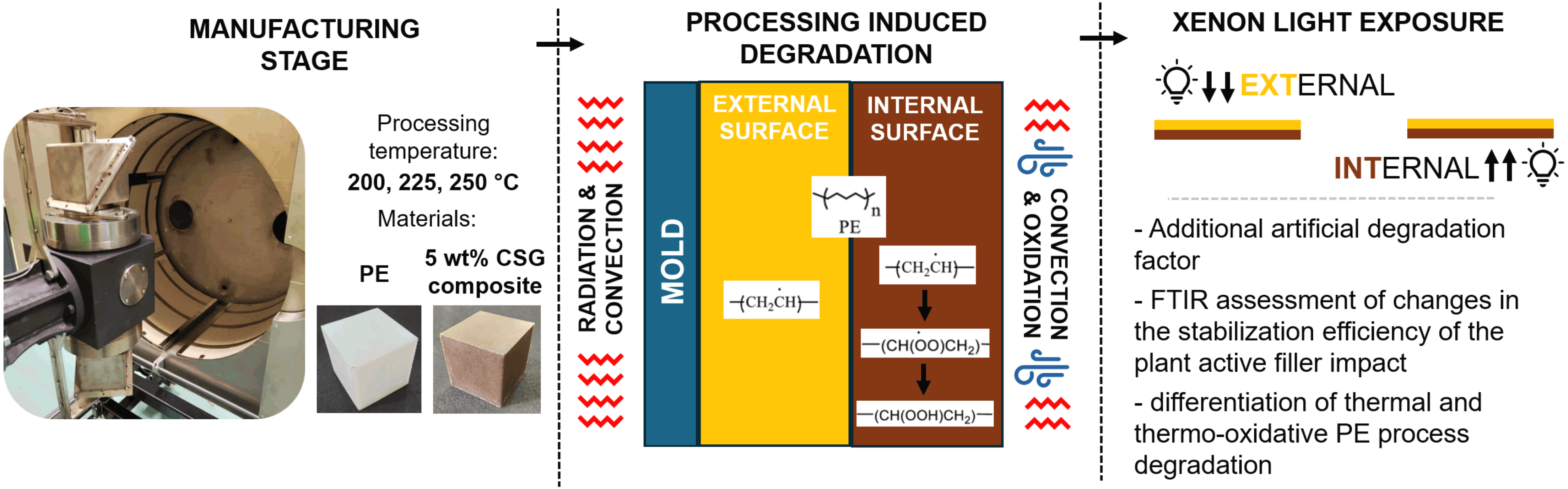
Case study: The use of coffee-spent grounds



Scheme of the experimental concept and research work.

Case study: The use of coffee-spent grounds

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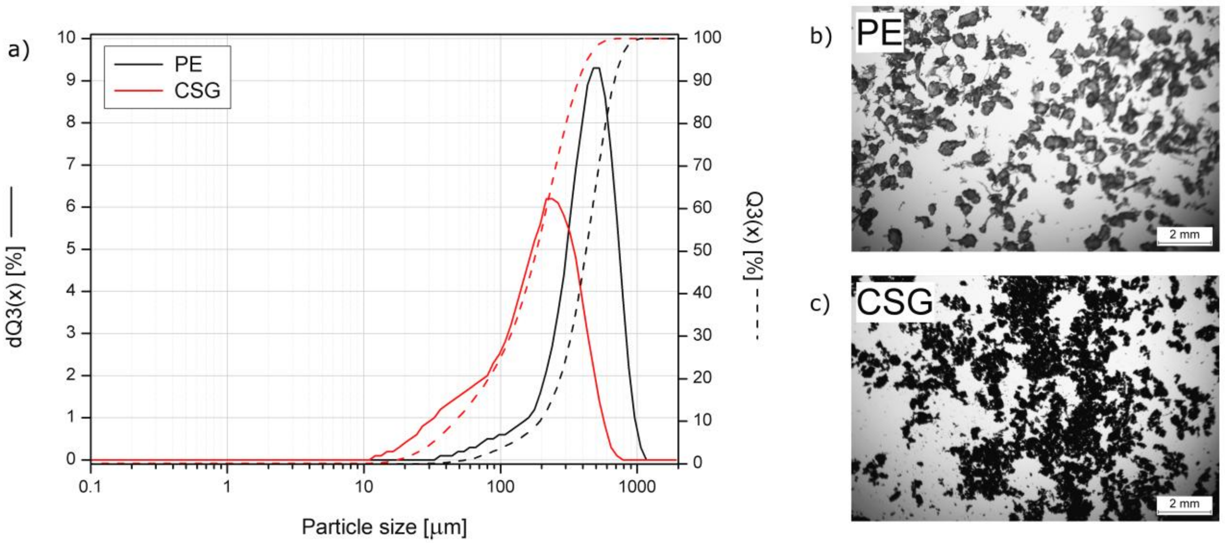
Filler:

Coffee-spent grounds (**CSG**); local franchise café – Poznań (Poland)



Particle size distribution of PE and CSG (a); digital microscope images of PE (b) and CSG (c) before processing by rotational molding.

Parameter	Unit	CSG
Chemical composition		
Holocellulose	[%]	50.73
Cellulose	[%]	20.85
Lignin	[%]	24.73
Mineral substances	[%]	2.22
Extractives	[%]	34.33
Total flavonoid content		
Catechin equivalent	[mg/g dry mass]	13.64
Antioxidant activity		
Inhibition	[%]	20.7
DRSC	[mg/g dry mass]	25.8
Thermal properties		
TGA 5% temp.	[°C]	127
TGA 10% temp.		255

Chemical composition and antioxidant activity evaluation of CSG.

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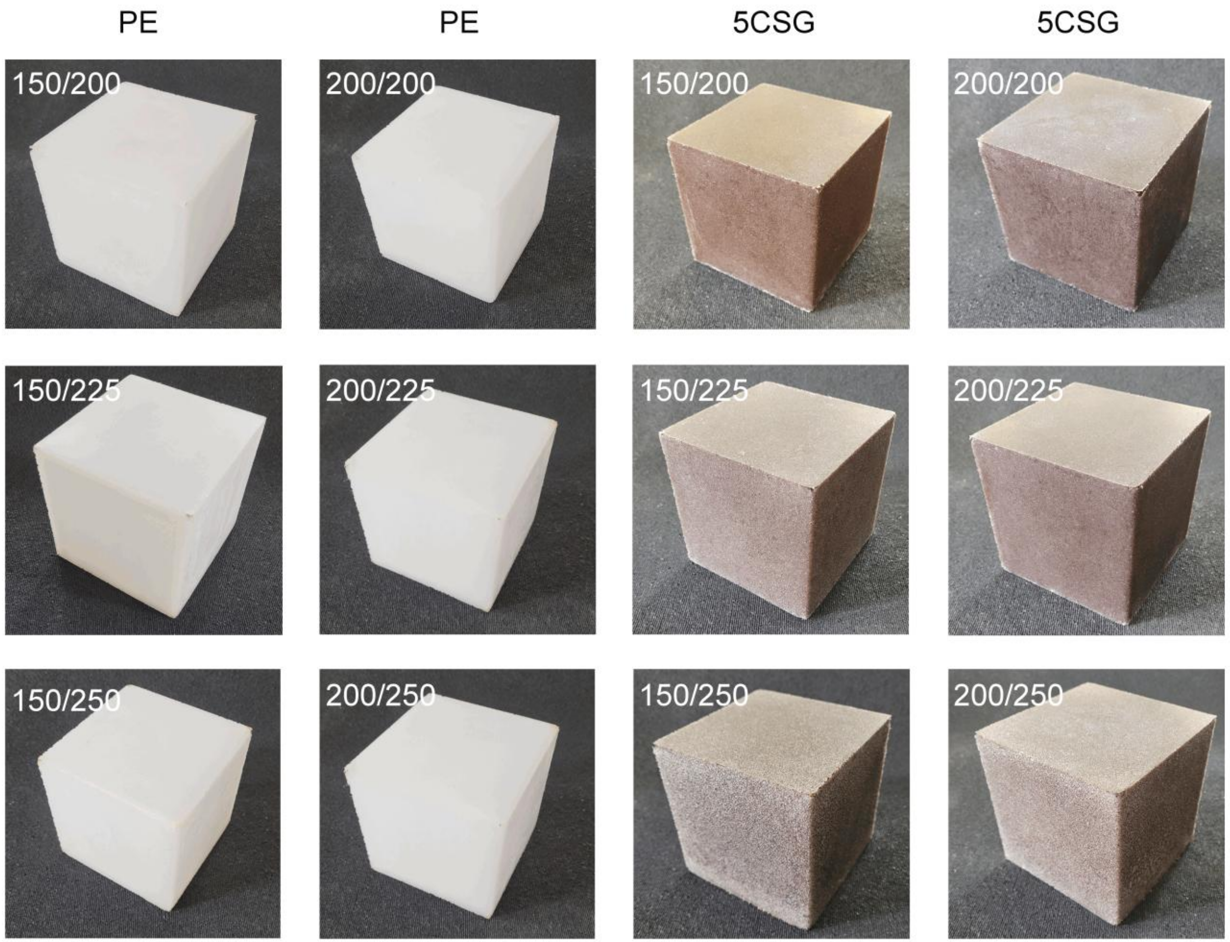
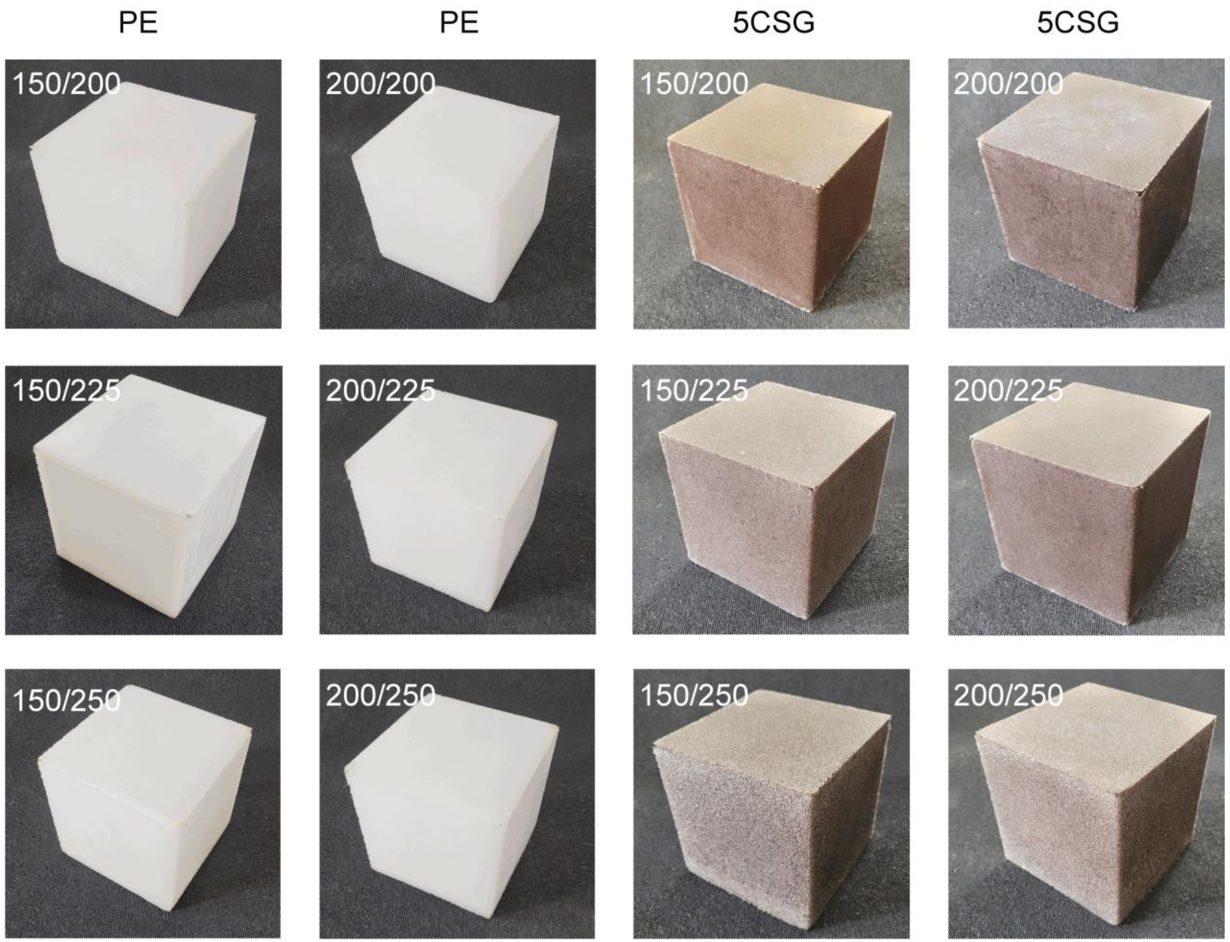


Photographs of rotomolded PE and composite parts manufactured with different temperature sets and material weights.

Łukasiewicz
Instytut Inżynierii Materiałów Polimerowych i Barwników

A prototype rotational molding machine built for the project

Case study: The use of coffee-spent grounds



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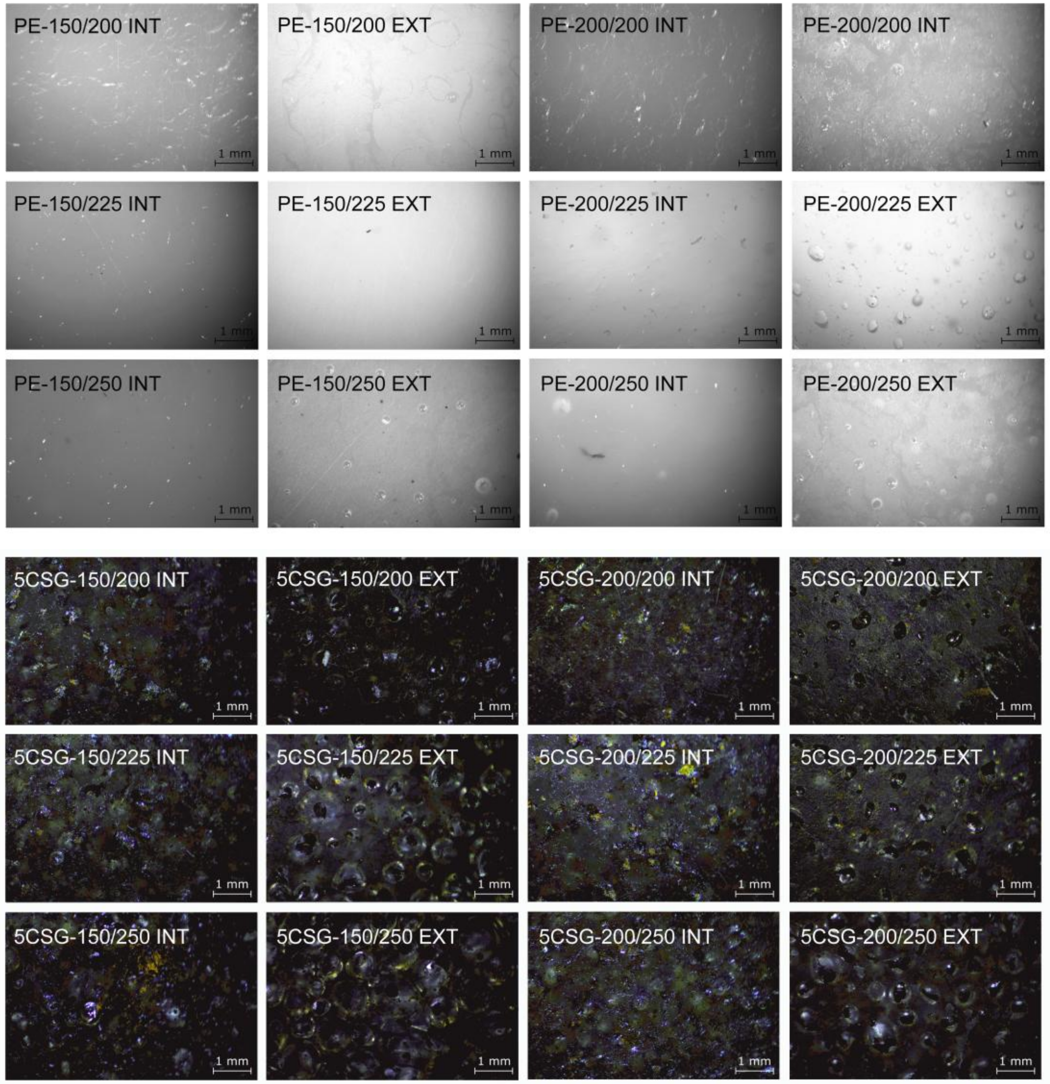

Microscopic images showing the internal and external surfaces of rotomolded products made of PE in processing temperatures.

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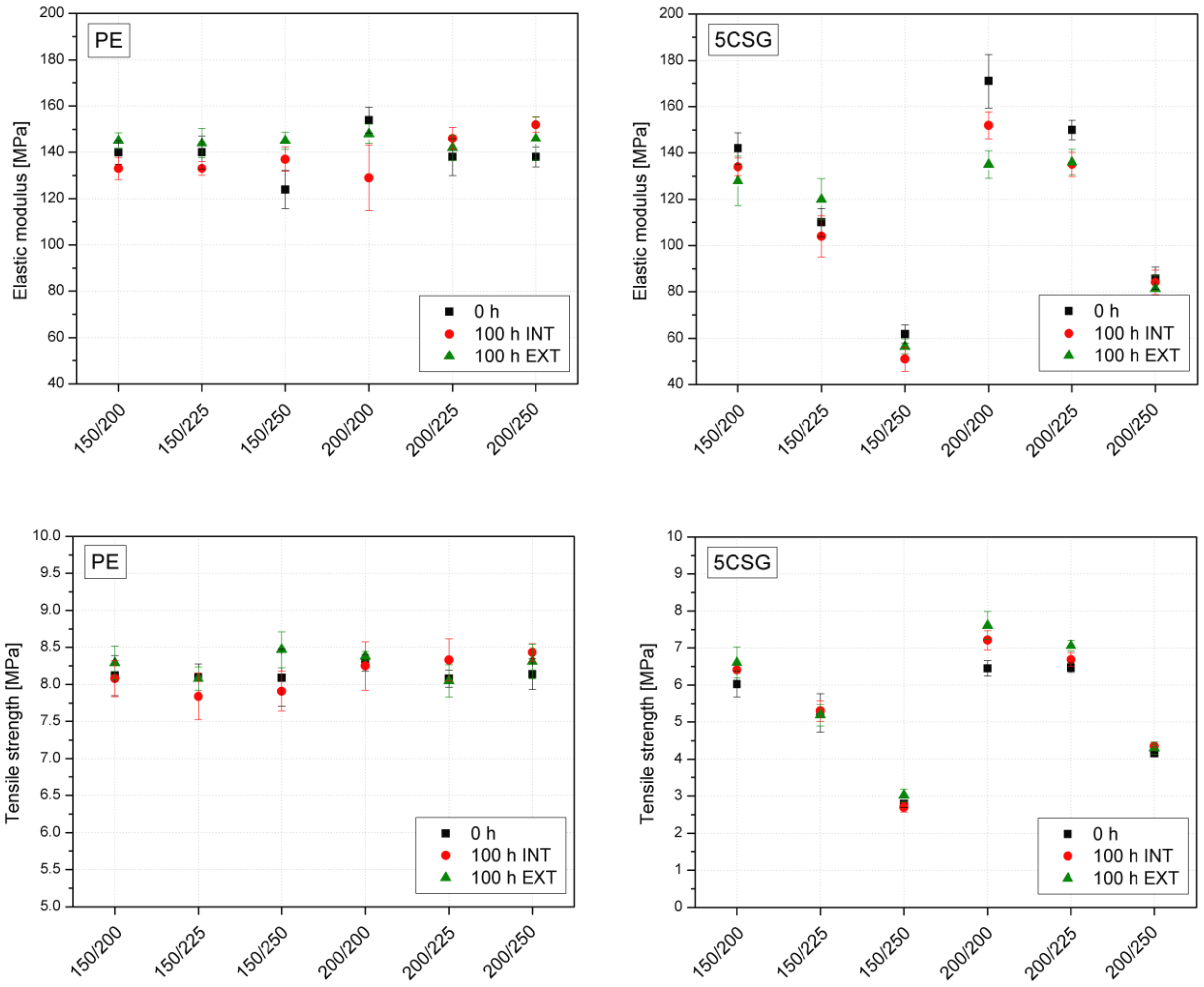
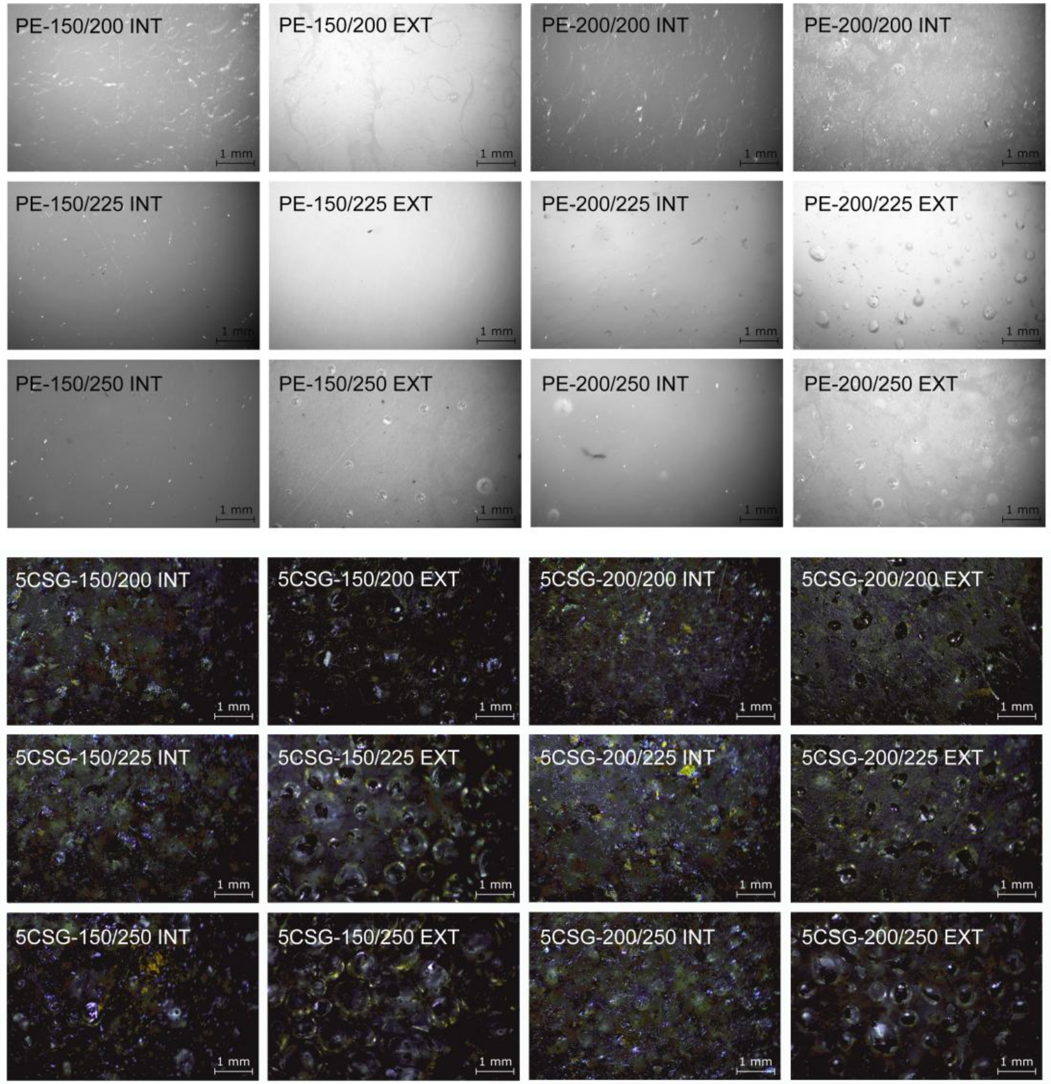


Mechanical properties of PE and composite containing 5 wt% CSG before and after exposure to 100 h UV-light irradiation of internal (INT) and external (EXT) surface.



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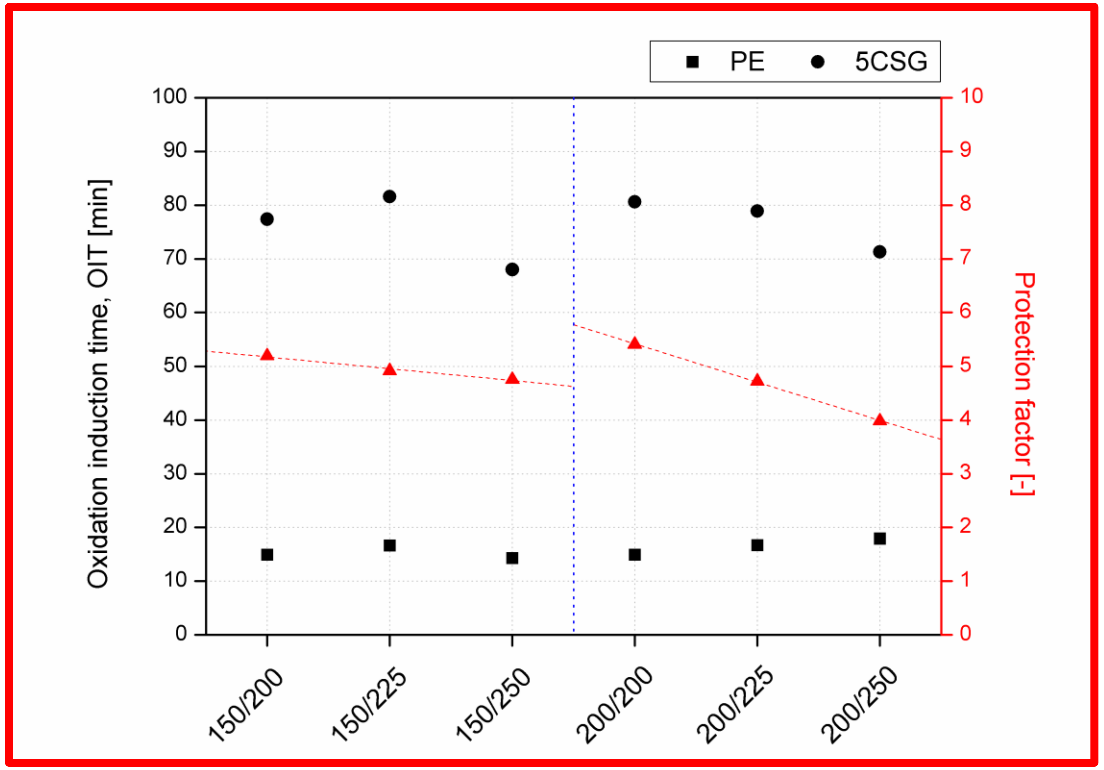
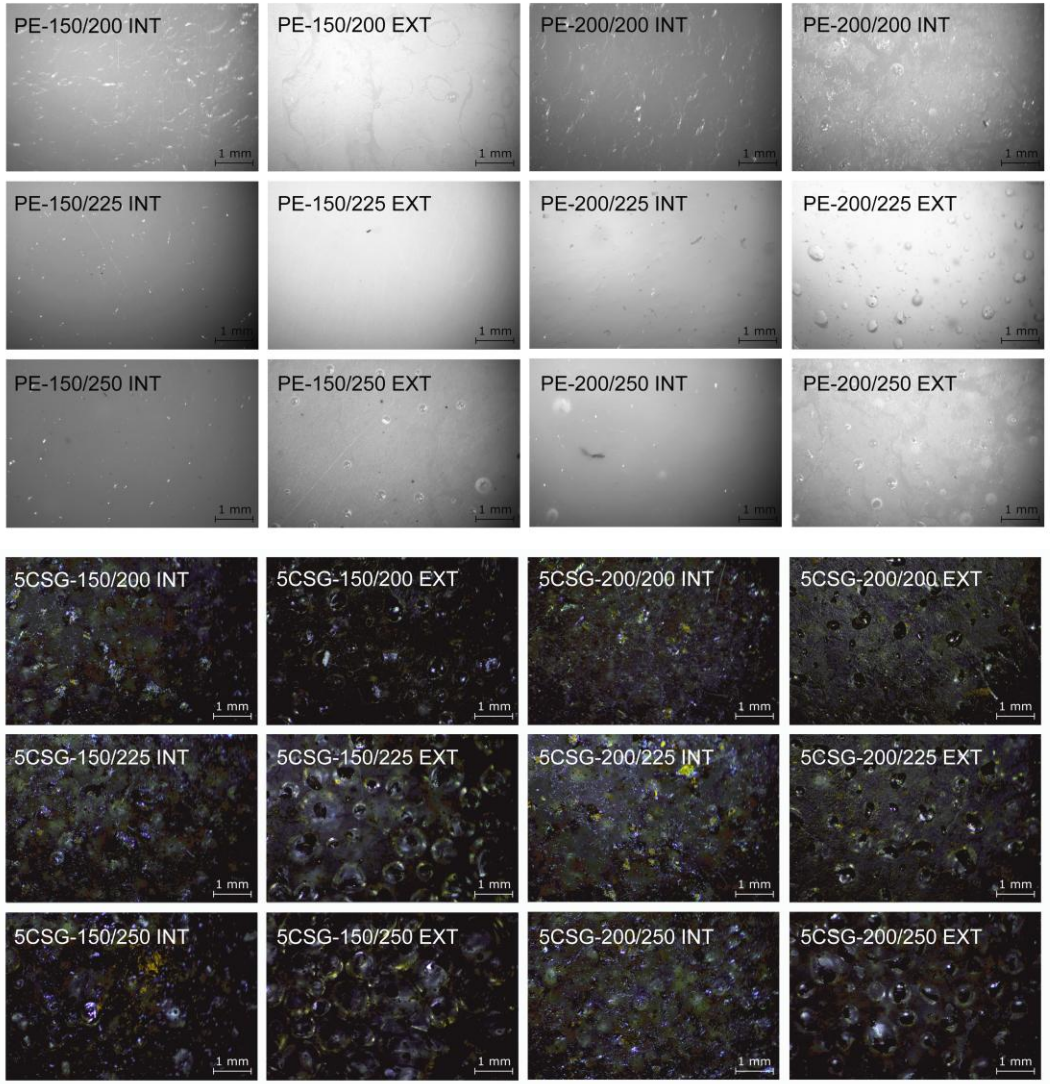


Oxidation induction time (OIT) and protection factor (PF) of PE and composite samples after rotomolding at various processing temperatures.



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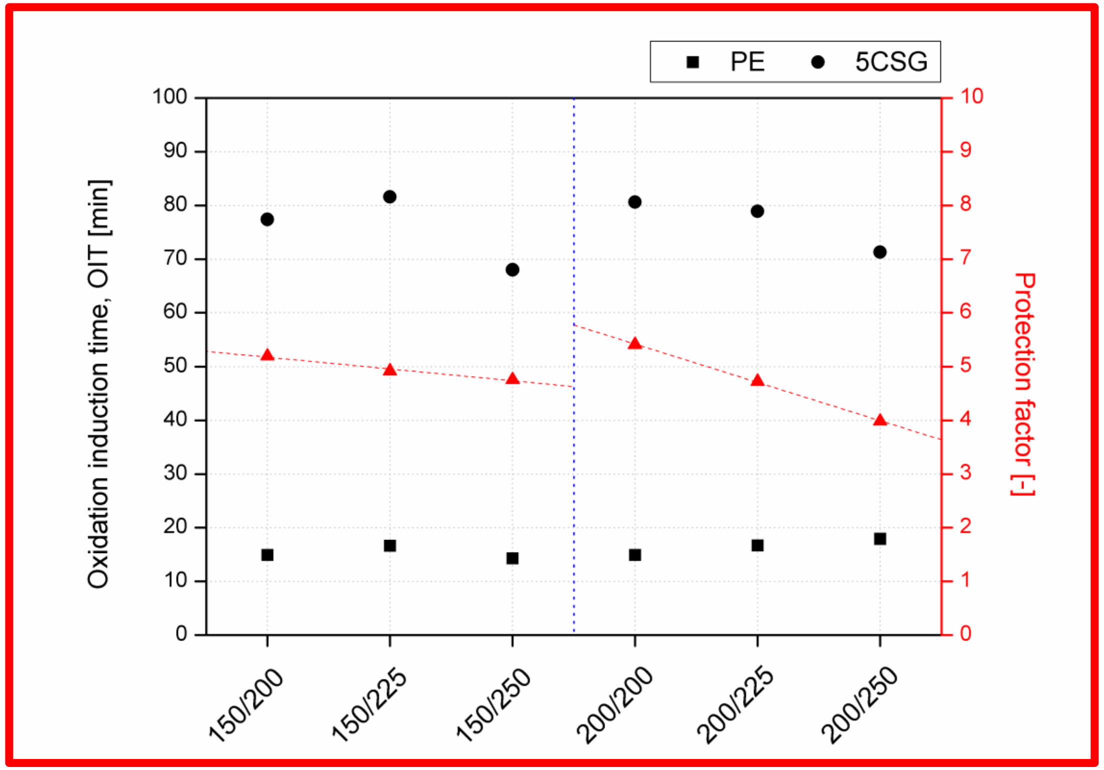
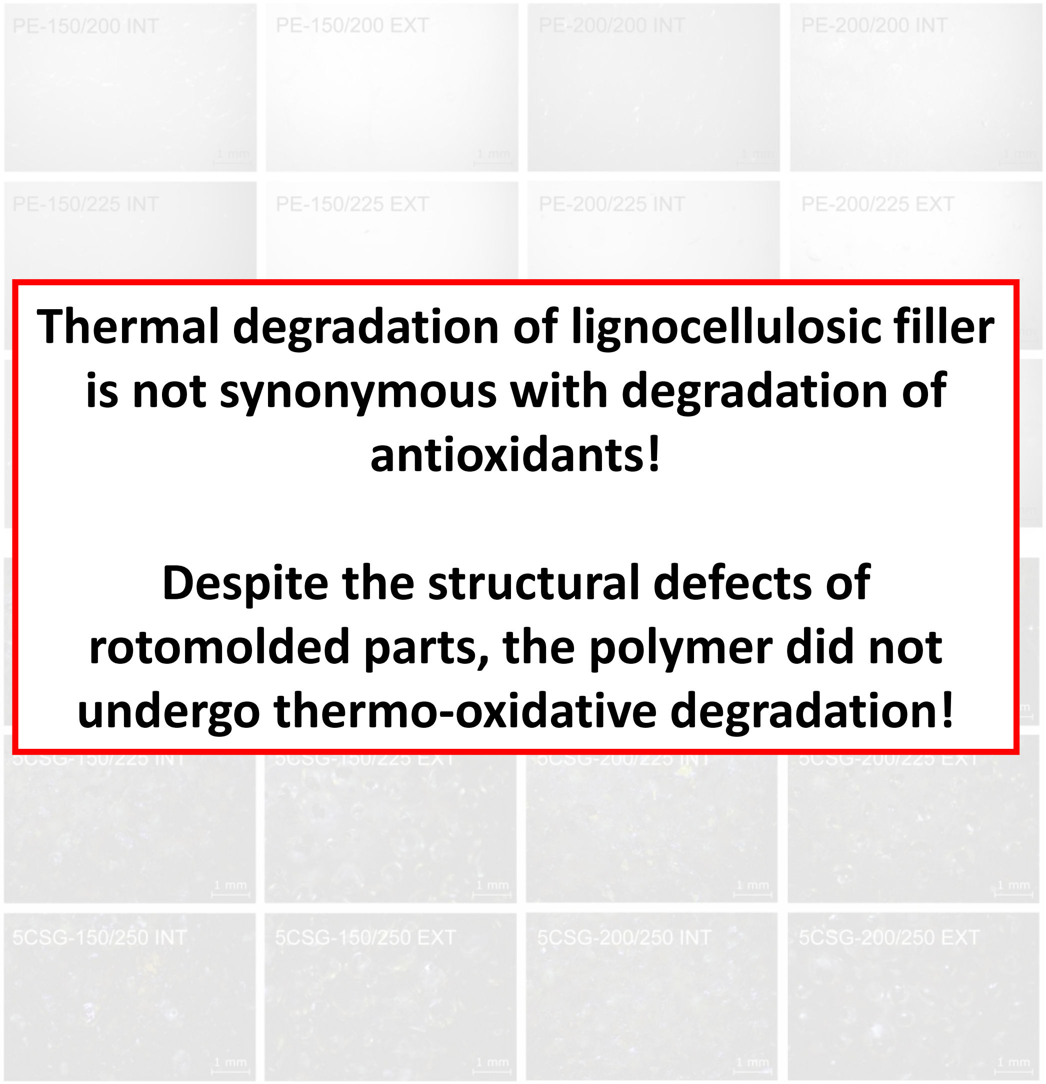


Oxidation induction time (OIT) and protection factor (PF) of PE and composite samples after rotomolding at various processing temperatures.

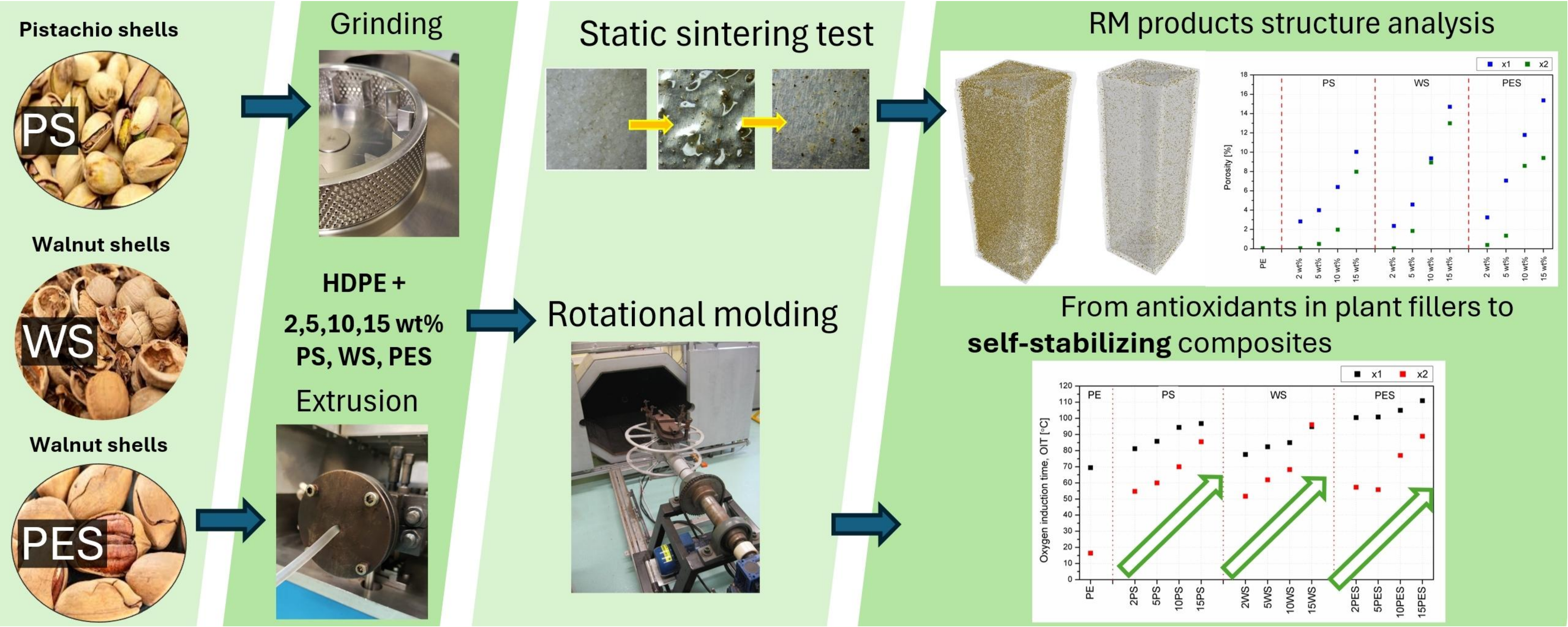


Thermal degradation of lignocellulosic filler is not synonymous with degradation of antioxidants!

Despite the structural defects of rotomolded parts, the polymer did not undergo thermo-oxidative degradation!

Microscopic images showing the internal and external surfaces of rotomolded products made of PE in processing temperatures.

Case study: Using nutshells as active fillers



Case study: Using nutshells as active fillers

Polymer

Bio-based high-density polyethylene (HDPE) SHC 7260 I'm Green® (Braskem, Brazil); melt flow rate (MFR) 7.2 g/10 min (190°C/2.16 kg), density 0.959 g/cm^3; content of ingredients of biological origin 94% (ASTM D6866).

Fillers

Pistachio shells (PS) country of origin: USA; supplier: NATURAL EXPERT PPH Kamil Chojnowski
Wallnut shell (WS) country of origin: Poland; supplier: MIREX Mirosław Gronert
Pecan shell (PES) country of origin: USA; supplier: Just-And Andrzej Błachowicz


PS


PES


WS

Parameter	Unit	PS	WS	PES
Chemical composition				
Holocellulose	[%]	88.26	84.66	75.88
Cellulose	[%]	32.69	29.98	27.92
Lignin	[%]	29.27	47.94	44.57
Mineral substances	[%]	0.2	0.46	1.67
Extractives	[%]	1.07	1.62	0.87
Fat	[%]	1.44	3.14	3.26
Total flavonoid content				
Catechin equivalent	[mg/g dry mass]	2.71±0.14	5.19±0.18	32.61±1.34
Antioxidant activity				
Inhibition	[%]	20.91±2.13	21.26±0.14	88.40±0.47
Trolox equivalent	[mg/l]	26.18±3.85	26.81±0.25	148.27±0.56
TAEC	[mg/g dry mass]	26.18±3.85	26.81±0.25	148.27±0.86
Thermal properties				
Decomposition temperature	[°C]	214.6	208.4	214.7

Chemical composition and results of antioxidant activity evaluation of fillers.

Case study: Using nutshells as active fillers

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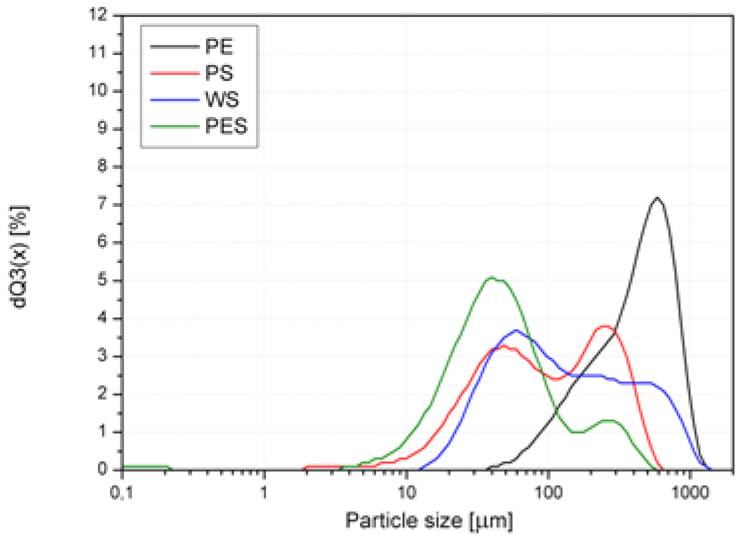

Histogram representing particle size distribution of fillers.

Parameter	Unit	PS	WS	PES
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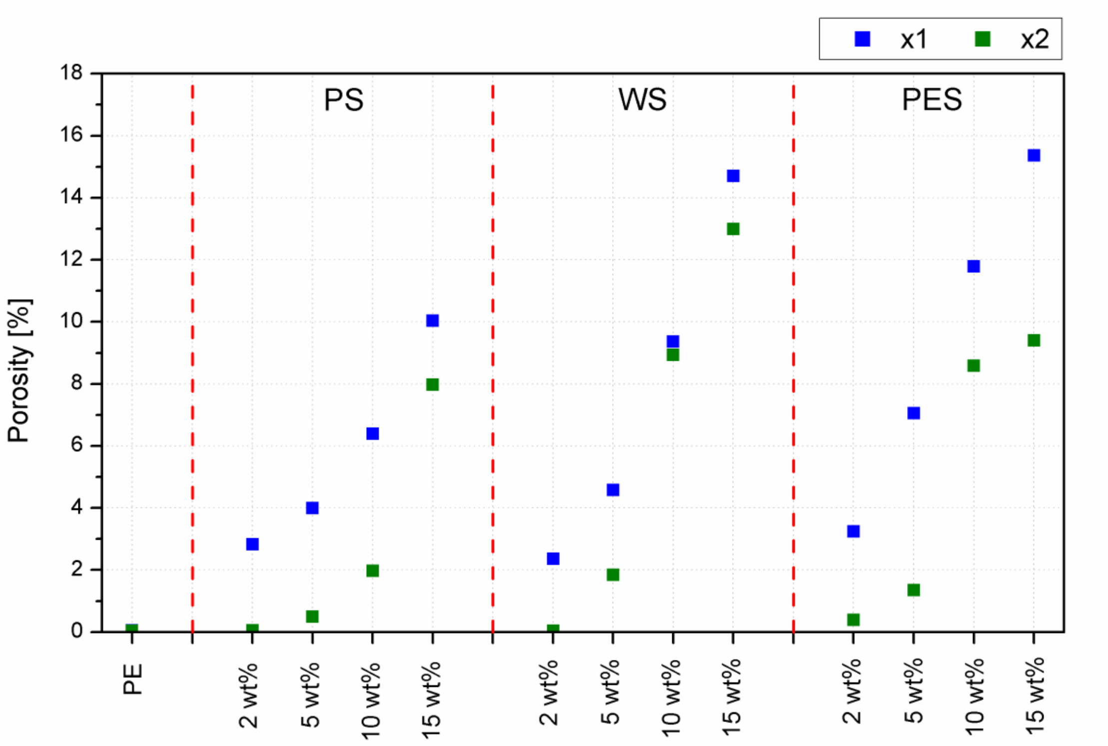
Chemical composition and results of antioxidant activity evaluation of fillers.

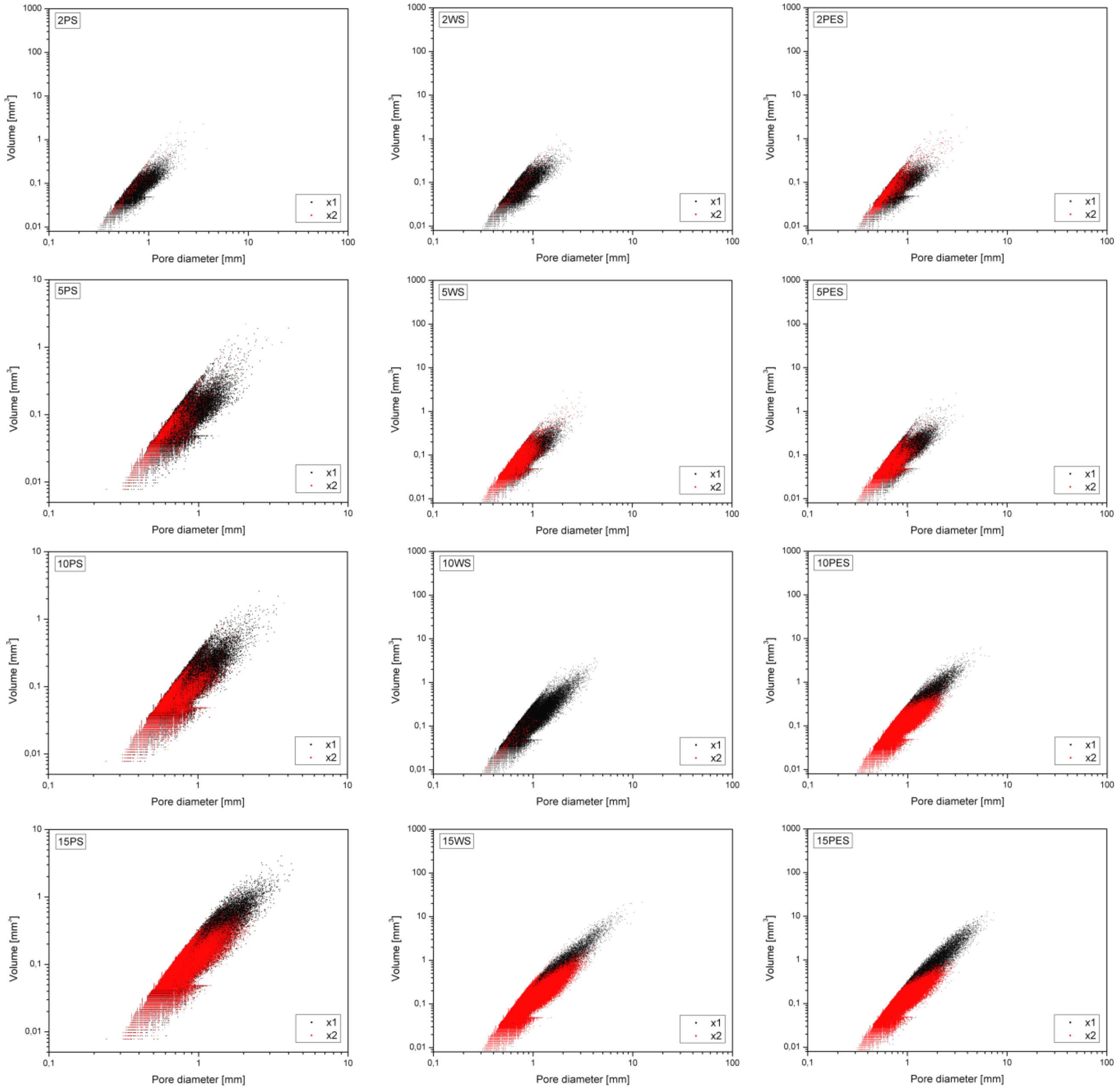
Case study: Using nutshells as active fillers

3D computed tomography (3D CT)



Total porosity of the rotomolded samples.

Porosity distribution in samples (pore volume vs. diameter plots) based on 3D tomography.



9th Rotopol Meeting 2025
29-30.05.2025 Wieliczka/Cracow

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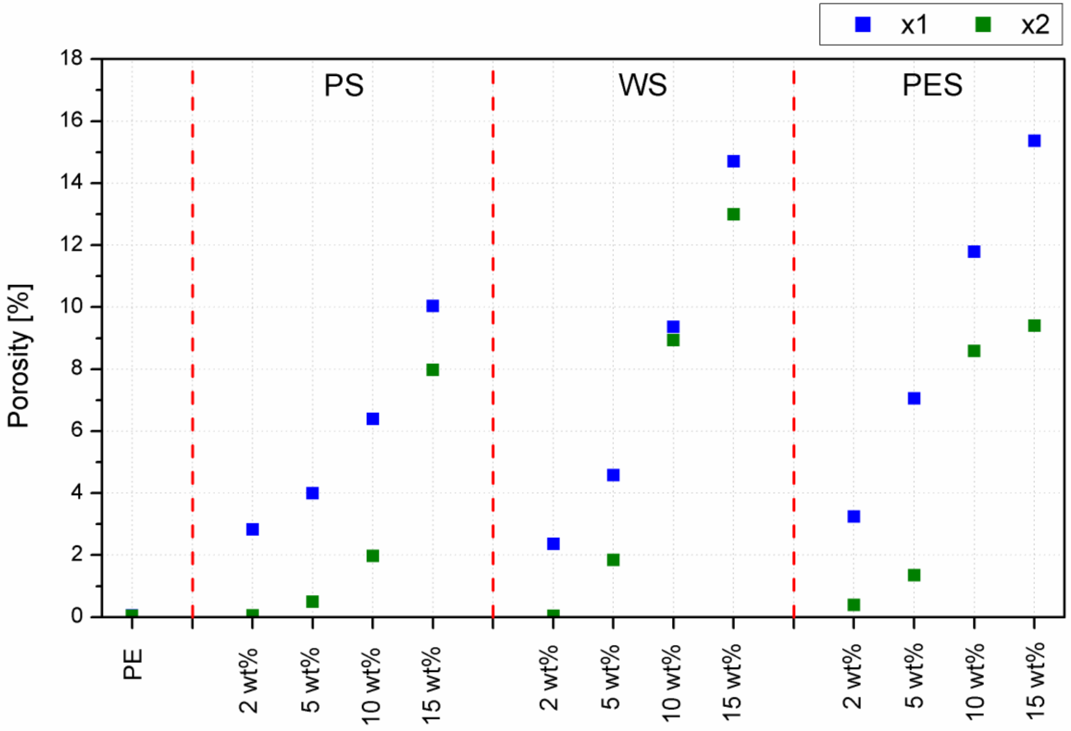
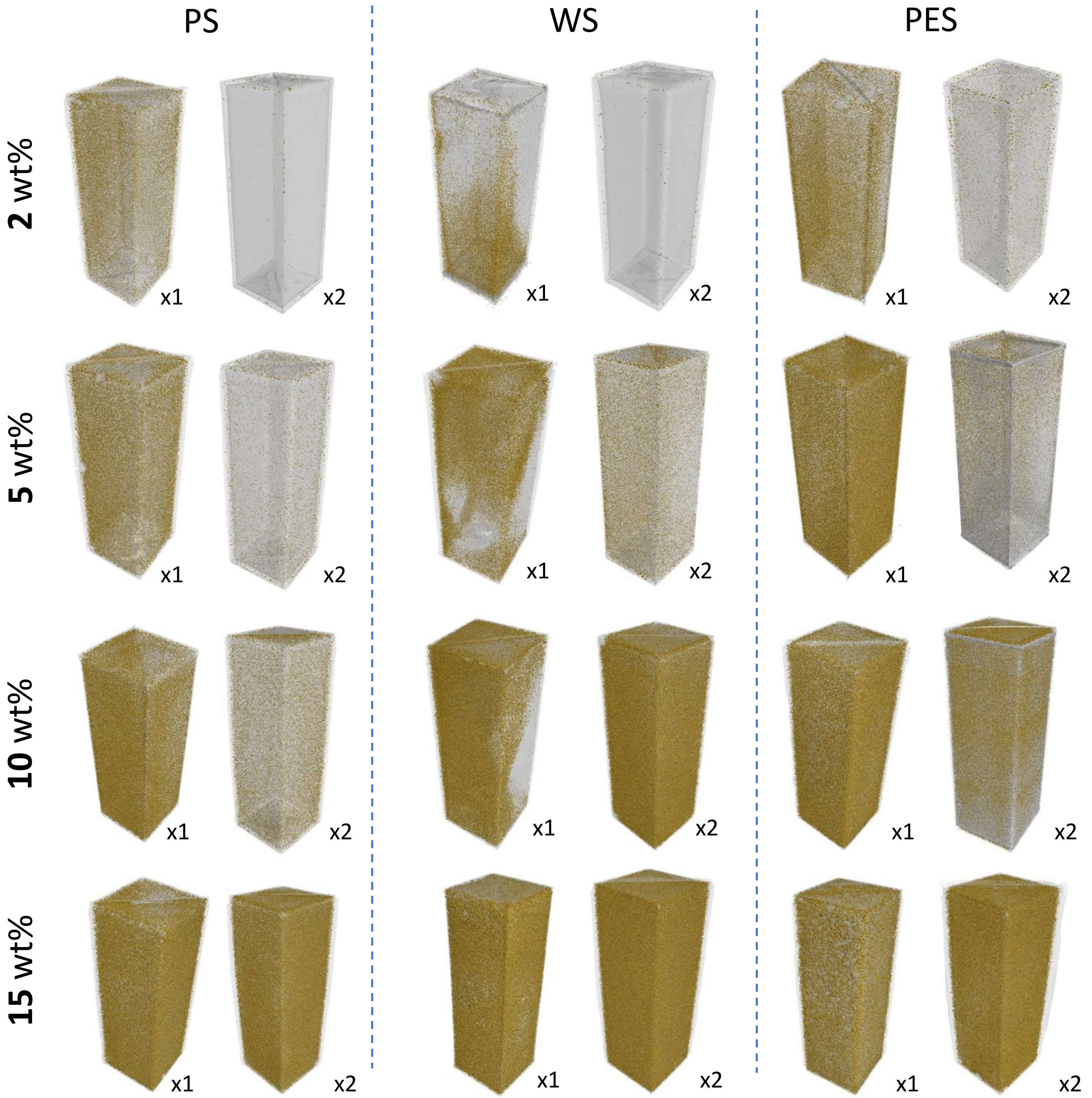


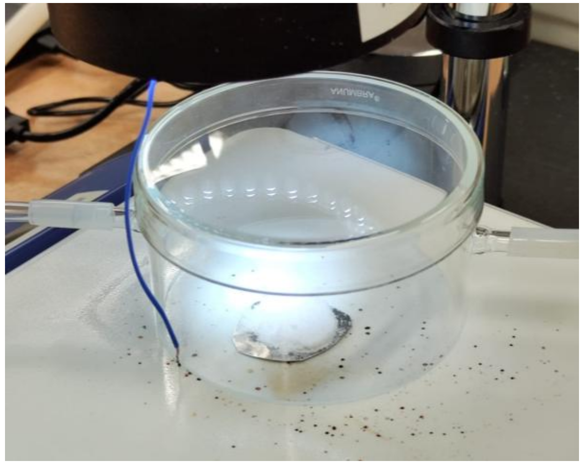
Total porosity of the rotomolded samples.

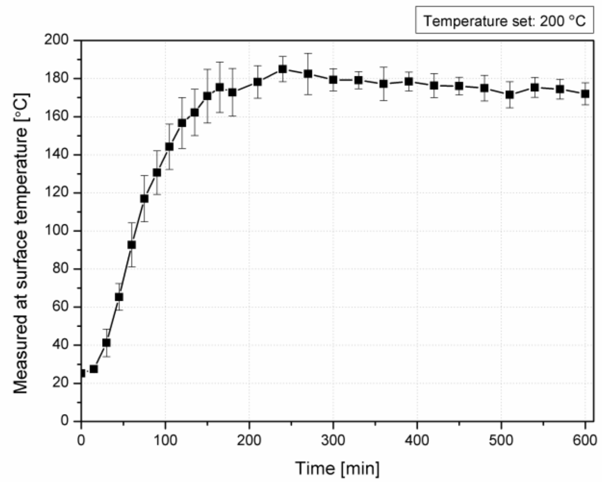
Porosity distribution in samples; 3D tomography images.



Case study: Using nutshells as active fillers

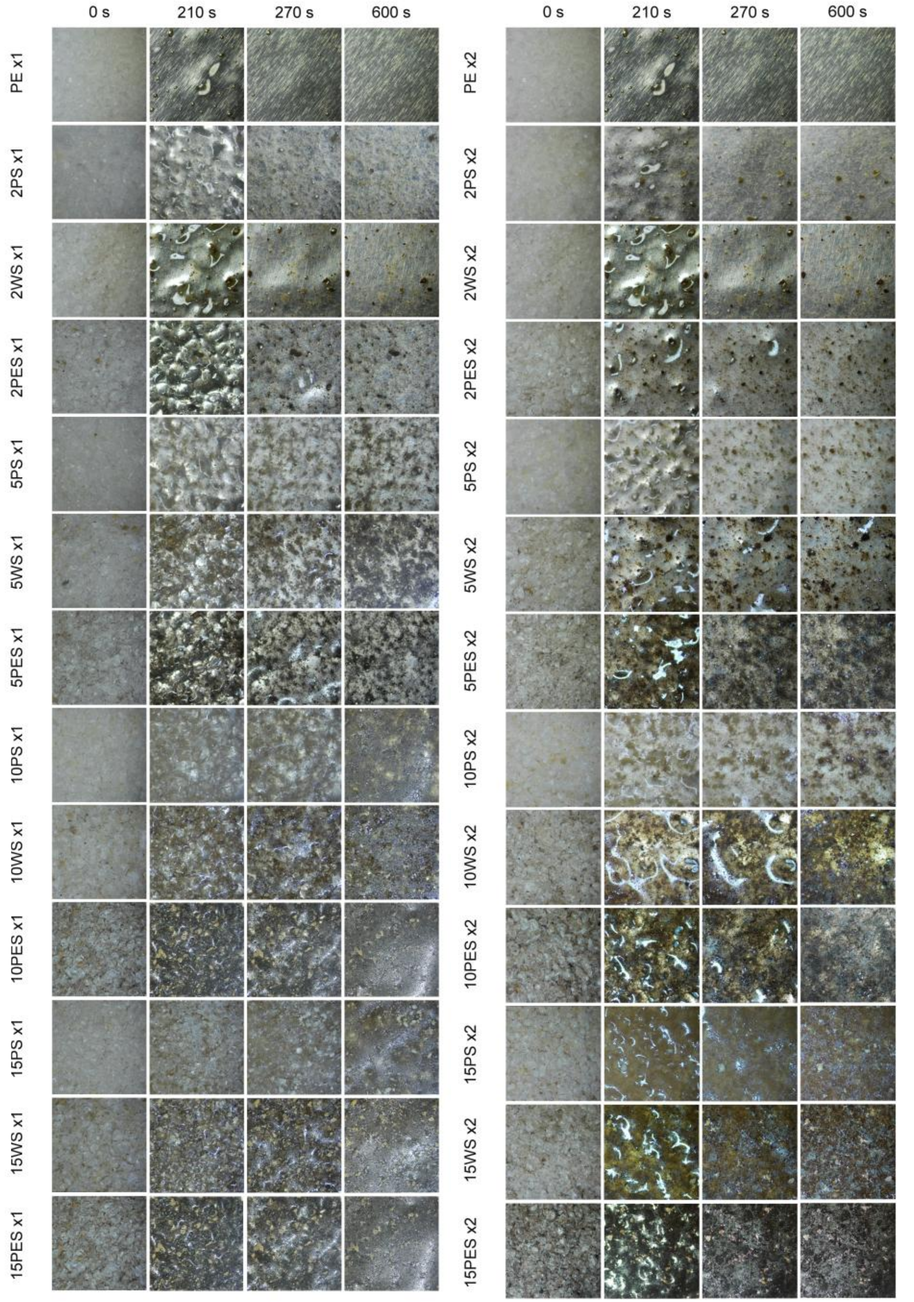
Static sintering analysis



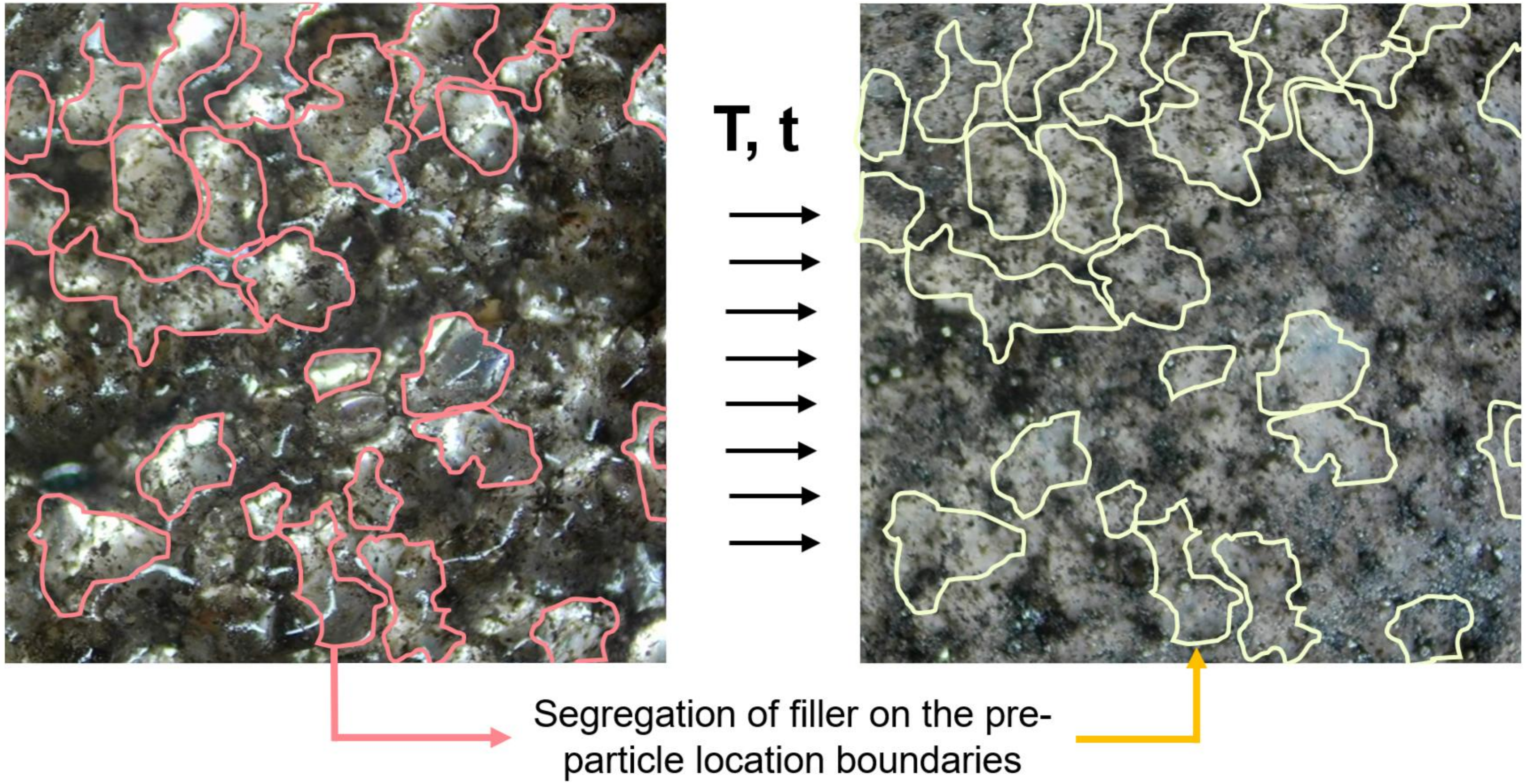


The course of temperature change of the heating table surface as a function of time.

Video of the static sintering-to-melting process of 5 wt% PES x1/x2.



Case study: Using nutshells as active fillers



Case study: Using nutshells as active fillers

Mechanical properties

Static tensile test

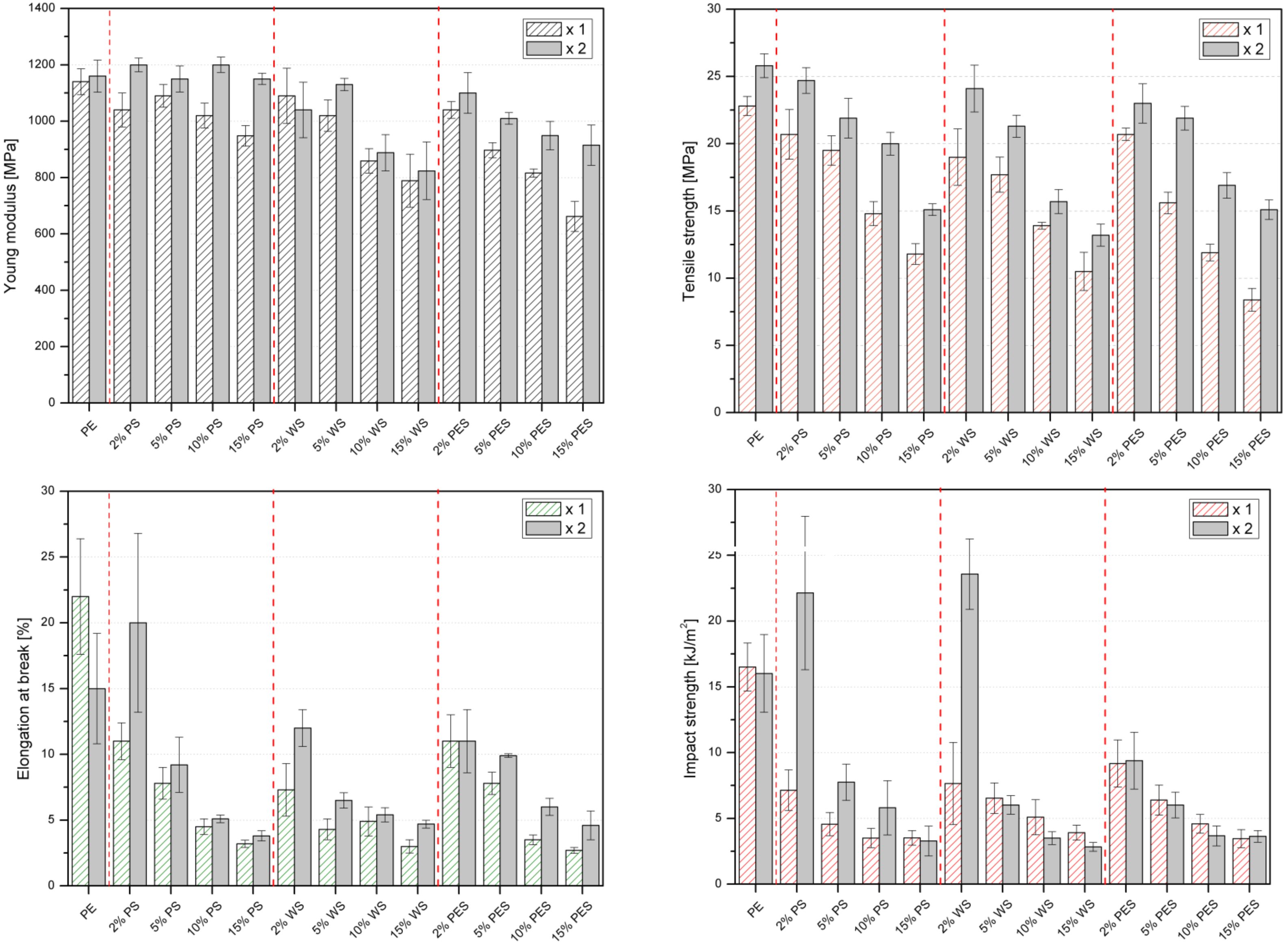
Apparatus: **Zwick/Roell Z020**

Standard: **ISO 527**
Cross-head speed: **10 mm/min**

Dynstat impact strength

Apparatus: **Dys-e 8421**

Standard: **DIN 53435**
Hammer: **0.98 J**



Mechanical properties of rotomolded PE and its composites.

Case study: Using nutshells as active fillers

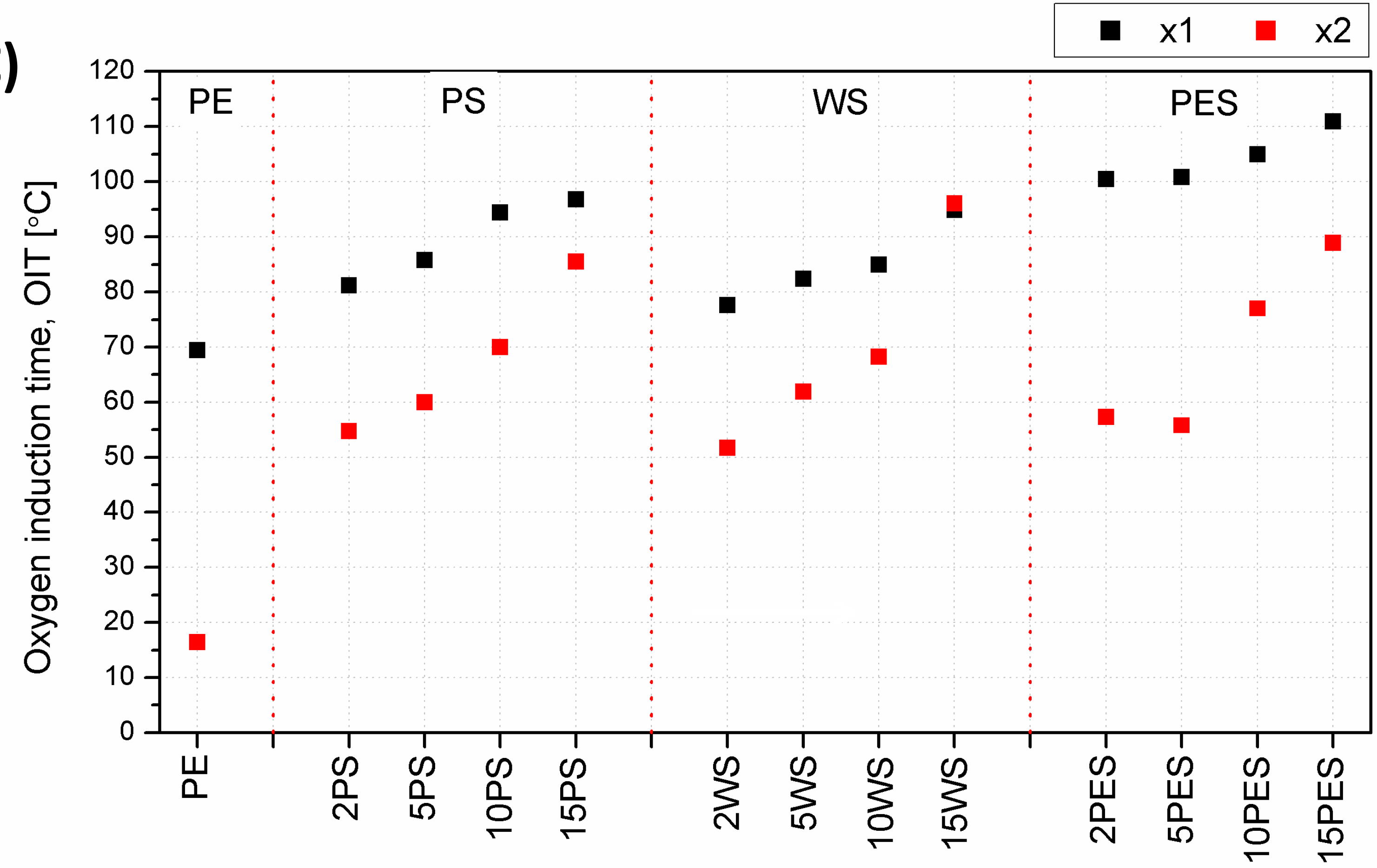
Differential scanning calorimetry (DSC)

Oxygen Induction Time (OIT)

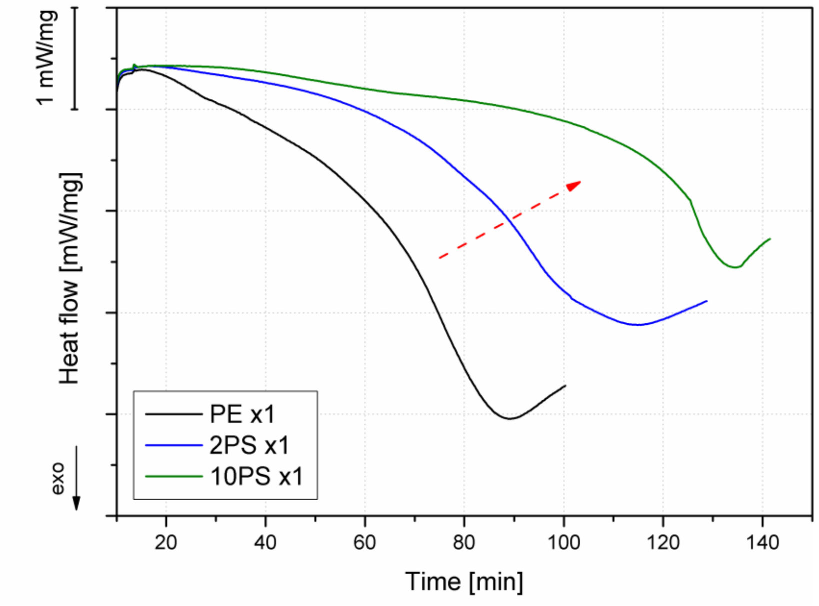
Apparatus: **Netzsch 204 F1 Phoenix**

Standard: **EN 728**

Cooling/heating rate: **10 °C/min**
Temperature range: **20 → 190 °C;**
5 min isothermal phase; nitrogen→oxygene



Averaged OIT values for HDPE and rotationally molded composite samples.



Exemplary DSC thermograms taken during OIT measurement for selected PS-filled composites.

Case study: Using nutshells as active fillers

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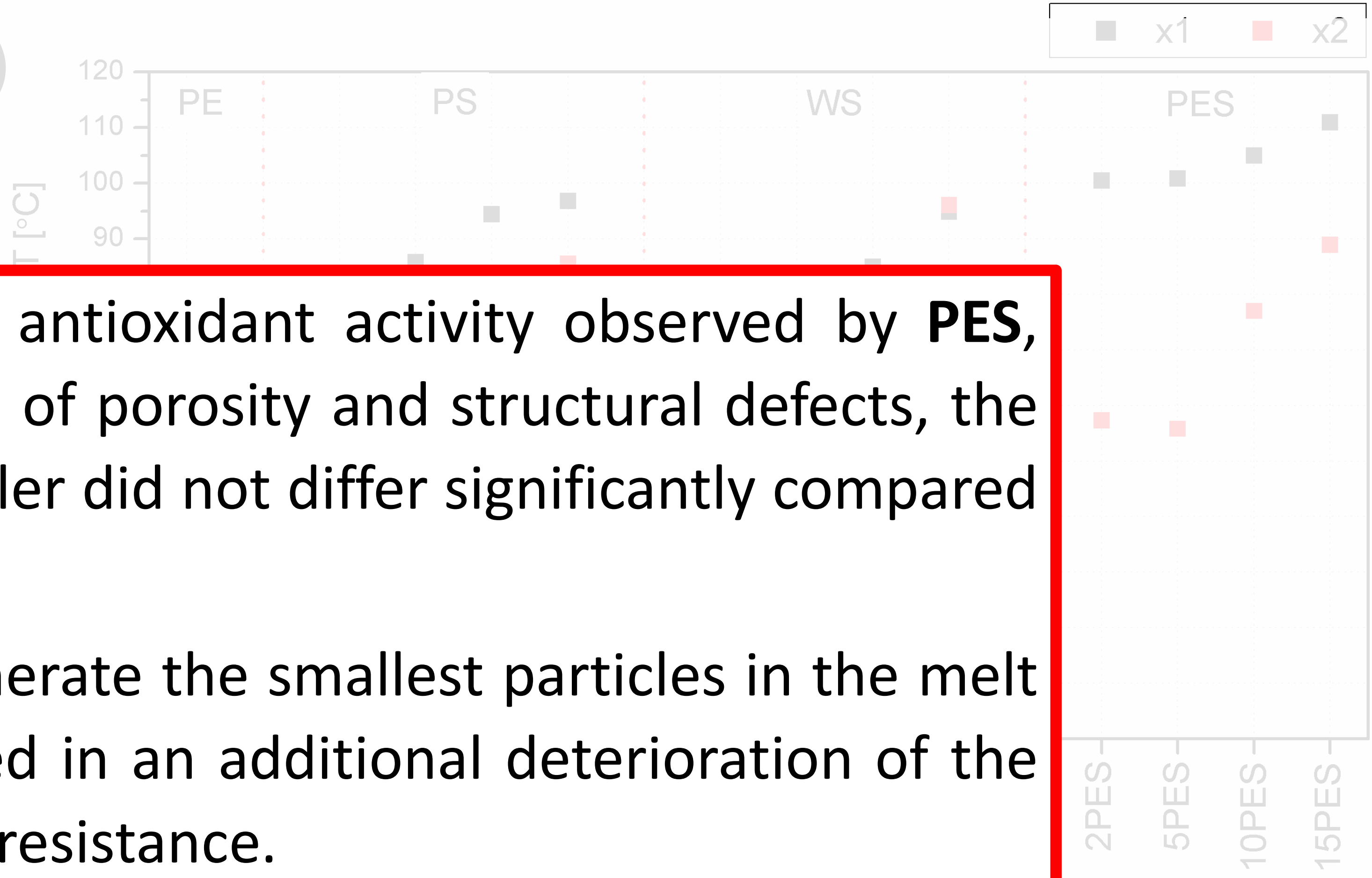

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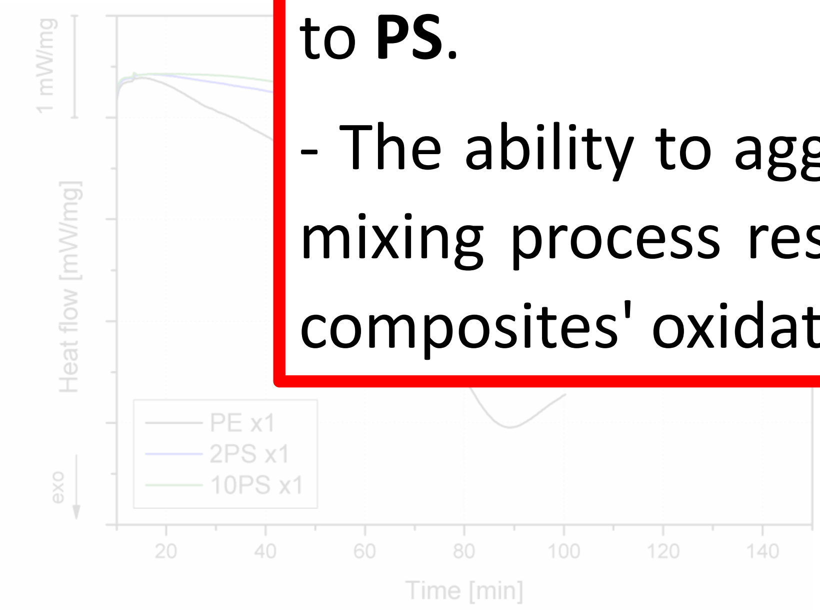


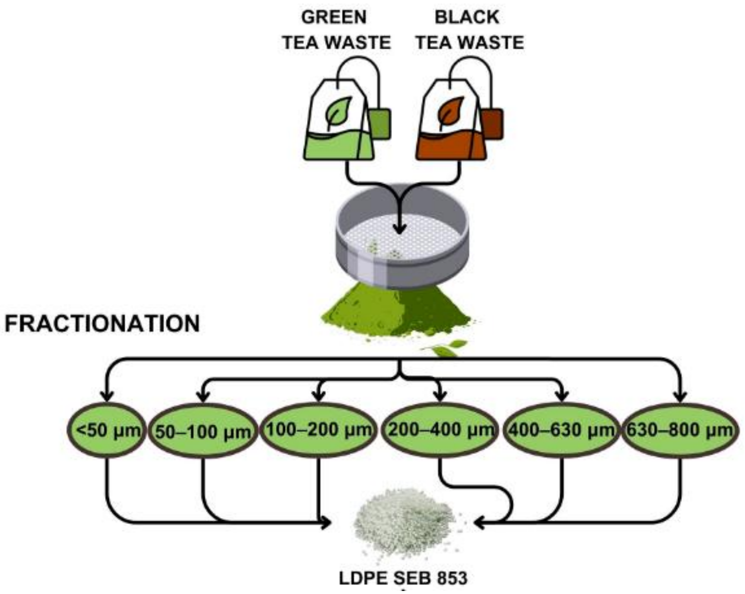
Exemplary DSC thermograms taken during OIT measurement for selected PS-filled composites.

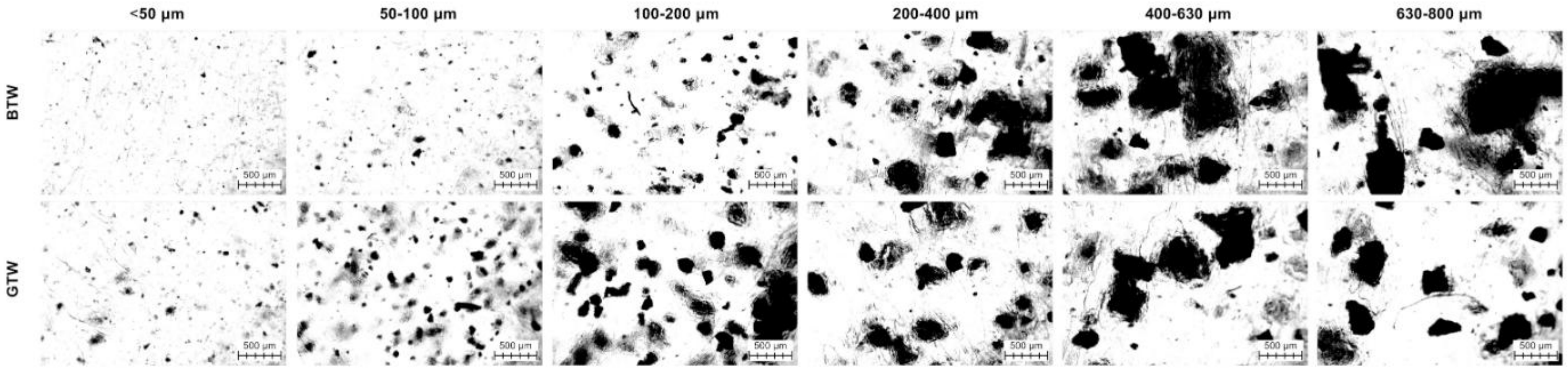
- Despite the highest antioxidant activity observed by **PES**, due to the large share of porosity and structural defects, the effectiveness of this filler did not differ significantly compared to **PS**.

- The ability to agglomerate the smallest particles in the melt mixing process resulted in an additional deterioration of the composites' oxidation resistance.

Filler fraction impact on stabilizing efficiency

On the balance between dispersion, processability, and stabilizing efficiency





Filler fraction impact on stabilizing efficiency

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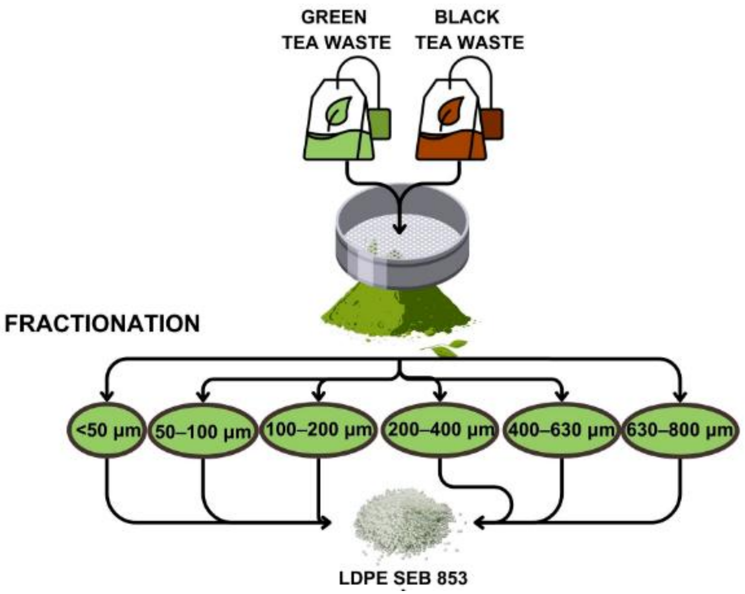
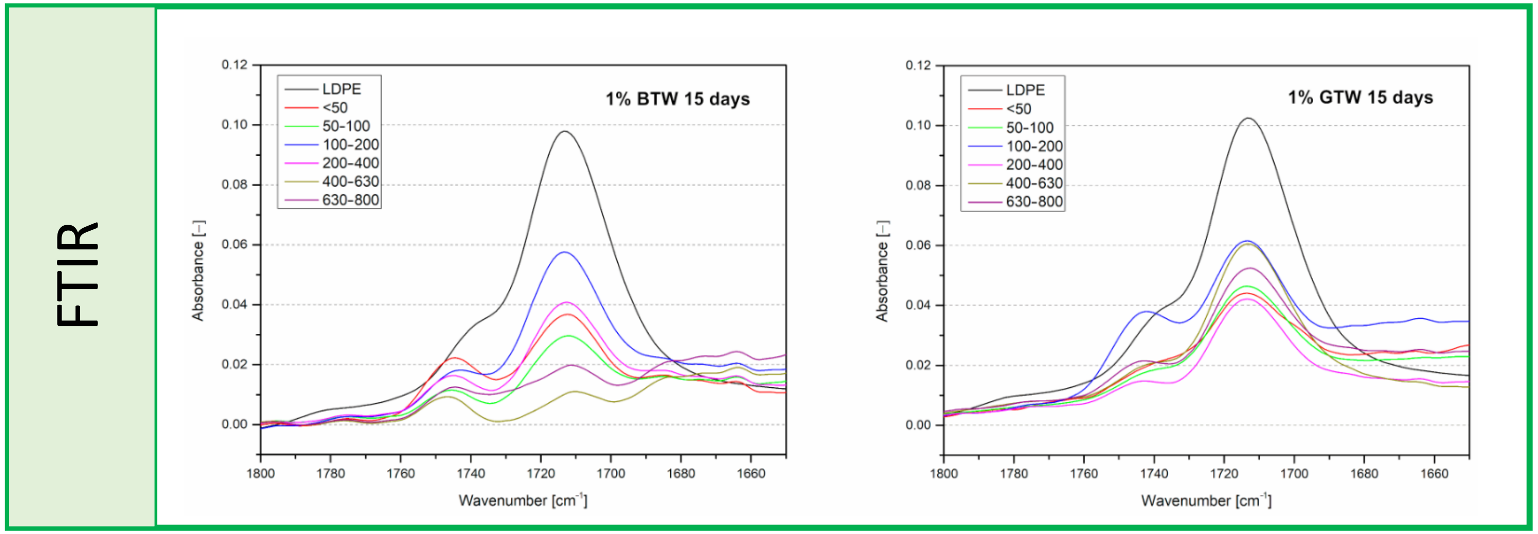
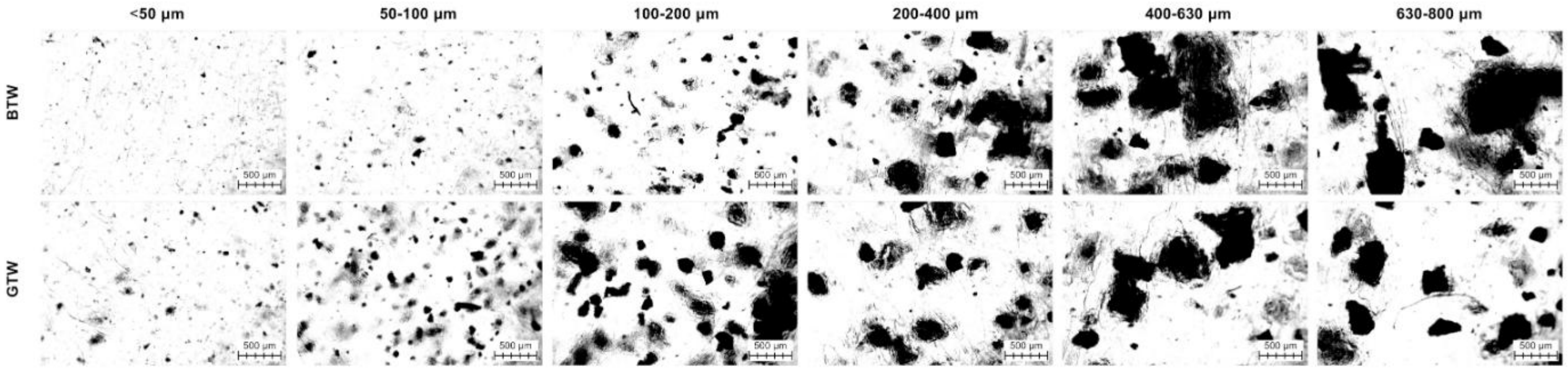
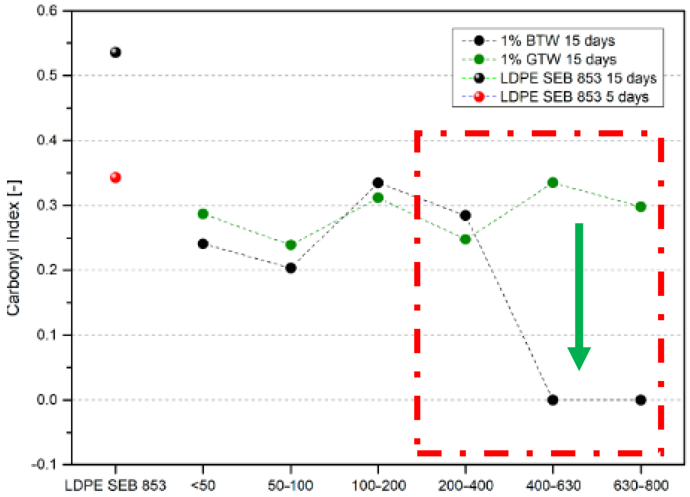




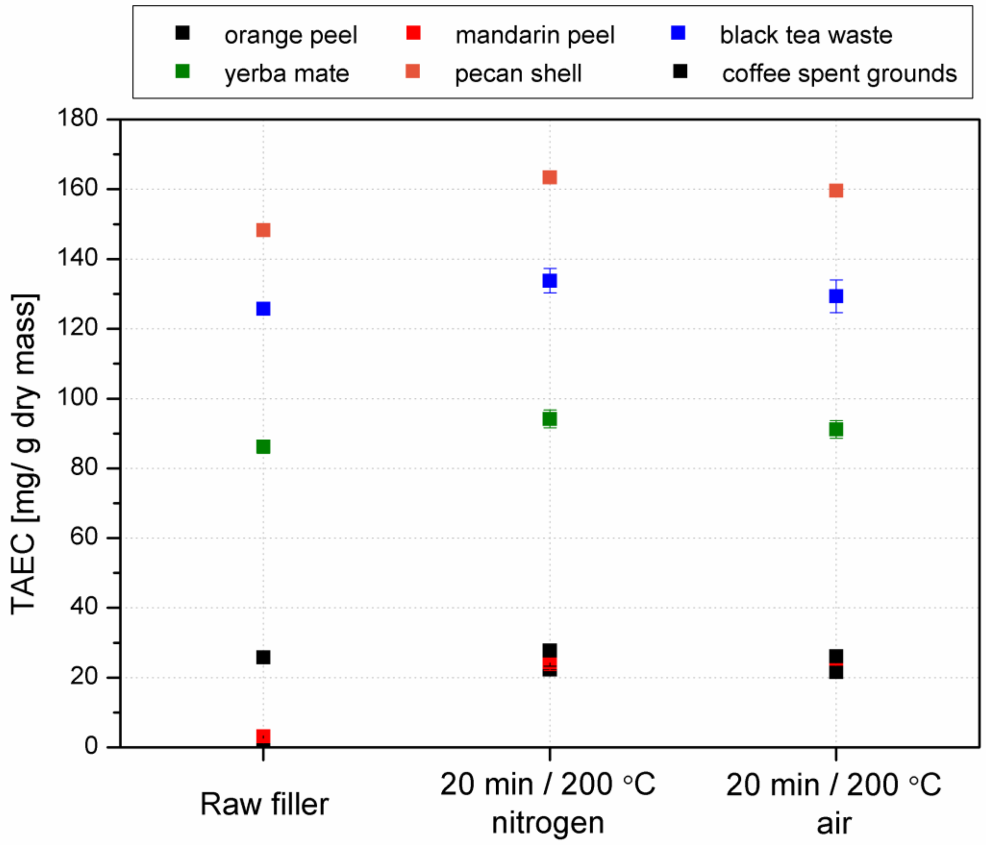




Stability of active compounds

Does the effectiveness of active compounds contained in the filler decrease with exposure to temperature?

Exposure to 200 °C for 20 min in an air and nitrogen atmosphere



Trolox equivalent antioxidant capacity values determined for fillers before and after 20 min exposure to 200 °C in inert and oxidative atmosphere.

Stability of active compounds

Does the effectiveness of active compounds contained in the filler decrease with exposure to temperature?

Coffee spent grounds

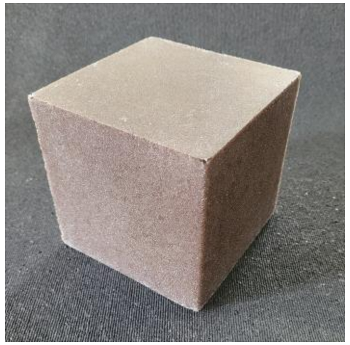
Beech wood

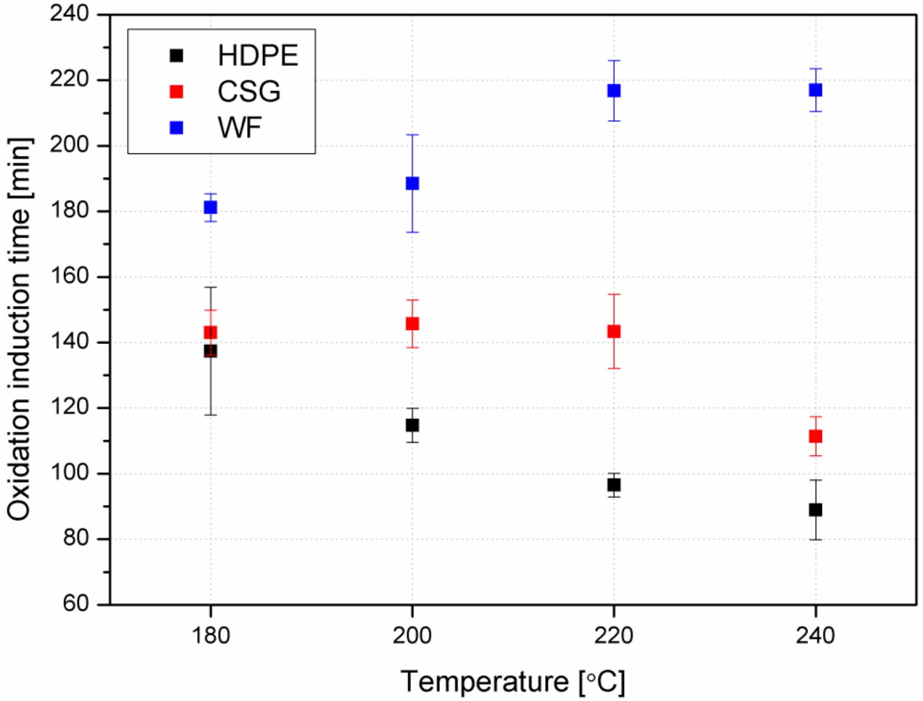
Twin-screw extrusion

180, 200, 220, 240 °C

+ HDPE

Rotational molding





OIT values determined for samples from RM-composite products, melt mixed at various temperatures.

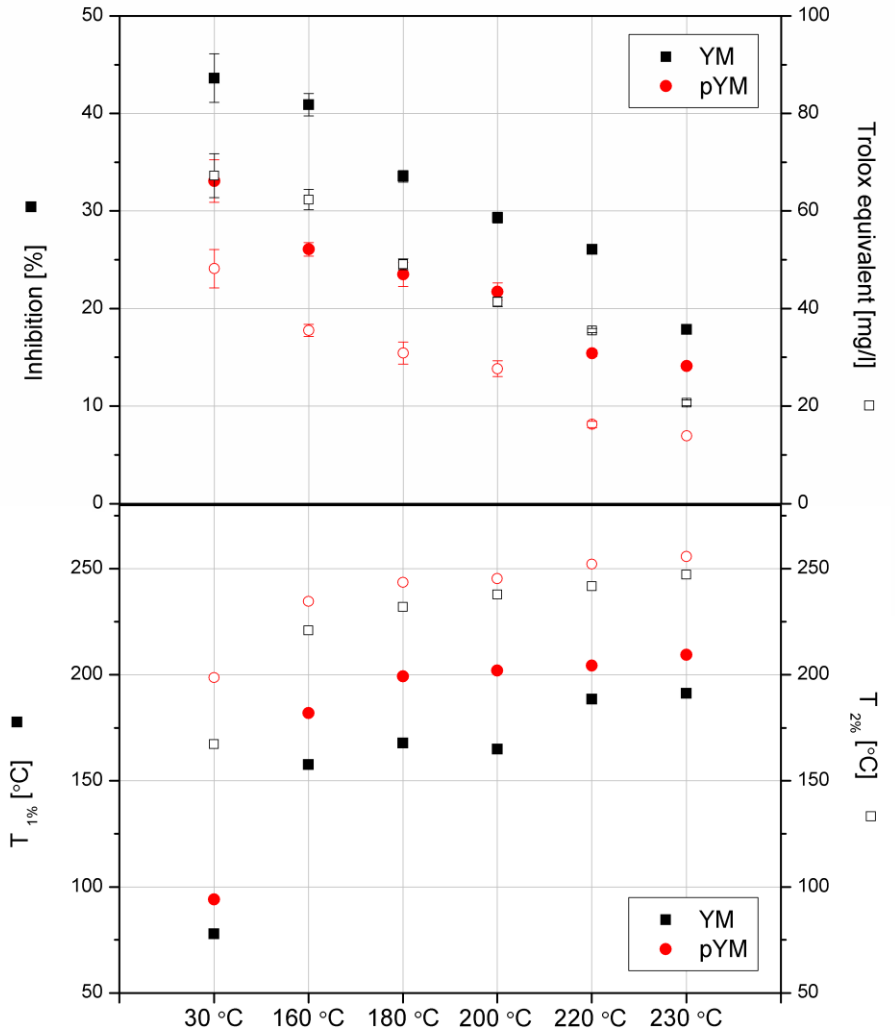
Conclusions

- Introducing plant-waste fillers rich in antioxidants into the polymer matrix allows for development of a new generation of self-stabilizing composites.
- As a result of optimization of the preparatory and forming processes, it is possible to produce composites with favorable structure properties and increased oxidation resistance.
- Thermal degradation of the lignocellulosic core of the plant-derived filler is not synonymous with limiting its antioxidant activity on the polymeric matrix.

Perspectives and actual work

- Current research is focused on controlled thermal decomposition of the various plant-derived fillers, in procedures that ensure the retention of antioxidant activity.
- Attempts to describe the effect of releasing bound polyphenols in thermally induced processes.

Thermomechanical biomass conversion process.



Relationship between biomass processing temperature and change in thermal stability and antioxidant activity.

Perspectives and actual work

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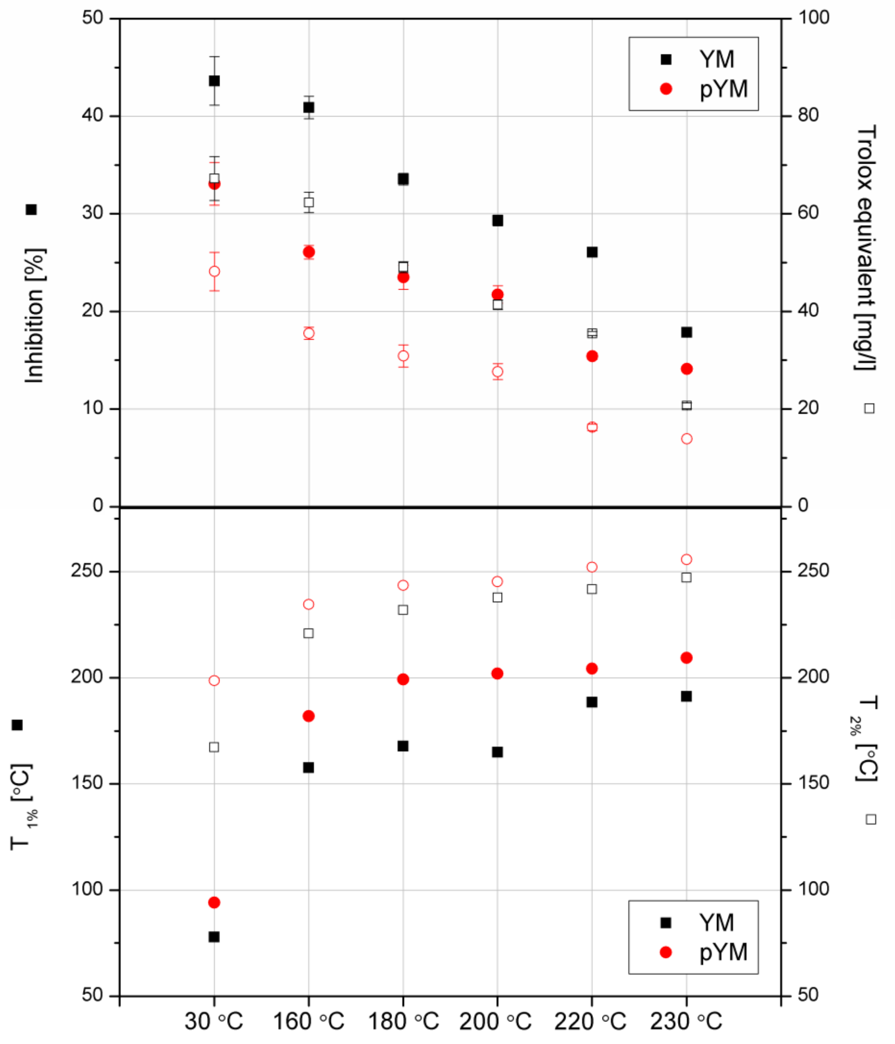
In pursuit of degradation - seeking beneficial effects of thermal and thermomechanical modification of plant-based materials used in polymeric materials

OPUS 2024/53/B/ST8/02082

Implementation period : **03.03.2025- 02.03.2029**

Principal Investigator: dr inż. Aleksander Hejna

NATIONAL SCIENCE CENTRE POLAND



Relationship between biomass processing temperature and change in thermal stability and antioxidant activity.

Research on the influence of the polymer composites processing conditions on the stabilizing effect of functional plant-derived fillers

SONATA-17 2021/43/D/ST8/01491

Principal Investigator: dr hab. inż. Mateusz Barczewski, prof. PP

Implementation period : **11.07.2022 – 10.07.2025**

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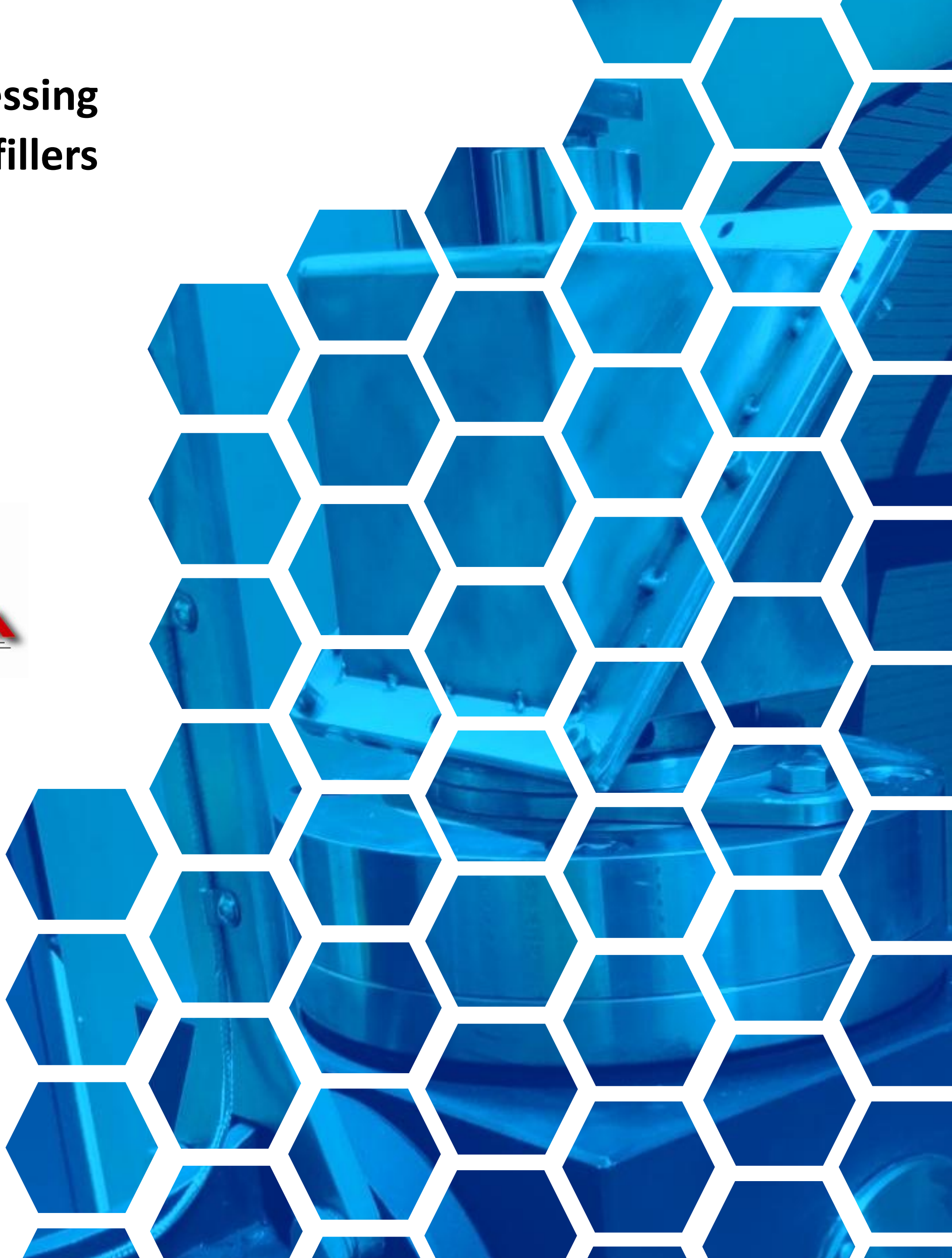

Thank you!