

Synthesis process of polylactide by reactive extrusion



Julien CAYUELA

Technical conference, Ghislenghien
25/05/2011



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

 **MateriaNova**
MATERIALS R&D CENTRE

Synthesis process of polylactide by reactive extrusion



TOTAL

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

Agenda

- 1- The main chemical reactions of the process**
- 2- The aimed characteristics for the PLA**
- 3- The synthesis process by reactive extrusion**

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

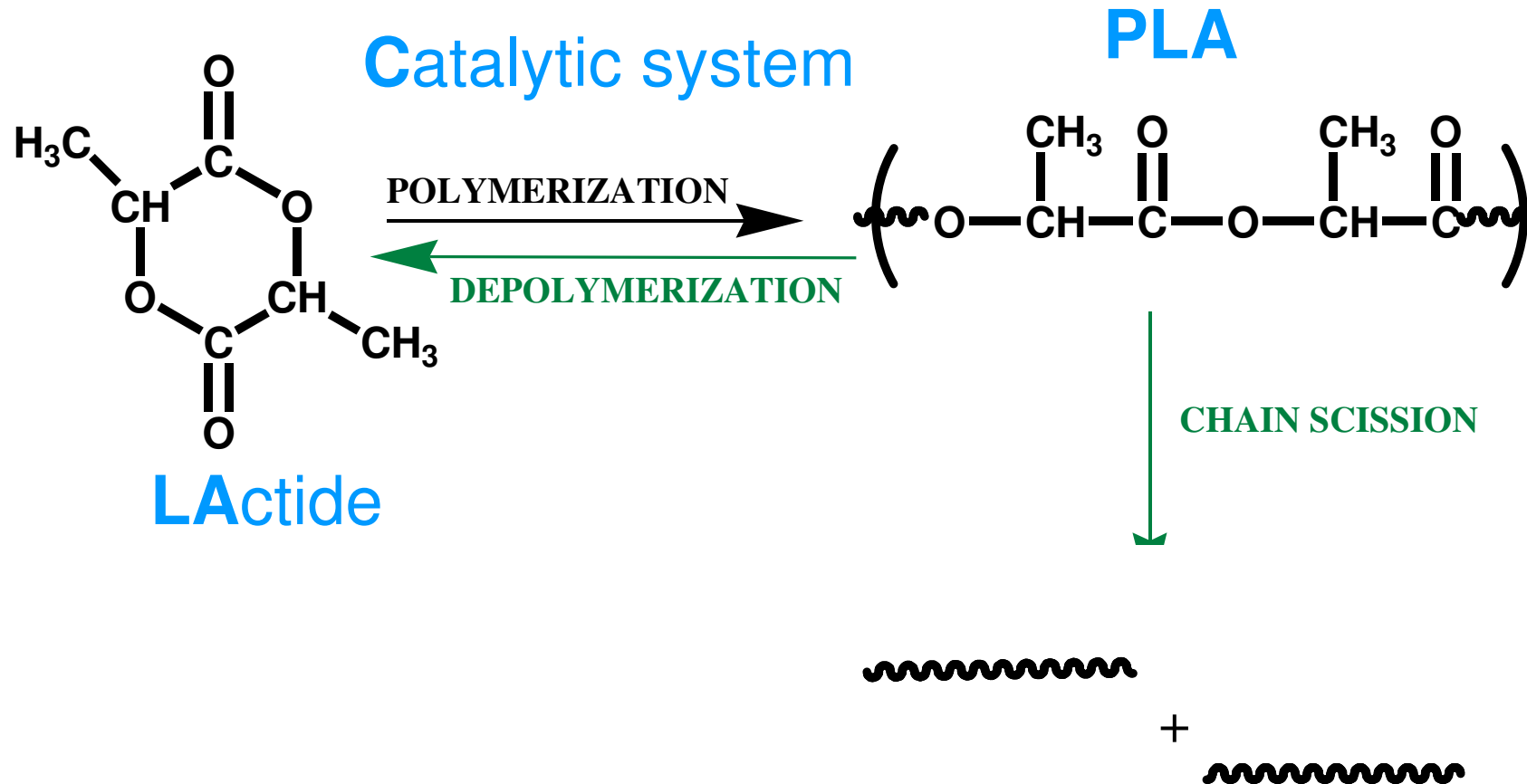
FUTURE MATERIALS MADE BY TODAY'S PEOPLE

 **MateriaNova**
MATERIALS R&D CENTRE

Chemical reactions
of the process

Aimed characteristics
for the PLA

Synthesis process by
reactive extrusion



©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	--	---

		TARGETS
1	Molecular weight (equiv PS)	<p>Mp=135 000 (g/mol) Mp=225 000 (g/mol)</p>
2	Residual LA level (% mass)	< 0,3%
3	Color (YI and b) (Cristallized pellets)	YI<11 and b<4
4	Transparency (amorphous pellets)	OK
5	Stability during the processing Brabender mixer, T=225°C, t=30 min	↘ Mp < -15%
		↗ LA < +0,3%
		↗ b < +3

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova




Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	--	---

	Measurements	Time to obtain the results
Molecular weight	GPC	2 h
Residual LA level	¹ H NMR	1 h 20 min
	TGA	20 min
Color (YI and b) (Cristallized pellets)	Densitometer	1 min
Transparency (amorphous pellets)		1 sec

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



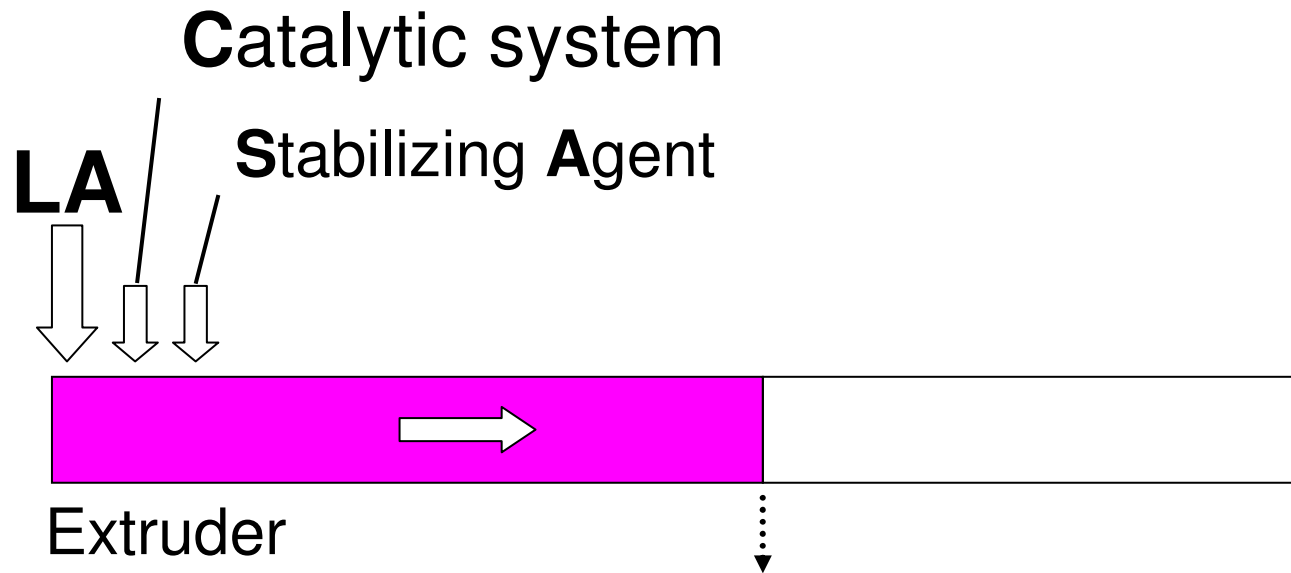
Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	-----------------------------------	--

→ Polymerization



Complete polymerization

Molecular weight : OK

LA = 5%

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

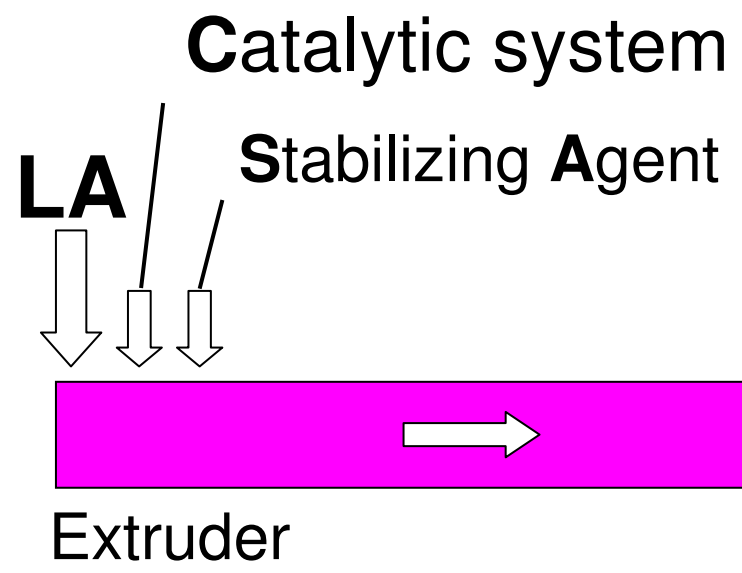
FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	--	---

→ **Polymerization**

Methodology



Lab scale: composition of the catalytic system

- A molecular weight control
- A fast kinetic
- A compatible kinetic

Complete polymerization
Molecular weight : OK
LA = 5%

©MateriaNova – All rights reserved for all countries. Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

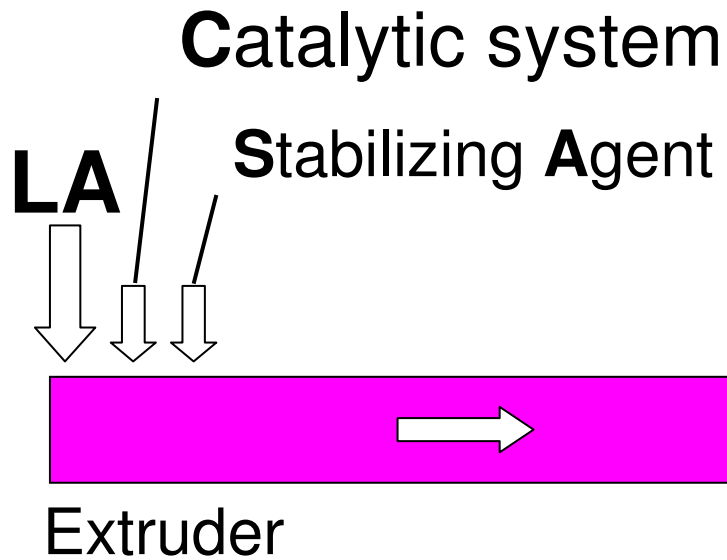
FUTURE MATERIALS MADE BY TODAY'S PEOPLE



Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	--	---

→ **Polymerization**

Methodology



Extruder:

- Introduction of the reactants
- Conditions to have the wished residence time

Complete polymerization

Molecular weight : OK

LA = 5%

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



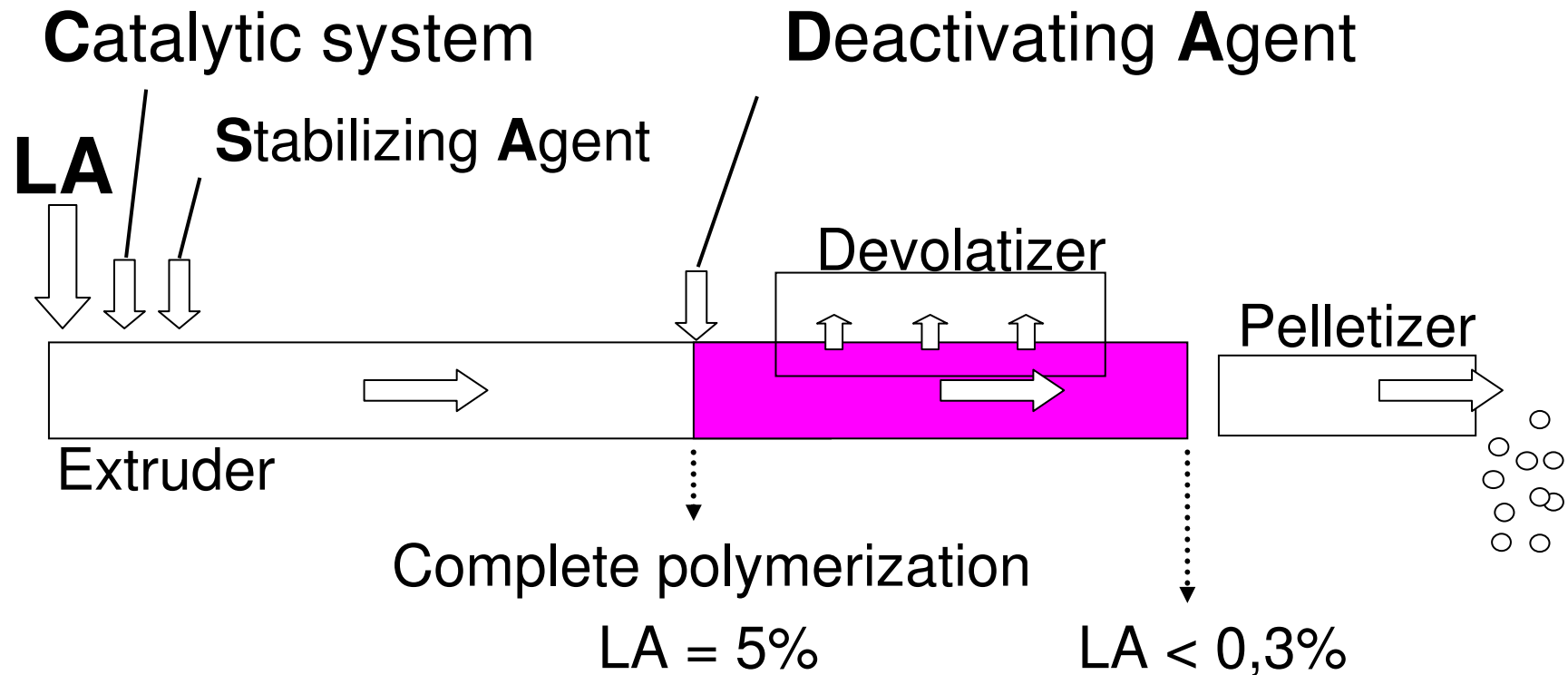
Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE



Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	--	---

→ Deactivation and devolatilization



©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



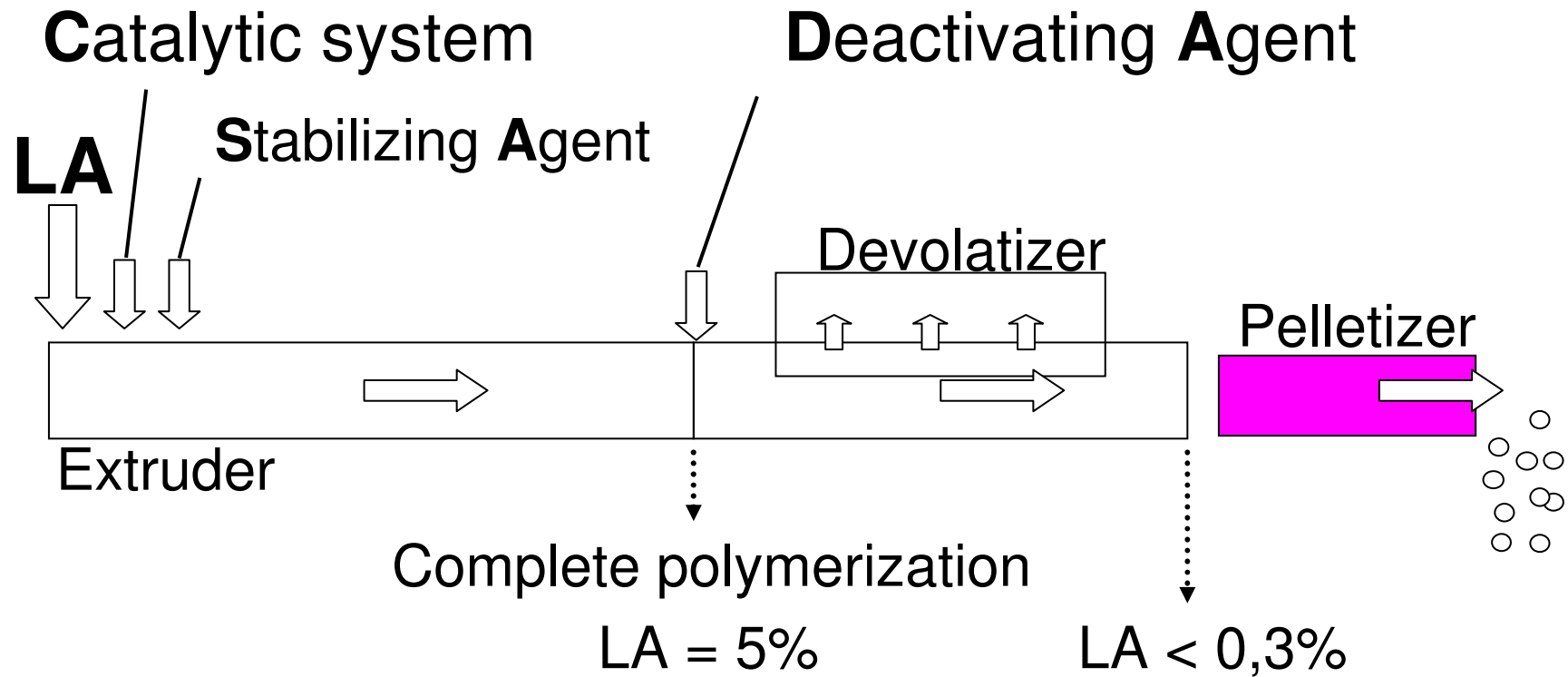
Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Chemical reactions of the process	Aimed characteristics for the PLA	Synthesis process by reactive extrusion
-----------------------------------	--	---

→ Pelletizing



©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova

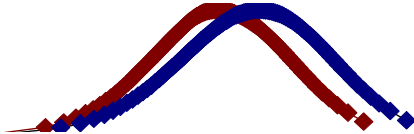


Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Conclusion

		TARGETS
1	Molecular weight	
2	Residual LA level	< 0,3%
3	Color (YI and b)	YI<11 and b<4
4	Transparency	OK
5	Stability during the processing Brabender mixer, T=225 °C, t=30 min	↘ M _p < -15%
		↗ LA < +0,3%
		↗ b < +3

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova

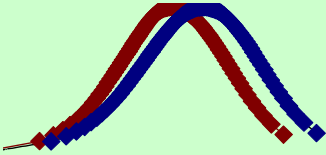


Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Conclusion

		TARGETS	
1	Molecular weight		
2	Residual LA level	< 0,3%	> 1,5%
3	Color (YI and b)	YI < 11 and b < 4	↑ Without Deactivating Agent
4	Transparency	OK	
5	Stability during the processing Brabender mixer, T=225 °C, t=30 min	↘ Mp < -15%	↘ Mp = -35%
		↗ LA < +0,3%	↗ LA = +3%
		↗ b < +3	↗ b = +9

©MateriaNova – All rights reserved for all countries
Cannot be disclosed, used or reproduced without prior written specific authorization of MateriaNova



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

MateriaNova
MATERIALS R&D CENTRE

Acknowledgement

Olivier Talon : R&D Scientist,
Coordinator Bioplastics Unit

Ibrahim Barakat, Benjamine Belloncle : R&D Scientists

Jonathan Wolfs : Laboratory Assistant

Philippe Lewillion : Laboratory Assistant,
Person in charge of infrastructure

Thanks for your attention!



Le Fonds Européen de Développement Régional et la Région Wallonne investissent dans votre avenir

FUTURE MATERIALS MADE BY TODAY'S PEOPLE

 **MateriaNova**
MATERIALS R&D CENTRE