

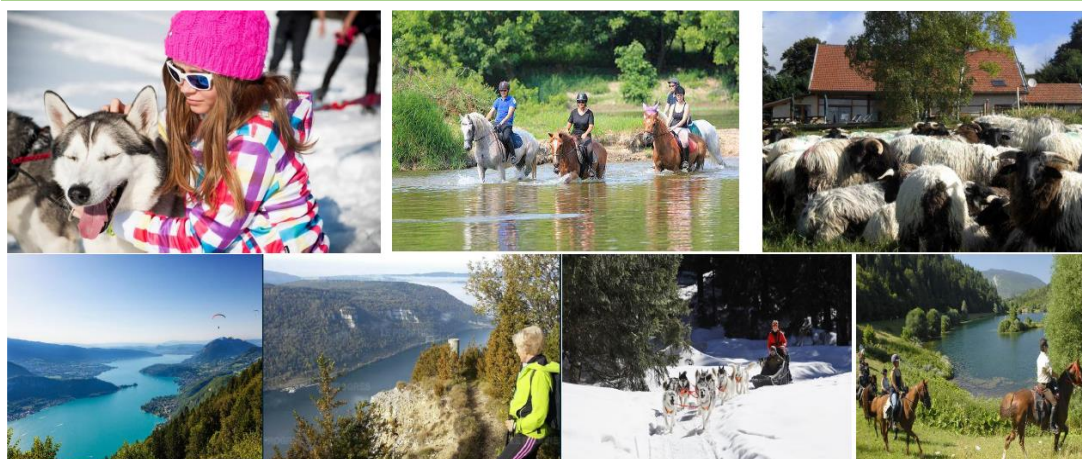
International Conference of the **YOUNG RHEOLOGISTS DAYS**



July 06-09, 2021 **Giron, FRANCE**



Technical Program



THE EUROPEAN SOCIETY
OF RHEOLOGY
Everything for the Rheologist



Ingenierie et Matériaux Polymères

INSTITUT NATIONAL
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Welcoming Words by the YRD 2021 Chairman



Dear ladies and gentlemen,
Dear colleagues and friends,
Dear YRD attendees,

It is a great pleasure for us to welcome you from all over the world at the international conference of Young Rheologists days 2021 to be held from 06-09 July 2021. Initially scheduled in June 16-19, 2020 and postponed to those new dates owing to the COVID-19 pandemic situation, I've been delighted to organize this conference in hybrid form consisting of both live performances and remote presentations. I'm so glad to be able to enjoy this conference with you here after this pandemic crisis.



For the first time, we aim at giving a more international dimension to this meeting, thanks to the privileged location of Giron (Jura) and the great support of the European Society of rheology (ESR) and the French Group of Rheology (GFR).

The main focus of this conference is to establish a discussion forum for the community of engineers and scientists in the rheology field. We aim at giving, for the first time, a more international dimension to our meeting. YRD conference will gather scientists and engineers who are working in the field of rheology. Indeed, rheologists from universities, research centers or industries will be able to report on the most recent advances in the field.

To date, we have received more than 80 abstracts from *France, Italy, Poland, Morocco, Romania, China, France, Israel, Portugal, Spain, Colombia, Tunisia, Canada, Spain, Germany, UK, Greece, USA, Switzerland*. We have selected after review 60 presentations from PhD, post-doctoral fellows and engineers. Moreover, the program includes 5 renowned guest speakers for Plenary Lectures. Six Keynotes lectures for round tables will be held by eminent researchers from both the academic and industrial world. The attractiveness of these lectures has led to a great interest in this conference; in the end, more than 100 participants are expected making this event a great success. The objective is to give to all applicants the opportunity to present their work and exchange with rheologists from a wide range of backgrounds and promote discussions inside and out of conferences.

We hope that you will enjoy the scientific program and the charms of Giron and Jura region. At almost 1100 m altitude, the new YRD 2021 will give you a special chance to enjoy the splendid Jura mountain chain, making it the first tourist destination of Auvergne-Rhone Alpes. Giron offers a wide variety of unspoiled natural landscapes alternating with rivers, magnificent lakes, alpine pastures and spruce forests. Nonetheless, Jura region is also cities and villages with a rich heritage, renowned vineyards offering great varieties of wine, delicious cheeses, diverse crafts... These numerous attractions make this region a popular destination for lovers of nature, snow, exploring and fine food and gastronomy. Besides, a varied social program will accompany the conference: hiking, private Jacuzzi, visits of Lac Genin, Lac Nantua, Formagerie, INSA's Rheology platform-Oyonnax...

On behalf of the Organizing committee, we would like to thank the YRD 2021 attendees, presenters and accompanying persons, involved institutions and the sponsors.

We wish you a memorable YRD 2021 and Stay in Giron, France.

Sincerely,
Khalid LAMNAWAR, General Chair

Local Organizing Committee

Conference Chair: Khalid LAMNAWAR

Accounting: Muriel Personne and Laure Corrigan (INSAVALOR)

Committee Members:

Melinda Desse, Mohamed Yousfi, Matthieu Zinet.



GFR

The French Group of rheology GFR was founded in 1964 and represents over 250 rheologists from more than 20 Universities, and 30 professionals from our major industries, actively engaged with a common interest in rheology. The GFR is a member of the International Committee of Rheology (ICR) and the European Society of Rheology (ESR). The group organizes a “National Congress” each year, two annual workshops linking academia and industry and the “Young Rheologists Days”, mainly devoted to Phd students, Post-doctoral fellows and young researchers, every two years.

<http://legfr.fr/site/>

European Society of Rheology (ESR)

The European Society of Rheology is open for everyone interested in rheology in all countries of Europe. Rheology is defined as the science of the deformation and flow of matter which means that rheology in some form enters almost every study of material properties. The ESR involves rheologists engaged in both industrial and academic research and development and is therefore a common meeting ground for engineers, physicists, chemists and biologists with a common interest in rheology.

<https://rheology-esr.org/>

Sponsors



Exhibitors



The platform for
formulation development

NETZSCH



General Information

Late Registration

The registration desk will be located in the main hall of the Giron conference center/hotel. It will be in service:

Monday, July 05	From 02:00 to 07:00 pm
Tuesday, July 26	From 08:00 am to 10:00 am

Instructions for Speakers/ Presentation Guideline

Presentation Time

1. Oral Presentation Time: 20 min (Including 5 min Q&A, discussion).
2. Keynote Presentation Time: 25 min (Including 5 min Q&A, discussion and changeover of the session).
3. Keynote Presentation and round table Time: 30 min (Including 10 min Q&A, discussion and changeover of the session).
3. Plenary Presentation Time: 45 min (Including 5 min Q&A).

The time allocated for your presentation is including the time for discussion. Therefore, we strongly recommend that you do not exceed the time limit in order to give the other speakers enough time for their talk, and give the audience the opportunity to ask questions and/or make their own statements.

Speakers are requested to respect the schedule and present themselves to their session chair 15 minutes before the beginning of the session.

For virtual presentation: The remote talks will be presented directly. The attendees have to be connected to the link below , follow the other slots and present their work.

Link :

<https://insa-lyon-fr.zoom.us/j/92754784921?pwd=N3JoNVJrMmtEVGF6UC9jOGlScKJrUT09>

ID de réunion : 927 5478 4921

Code secret : 678779

Submitting the Presentation File

The lecture rooms will be equipped with projectors, computers and laser pointers. The face to face **speakers are invited to upload their presentations at desk the day before their presentation. Otherwise,** please bring your presentation on a memory stick and hand it in good time before your session starts, **4 hours prior at least.**

Presentation Equipment

1. Presentation files can be only accepted as *.PDF, *.PPT, or *.PPTX (Powerpoint)
2. Each presentation room will be connected to the "Media Centre" with a laptop which is linked to LCD projector, laser pointer and microphone. If you require other audio-visual equipment, please contact conference secretariat (E-mail: [jjr2020 <jjr2020@sciencesconf.org>](mailto:jjr2020@sciencesconf.org)).
3. The modern and efficient computers for your presentation are provided with Microsoft® Windows® 7 or 8, OFFICE 2010, Adobe Acrobat 11. Please make sure to enclose all used fonts and media data to your Microsoft® PowerPoint® presentations
4. The standard set up does not allow for audio in your presentation.

If you use clips or attachments, make sure to create a presentation folder where these are included with your presentation. It is very important that you do not include uncompressed video files in your presentation.

We kindly ask all speakers to follow the mentioned information in order to guarantee a fluent proceeding during the whole conference.

If you have any questions please do not hesitate to contact the program coordinator at the secretary desk on-site or one of our hostesses at the registration desk.

For any specific questions regarding your presentation please contact us at jjr2020@sciencesconf.org with your reference of YRD 2021 presentation.

Abstracts and Conference Proceedings

All accepted abstracts are included in the digital Book of Abstracts in USB flash drive. They are also included in the technical program.

Exhibit

Exhibit of industrial equipments will be held in the conference center. Poster sessions will be held close to the exhibitors.

Coffee Breaks

The coffee breaks will be held in the Exhibit and poster hall of the conference center as indicated in the technical program.

Facilities (More details are given in YRD 2021 website)

- An attractive social program is also given in the website <https://jjr2020.sciencesconf.org/resource/page/id/4>
- How to get to Giron conference center: <https://jjr2020.sciencesconf.org/resource/page/id/3>

The attendees can be picked up from Geneva Airport, Bellegarde Sur Vlaserine TGV Station or (only 35 minutes from the conference center). Please send us an email to organize it by July, 5th and 09 July.

A shuttle from Lyon to Giron is also scheduled on July, 5th at 17h00 for attendees from Lyon. They have to take a tramway T1 from Lyon Part-Dieu and join the shuttle bus the "INSA-Einstein".

https://www.insa-lyon.fr/sites/www.insa-lyon.fr/files/planinsa_17092019.pdf

Conference center-hotel

The Young Rheologists Days 2021 is organized at the splendid “Relais Nordique” (<http://www.le-relais-nordique.com/actualites-webcams.htm>). At almost 1100 m altitude, we hope to have a friendly conference enticing our young researchers to join our society.



JJR 2020 will give you a special chance to enjoy Ain/Jura Region and its splendid mountains, making it the first tourist destination of Auvergne-Rhone Alpes. Giron offers a wide variety of unspoiled natural landscapes alternating with rivers, magnificent lakes, alpine pastures and spruce forests. Nonetheless, Ain region is also cities and villages with a rich heritage, renowned vineyards offering great varieties of wine, delicious cheeses, diverse crafts...



Banquet

The banquet will be organized at the “ Belle Rive restaurant” around **the gorgeous Lac Nantua**. (an authentic and historical place in Nantua’s gastronomy near the famous Lac of Nantua) (<https://www.bellerive-nantua.com/fr/restaurant.html>). You can still book your ticket at the registration desk.



Poster Guideline

The poster title, author(s)'s name(s) and affiliation(s) should be placed at the top of the poster. The organization committee will send to you the logo of YRD 2021 to be printed on the poster. You can download it on the YRD Website. The recommended printed size for title is 2.5 cm (1 IN) in height. All illustrations, charts and pictures should be visible from a distance of 1.5 m.

Poster Size

The poster size should be in DIN A 0 format, 85 cm (~ 34 IN) in width and 120 cm (~ 48 IN) in height.

Poster Area

There is a designated location for poster presentations. Each display board will be identified by a poster number assigned to each poster according to the conference program. Poster presenters are responsible for putting up and removing their posters themselves. Equipment to fix the posters on the boards will be provided at the poster area.

Presentation Time

Posters will be available for viewing throughout the conference and must be taken down July, 9th. Poster presenters are asked to put up their poster after registration and be available for discussion at their posters during the formal poster sessions. All delegates are encouraged to attend. There will be no audio-visual equipment for Poster sessions.

Language

Please be advised that both oral & poster presentations must be conducted in English only.

Internet facility

Wireless internet will also be available for the delegates at the exhibition and posters hall. The wifi zone will be available during the conference.

Instructions to the session chairs

The chairs of each session are assigned in the timetable program.

Important:

- a) Please respect strictly the timing as indicated on the program.
- b) If a speaker is not present to give his/her lecture, please do not change the time order of the next presentation. Do strictly keep the schedule as printed on the program.



Invited lectures and keynotes

The conference will include 5 Guest speakers for Plenary Lectures from scientific and industrial pioneers in the field.

Prof. Jan Vermant (ETH Zurich, Switzerland), Weissenberg Award of the European Society of Rheology (EU)

<https://softmat.mat.ethz.ch/people.html>

Prof. Anke Lindner (ESPCI Paris), Maurice Couette GFR Prize, France.

<https://www.pmmh.espci.fr/?ERC-Consolidator-Grant-for-A>

Prof. Rudy Valette (MINES ParisTech)

<http://www.cemef.mines-paristech.fr/sections/cemef>

Prof. Associate-HDR Denis Roux (LRP-UGA)

<http://www.laboratoire-rheologie-et-procedes.fr/spip.php?article49>

Prof. Joao Maia (CWRU, Cleveland, USA),

<https://engineering.case.edu/profiles/jmm272>

Other **Keynotes lectures** are given by eminent rheologists and experts :

Dr. Della-Valle Guy, INRA, Nantes – France

Dr. Zhang Huagui, Fujian Normal University, China

Dr. Marchal Philippe, University of Lorraine - France

Dr. David Duval, Anton Paar

Dr. Divoux Thibaut, Laboratoire de Physique, ENS Lyon – France

Dr. Fraisse Frederic, NETZSCH GmbH, Dardilly – France

Dr. Bo Lu, Zhengzhou University (ZZU)-China

Conference Program at a glance

YRD 2021 GFR **Young Rheologists Days**
YRD 2021, Giron (France)



YRD 2021 Conference Program at glance

	Monday, July 05	Tuesday, July 06	Wednesday, July 07	Thursday, July 08	Friday, July 09	
Welcome at the hotel/conference center-shuttle organization for attendees		Registration- 8:00-10:00	Plenary Talk 3- 8:00-8:45	Plenary Talk 4- 8:00-8:45	Plenary Talk 5- 8:00-8:45	
		Opening-10h00-10:45	Oral presentations 08:45-10:05	Oral presentations 08:45-10:45	KN lectures and round tables 08:45-09:10 Oral presentations 09:10-9:50	
		Break- 10:45-11:10	Break- 10:05-10:30	Break- 10:45-11:10	Break- 10:00-10:25	
		Plenary Talk 1 -11:10-11:55	Oral presentations 10:30-12:30	Oral presentations 11:10-12:30	Oral presentations 10:25-11:25	
		Break- 11:55-12:15			KN lectures and round tables 11:25-11:55	
		Oral presentations 12:15-12:55				
		Lunch- 12:55-14:00	Lunch- 12:30-14:00	Lunch- 12:30-14:00	Lunch & picnic- 11:55-14:00	
		Exhibitors visits				
		Plenary Talk 2- 14:00-14:45	Oral presentations 14:00-16:00	Oral presentations 14:00-15:40	Oral presentations 14:00-16:20	
		Oral presentations 14:45-15:25	Break- 16:00-16:15	KN lectures and round tables 15:40-16:10	Closing Ceremony 16:20-16h30	
		Registration Giron Conference Center 50, Route de la Pesse 01130 GIRON 14:00 to 19:00	KN lectures and round tables 15:40-16:10	KN lectures and round tables 16:15-17:10	Break- 16:10-16:30	
	Welcome Reception conference Center 18:00 to 21:30	Poster Session 16:10-17h10	free time- 17:10-20:20 Visits & social program 2 Visit Lac Genin	free time- 16:30-19:00 Visits to compagnies Wine & cheese at INSA@oyonnax		
	Social program 1 & diner 17:10-23h00	Banquet Party (Lac Nantua, Restaurant Belle Rive) 20:20-23h00	Social program 2 & diner 19:15-23h00			

Registration	Black
Opening /Closing	Yellow
Plenary Lectures	Green
Oral presentations	Orange
KN/round tables	Purple
Poster Session	Light Green
Social Activities	Red
Break	Grey
Meeting	Blue
Lunch	Light Blue
free time	Pink

Program Schedule

Plenary lecture (PL):
45 min including 5min Q/A

Keynote presentation (KN): 25
min including 5min Q/A

Oral Presentaion (OP):
20min including 5min Q/A

Keynote presentation & round table
(KN-RT): 30 min including 10 min
Q/A

Monday, July 5, 2021

Local Organising Committee - (Only for YRD committee, Giron Conference Center- 50 route de la Pesse, 01130 Giron)

Registration- Giron Conference Center- 50 route de la Pesse, 01130 Giron

Welcoming reception : Hall of Le Relais Nordique (Giron)

Tuesday, July 6, 2021

08:00am-10:00 am

Registration

10:00am-10:45 am

Opening Ceremony

10:45am-11:10am

Break Coffee

11:10am-11:55am Plenary Lecture 1: Stability of thin liquid films (Jan Vermant, ETH Zurich, Switzerland)

11:55am-12:15 pm

Coffee Break

Room

Room 1 (Maxwell)

Chair Guy Della-Valle

12:15pm-12:55pm

OP 1: 12:15-12:35
Impact of bulk rheology of Kappa-Carrageenane on the oil/water interface, [Jaber Ahmad](#), [Benyahia Lazhar](#), [Marchal Philippe](#), [Roques-Carnes Thibault](#), [Hamieh Tayssir](#),
University of Lorraine, France
France

OP 2 (remothly):12: 35-12:55
Non-linear rheology characteristics of the gel point: large amplitude oscillatory shear behavior of chemically and physically crosslinked hydrogels, [Kogan David](#), [Gottlieb Moshe](#), Ben-Gurion University of the Negev, Beer-Sheva - Israel

12:55pm-02:00pm

Lunch Break

Exhibitors visits and discussions

Chair Rudy Valetta

2:00pm-2:45pm Plenary Lecture 2: Rheology as a critical Enabler of polymer processing: Extensional-driven mixing in extrusion and Multi-Layered Tubes, Pipes and Bottles Joao Mala (CWRU, Cleveland, USA)

02:45pm-03:25pm

OP 3: 2:45-3:05
Structure-rheology-properties relationships of biocomposites suspensions based on poly(lactic acid) and bioactive glass fillers [Xavier Lacambra](#), [Nora Dergham](#), [Jean-Marc Chenal](#), [Abderrahim Maazouz](#), [Khalid Lammawar](#)

OP 4 (remothly): 3:05-3:25
Miscibility, rheological and thermo-mechanical properties of compatible biopolymer blends: influence of process parameters and natural surfactants, [Danna Alessandra](#), [Arrigo Rossella](#), [Frache Alberto](#), Politecnico di Torino, Italy

03:25pm-03:40pm

Coffee Break

Room

Room 2 (Kelvin-Voigt)

03:40pm-04:10pm

KN-RT
Rheology and sustainable development
Della-Valle Guy, INRA, Nantes
- France

4:10pm-5:10pm

Poster Session

5:10pm-7:30pm

Social program 1 & diner (8:30 pm)

Wednesday, July 7, 2021

Room		Room 1	
Chair	Denis Roux		
08:00am-08:45am	Plenary Lecture 3: Morphological transitions of flexible fibers in viscous flows Lindner Anke (ESPCI, Paris – France)		
08:45am-09:25am	<p>OP 5: 8:45-9:05 Detection of damage localization in Polymer Networks by Space-resolved Multi-speckle Diffusing Wave Spectroscopy (MSDWS) <u>Ju Jianzhu</u>, Narita Tetsuharu, Creton Costantino, Espci, France</p>	<p>OP 6: 9:05-9:25 Multi-Micro/Nanolayer Films Based on Polyolefins: New Approaches from Eco-Design to Recycling <u>Masghouni Emma</u>, Cabrera Gerakline, Touil Ibtissam, Maazouz Abderrahim, Lamnawar Khalid, INSA Lyon, France</p>	
09:25am-10:05am	<p>OP 7 (remotely): 9:25-9:45 Injectability of thermosensitive chitosan hydrogels in terms of capillary rheometry <u>Ryl Anna</u>, Lodz University of Technology, Lodz - Poland</p>	<p>OP 8: 9:45-10:05 Studies on Rheology of Wood Polymer Composites Kamila Buziak Warsaw University of Technology, Poland</p>	
10:05am-10:30am	Coffee Break		

Exhibitors visits

Room		Room 1 (Maxwell)	
Chair	Thibaux Divoux		
10:30am-11:10am	<p>OP 9: 10:30-10:50 Rheology and Process Modeling for Polymeric Materials with Yield Stress and Wall Slipping, <u>Lewandowski Adrian</u>, Warsaw University of Technology, Warszawa - Poland</p>	<p>OP 10: 10:50-11:10 New Poloxamer 407 / PEG self-healing suspended hydrogel for 3D printing fluid materials <u>Colly Arthur</u>, Courrial Edwin-Joffrey, Marquette Christophe, University of Lyon 1 - France</p>	
11:10am-11:50am	<p>OP 11: 11:10-11:30 How to predict cell viability in Bioprinting <u>Lemaré Lucas</u>, Courrial Edwin-Joffrey, Petiot Emma, University of Lyon 1 - France</p>	<p>OP 12: 11:30-11:50 The role of solvent on the rheology and the supramolecular self-assembly of N-alkylamide organogels <u>Khacel Lella</u>, Ovarkez Guillaume, Medina-Gonzalez Yaocihuatl, Solvay - France</p>	
11:50am-12:30pm	<p>OP 13: 11:50-12:10 From microstructure to rheology of soft dense suspensions <u>Cuny Nicolas</u>, Mari Romain, Bertin Eric, Grenoble University, France</p>	<p>OP 14: 12:10-12:30 Percolation theory applied to Pickering Emulsions: Rheological behavior from dilute to concentrated domain Velandia Rodriguez Santiago Felipe, Sadtler Véronique, Marchal Philippe, University of Lorraine, France</p>	

12:30pm-02:00pm

Lunch Break

Exhibitors visits

Room		Room 1 (Maxwell)	
Chair	Joao Mala		
02:00pm-02:40pm		<p>OP 15: 2:00-2:20 3D real-time and in situ characterisation of the forming of ice-templated nanocellulose-based foams using ultrafast X-ray synchrotron microtomography <u>Martoja Florian</u>, Lakroune Yassine, Orgeas Laurent, Boker Elodie, Abdul Ghafour Tarek, Dumont Pierre J., INSA Lyon, France</p>	<p>OP 16 (remotly): 2:20-2:40 Influence of ultrasonication process parameters on the rheological behavior of cellulose nanocrystal dispersions <u>Girard Melanie</u>, Bertrand François, Tavares Jason R., Heuzey Marie-Claude - Canada</p>
02:40pm -3:20pm		<p>OP 17: 2:40-3:00 Relation between inter-particle force profiles, particle friction, and rheology in dense suspensions: an experimental proof using AFM. <u>Nguyen-Le Anh-Vu</u>, Colin Annie, Espci - France</p>	<p>OP 18 (remotly): 3:00-3:20 Constraining The Origin Of Fore-Aft Asymmetrical Shape Of Bubbles Rising In Viscoplastic Fluids <u>Zare Marian</u>, Pourzahedi Ali, Frigaard Ian, University of British Columbia, Vancouver - Canada</p>
03:20pm -04:00pm		<p>OP 19: 3:20-3:40 The influence of starch microparticles concentration on nonlinear rheological properties of Pickering emulsions. <u>Kijowska Karolina</u>, Ptaszek Anna, Ptaszek Pawel, Krak Joana, Nowak Marcelina University of Agriculture in Krakow - Poland</p>	<p>OP 20: 3:40-4:00 Transfer ratio for viscoelastic solutions. <u>Pingulkar Hrishikesh</u>, Peixinho Jorge, Crumeyrolle Olivier, Le Havre University - France</p>
04:00pm-04:15pm Coffee Break			
Room		Room 2 (Kelvin-Voigt)	Room 2 (Kelvin-Voigt)
04:15pm-05:10pm		<p>KN: 4:15-4:40 Interfacial engineering in complex multiphase systems. Zhang Huagui, Fujian Normal University, China</p>	<p>KN-RT: 4:40-5:10 Investigation of the rheological properties of methylcellulose in pharmaceutical products Christina Reichart, <u>David Duval</u>, Anton Paar</p>
5:10pm-8:20pm Visits & social program 2			
08:20pm-11:00pm Banquet Party (Lac Nantua, Restaurant Belle Rive)			

Thursday , July 8, 2021

Room	Room 1 (Maxwell)	
Chair	Philippe Marchal	
08:00am-08:45am	Plenary Lecture 4: Rheophysics of soft matter, Denis Roux (Laboratoire Rhéologie et Procédés, Grenoble University, France)	
08:45am-09:25am	<p>OP 21: 8:45-9:05 Rheological study of ceramic pastes for nuclear fuel extrusion shaping, Mougard Camacho Pierre-Francois, Valette Rudy, Castellani Romain, Doreau Franck, Poulesquen Arnaud, Mines ParisTech, Sophia Antipolis - France</p>	<p>OP 22 (remothly): 9:05-9:25 ATR-FTIR, a potential tool to evaluate apple puree rheology and structure, Lan Weige, Leca Alexandre, Jallais Benoit, Renard Catherine, Bureau Sylvie, Inrae, France</p>
09:25am-10:05am	<p>OP 23: 9:25-9:45 Percolation theory applied to Pickering Emulsions: Rheological behavior from dilute to concentrated domain - Velandia Rodriguez Santiago Felipe, Sadler Veronique, Marchal Philippe, Lemaitre Cecile, Bonn Daniel, Roques-Carnes Thibault, University of Lorraine - France</p>	<p>OP 24: 9:45-10:05 Coalescence in complex physicochemical systems Bertrand Torres Almeida Laura Melissa, Henaut Isabelle, Dalmazzone Christine - France</p>
10:05am-10:45am	<p>OP 25: 10:05-10:25 A novel measuring setup for characterizing the interfacial rheology of molten polymer systems: effect of molecular weight and temperature on the interfacial properties, El Omari Younes, Yousfi Mohamed, Jannick Duchet-Rumeau, Maazouz Abderrahim - INSA Lyon, France</p>	<p>OP 26: 10:25-10:45 Puncture Mechanics of Ultra-soft Hydrogels at Elasto-capillary Length Scale, Wei Yuanyuan, Creton Costantino, Narita Tetsuharu, Espci - France</p>
10:45am-11:10am	Coffee Break	

Room Room 1 (Maxwell)

Chair Rudy Valette

<p>11:10am-11:50am</p>		<p>OP 27: 11:10-11:30 Analysis and Modelling of Extrusion Foaming Behaviour of Low-Density Polyethylene using Isobutane and CO₂ – <u>Sandino Carlos</u>, Boyer Severine, Peuvrel-Disdier Edith, Laure Patrice, Hibert Geoffrey, Trolez Yves, Mines Paristech, Sophia Antipolis - France</p>	<p>OP 28: 11:30-11:50 Numerical simulation of the behavior of thermoplastic polymers during the extrusion and forming process. <u>E. Rahji</u>, T. Barriere and T. Cousin, University of Bourgogne Franche-Comté - France</p>
		<p>OP 29: 11:50-12:10 Rheology of hyper-concentrated nanocellulose gels during lubricated compression. Gupta Shubham, Martoia Florian, Orgeas Laurent, Dumont Pierre J., Grenoble University - France</p>	<p>OP 30: 12:10-12:30 Rheological criteria to predict polymer foamability using ScCO₂. <u>Rainald Benoit</u>, Delage Karim, Bounor-Legare Véronique, Chalaret Yvan, Cassagnau Philippe, University Jean-Monnet, Saint-Etienne France</p>

12:30pm-02:00pm Lunch Break

Exhibitors visits

Room Room 1 (Maxwell)

Chair Denis Roux

<p>02:00pm-02:40pm</p>		<p>OP 31 : 2:00-2:20 Structure, shear and elongation rheology of Multi-Micro-Nanolayers polymers based on polyethylene with varying macromolecular architectures <u>Touil Ibtissam</u>, Mazouz Abderrahim, Lamnawar Khalid. INSA Lyon, France</p>	<p>OP 32: 2:20-2:40 Flow of heterogeneous viscoelastic fluids in porous media <u>Esteban Lydia</u>, Delion Martial, Robert De Saint Vincent Matthieu, Bodiguel Hugues, De Loubens Clement, Morocho Luis, Laboratoire Rhéologie et Procédés, Grenoble University - France</p>
		<p>OP 33: 2:40-3:00 Rheological and morphological investigation of Poly (Lactic Acid)/Polyamide 11 biosourced multiphase systems from blends to multi-Micro-Nanolayers polymers. <u>Jaouadi Nour</u>, Mazouz Abderrahim, Lamnawar Khalid. INSA Lyon, France</p>	<p>OP 34: 3:00-3:20 Data-Driven Modelling of Polyethylene recycling under High Temperature Extrusion. <u>Casteran Fanny</u>, Delage Karim, Cassagnau Philippe, Hascoet Nicolas, Champaney Victor, Chinesta Francisco. University of Lyon 1, France</p>
		<p>OP 35 (remothly): 3:20-3:40 The Influence of Stearic Acid on Rheological Characteristics of Highly Loaded Polypropylene-based Compounds. <u>Patti Antonella</u>, Aciermo Domenico, Lecocq Hubert, Serghei Anatoli, Cassagnau Philippe University of Catania - Italy</p>	<p>KN-RT- Room 2: 3:40-4:10 Systemic rheology : Couette and Poiseuille analogies. <u>Marchal Philippe</u>, University of Lorraine - France</p>

04:10pm-04:30pm Coffee Break

04:30pm-07:00pm Free time for social activities

07:00pm-11:00pm Bus+Shuttle: Visit Lac Genin (30 min) & Wine & cheese at INSA Lyon @oyonnax + diner (08:00pm-11:00pm)

Friday , July 9, 2021

Room		Room 1 (Maxwell)	
Chair		Joao Maia	
08:00am-08:45am	Plenary Lecture 5 : A few examples of viscoplastic flow instabilities: experiments, theory and numerical simulations (Rudy Valette, CEMEF, MINES-ParisTech, Sophia-Antipolis, France)		
08:45am-09:10am		<p>KN Rheological Hysteresis in Soft Glassy Materials Divoux Thibaut, Laboratoire de Physique, ENS Lyon - France</p>	
09:10am-09:50am	<p>OP 36: 9:10-9:30 Rheological characterization of injectable hydrogel based on bio ceramic microspheres/ Pluronic (F127-F68)/Water system for bone repair. <u>Carrelo Henrique</u>, Soares Stephanie, Lopes Maria, Borges Joao Paulo, Cidade Maria Teresa, Universidade NOVA de Lisboa - Portugal</p>	<p>OP 37: 9:30-9:50 Advantages of systemic rheology to measure the rheological behaviour of an evolving system: How to deal with the kinetic instability of a Pickering emulsion ? <u>Ramos Diego</u>, Sadlier Veronique, Marchal Philippe, Lemaître Cecile, Roques-Carmes Thibault - France</p>	
9:50am-10:25am	Coffee Break		
10:25am-11:25am	<p>OP 38: 10:25-10:45 Recycling opaque and colored PET bottles into fibers by spunbond process. <u>Chet Elia</u>, Cassagnau Philippe, Ylla Noëlie, Brinkert Luc, University of Lyon 1 - France</p>	<p>OP 39: 10:45-11:05 Rheological Behavior Of Nanofluids: Influence Of Silica Nanoparticles In Xanthan Gum Solutions. <u>Buitrago Rincon Davan Lizeth</u>, Sadlier Veronique, Mercado Ojeda Ronald A., Marchal Philippe, Roques-Carmes Thibault, Pedraza Avella Julio A., Lemaître Cecile, University of Lorraine - France</p>	<p>OP 40: 11:05-11:25 Rheology Of Non-Brownian Dense Suspensions: An Optimised Implementation Of The Felicitous Domain Method In Openfoam <u>Orsi Michel</u>, Lobry Laurent, Gallier Stany, Peters François, University of Côte d'Azur, Nice - France</p>
Room		Room 2 (Kelvin Voigt)	
11:25pm-11:55am		<p>KN-RT: 11:25-11:55 Rotational and capillary rheometers complementarity. <u>Fraisse Frederic</u>, NETZSCH GmbH, Dardilly - France</p>	
11:55am-02:00pm	Lunch Break & picnic		

Exhibitors visits

Room		Room 1 (Maxwell)	
Chair	Philippe Marchal		
02:00pm-02:40pm		<p>OP 41 (remotely): 2:00-2:20 Rheological behavior of concentrated phosphate slurry. <u>Maazouzi Souhail</u>, Maazouz Abderrahim, Benkhaldoun Fayssal, Ouazar Driss, Mohammed VI Polytechnic University, Benguerir -Marocco</p>	<p>OP-42: 2:20-2:40 Rheology, dynamics and morphological investigations of PLA, PHBV and their blends: A suitable tools to probe miscibility and thermal stability. <u>Qiao Hu</u>, Maazouz Abderrahim, Lamnawar Khalid, INSA Lyon, France</p>
02:40pm -03:20pm		<p>OP 43: 2:40-3:00 Relaxation Mechanisms at long times of Crosslinked EPDM: Influence of the Chemistry and Nanofillers. <u>Le Hel Cindy</u>, Bounor-Legare Véronique, Cassagnau Philippe, Chevalier Christophe, Lucas Antoine, University of Lyon 1 - France</p>	<p>OP 44 (remotely): 3:00-3:20 Extensional rheological behaviour of polyolefin based nanocomposites exhibiting yield stress. <u>Yaeli Nanjunda</u>, Mapiété Jean-Charles, University of Jean-Monnet, Saint-Étienne - France</p>
03:20pm -04:00pm		<p>OP 45 (remotely): 3:20-3:40 Preliminary Rheological Study of Highly Loaded Composites by Magneto-Caloric Powder for Additive Manufacturing Applications <u>Ndri Dakmak</u>, Institut FEMTO-ST, Besançon</p>	<p>OP 46: 3:40-04:00 Investigating asphalt modified with polyurethane: morphology and rheology relationships <u>Gallu Raissa</u>, Méschin Françoise, Gerard Jean-François, Dalmas Florent, INSA Lyon - France</p>
04:00pm -04:20pm		<p>OP 47 : 4:00-4:20 Spreading and impregnation of drops of emulsions with banana leaves for phytosanitary applications <u>Alayan Abdallah</u>, Ligoure Christian, Banfignies Jean-Louis, Normand Alain, Charles Coulomb, Montpellier</p>	
04:20pm-04:30pm		Closing Ceremony (Room 1-Maxwell)	

Registration	
Opening /Closing	
Plenary Lectures	
Oral presentations	
KN/round tables	
Poster Session	
Social Activities	
Break	
Meeting	
Lunch	

Online oral presentations



Posters

Typdoc	Title	Speakers	Adress
poster	Relation between inter-particle force profiles, particle friction, and rheology in dense suspensions: an experimental proof using AFM.	Nguyen-Le Anh-Vu	ESPCI Paris-PSL, France
poster	The influence of starch microparticles concentration on nonlinear rheological properties of Pickering emulsions	Kijowska Karolina	University of Agriculture in Krakow, Poland
poster	The influence of sugar addition on rheological properties of black currant pectin gels.	Pancerz Michaël,	University of Agriculture in Krakow, Poland
Poster	Non-Linear Rheological Properties Of Fresh Food Foams Based On Ovoalbumin And Selected Polysaccharides	Nowak Marcelina	University of Agriculture in Krakow, Poland
Poster	Recycling of waste multilayer blown films towards the improving of their end-use values: Structure, rheology, tribology and morphological investigations	Geraldine Cabrera	IMP-UMR CNRS 5223, INSA Lyon, France

Abstracts

Stability of thin liquid films

Jan Vermant^{*†1}, Emmanouil Chatzigiannakis¹, Alexandra Aliche^{‡1}, Damian Renggli¹,
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Abstract

Thin liquid films determine the stability of many products in daily life and technology, ranging from beer foams, over the lung surfactant lining the walls of our alveoli, to emulsion and multiphase materials in general. The thinning of films represents a complex interplay between bulk rheology or hydrodynamics, capillarity and interfacial rheology and the role of intermolecular forces. This gives a quite formidable toolbox for the engineering of multiphase materials. We will discuss novel experimental and computational tools and the insights they provide.

Keywords: Rheology, thin films, Stability, multiphase materials.

*Speaker

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Rheology as a Critical Enabler of Polymer Processing: Extensional-Driven Mixing in Extrusion and Multi-Layered Tubes, Pipes and Bottles

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Abstract

The properties of plastic products are determined by their structure and morphology, which are dependent on the rheological behavior of the polymer melts during the complex flows that occur in most processing operations, such as extrusion, injection molding, thermoforming, blow molding and others. In this presentation, we will highlight the critical role of rheology in two recent significant developments in polymer processing technologies, those of (1) extensional-driven mixing of polymeric systems in extrusion and (2) continuous micro-layering of tubular structures.

The dispersive mixing capability of twin-screw extruders (TSE) is hindered by the fact that standard kneading elements (KE) impose a shear-dominated flow and, thus, suffer from limited ability to effectively disperse highly viscous disperse phases, be they polymeric or solids, in a polymeric matrix. Extensional flows, on the other hand, suffer from no such limitations and are theoretically capable of providing much improved dispersive mixing. However, attempts to perform extension-dominated mixing in extrusion operations have been few and far in between, with decidedly underwhelming results. Herein, a new type of extensional mixing element (EME) for single-screw extruders (SSE) and intermeshing TSEs will be presented and its efficiency in improving dispersive mixing significantly when compared, with nanoparticles of different geometries with standard two-lobed KEs will be demonstrated. In particular, we will show that the EMEs are highly efficient mixers for a number of systems, including immiscible and compatibilized polymer blends and polymer nanocomposites (0D, 1D and 2D). We will also demonstrate that by extending the EME concept to use in SSEs, it is also possible to significantly improve their dispersive mixing capability (a known shortcoming of SSEs), to the point the mixing levels become similar to those of TSEs equipped with standard KEs.

Continuous layer multiplication co-extrusion, able to yield structures with thousands of micro- and nan-scale layers is a process of substantial academic interest and achievement and one of proven industrial application. The industrial adoption of the process has primarily been in the realm of specialty and advanced products in film geometries for applications such as UV control and strength in windows, decorative coatings for marketing and aesthetics, and barrier applications for control of packaging environments. Recently, our group expanded the processing realm into annular geometries and has begun to demonstrate the functional application of such products. To date, novel layered foam tubing has been processed that could show potential strength improvements to light weight piping. Additionally, mechanical

*Speaker

strength improvements of layered pipes having weld lines have been demonstrated with the implementation of the technology of die head rotation in conjunction with layer multiplication. Most recently, the translation of this technology to the area of extrusion blow molding has been accomplished by the creation of the first bottles containing 131 layers.

Keywords: Extrusion, Polymer blending and compounding, Multilayering

Morphological transitions of flexible fibers in viscous flows

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Abstract

Here we present the individual dynamics of flexible and Brownian filaments under shear and compression. We use actin filaments as a model system and observe their dynamics in microfluidic flow geometries using fluorescent labeling techniques and microscopic tracking methods. Under shear we characterize successive transitions from tumbling to buckling and finally snake turns as a function of an elasto-viscous number. Under compression we reveal the formation of three dimensional helicoidal structures and characterize their formation. Finally, we attempt at linking the microscopic observations to the macroscopic suspension properties with preliminary measurements of the shear viscosity of dilute suspensions of actin filaments in microfluidic rheometers.

Keywords: filaments, suspensions, morphological transitions

*Speaker

Rheophysics of soft matter

Denis C D Roux*¹

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Abstract

In this presentation, soft matter is observed at macro-, meso- and nanoscale in order to seek spatio-temporal effects of elementary structures, constituting the complex fluids, when they are submitted to out of thermodynamic equilibrium state by mechanical solicitations. When a flow is applied, a coupling arises with architectural organization at the different scales. Understanding the mechanisms behind the different behaviors enables us to improve processes in the fields of food, biomedical, materials, environment, etc. In this presentation, various materials will be taken as an example in order to show the implication on the rheological behavior of the temporal and spatial dependencies of the aggregates. Aggregates can come from organizations of organized molecular systems such as: elongated and flexible micelles, living cells, biological molecules such as DNA or crystallizing lipid gels.

Keywords: Rheology, Rheophysics, Structure, Aggregate, Complex fluids

*Speaker

A few examples of viscoplastic flow instabilities: experiments, theory and numerical simulations

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Abstract

Many complex fluids (pastes, granular, suspensions, emulsions ...) exhibit a strongly non-Newtonian rheology. They thus flow very differently from fluids such as air, water or honey. We will show various examples of flows (stretching or compression of filaments, jets, impacts, extrusion ...) presenting instabilities linked to their viscoplastic behaviour. We will then discuss using experiments, numerical simulations and simple theoretical analysis the mechanisms involved in these flows. Several examples will be proposed and we will emphasize the interest of numerical simulation both for the understanding of simple phenomena, but also the prediction of more complex flows encountered in nature and industry.

Keywords: Modeling, Viscoplastic fluids, Filaments, Drops

*Speaker

Critical Role of Interfacial Diffusion and Diffuse Interphases in Multi-Micro-/Nanolayered Polymer Films: A Combined Rheological and Dielectric Study

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Abstract

It is known that the macroscopic properties of multilayer polymer films are largely dominated by the diffuse interphase formed via interfacial diffusion between neighboring layers. However, not much is known about the origin of this effect. In this work, we reveal the role of interfacial diffusion and the diffuse interphase development in multilayer polymer films mainly by rheological and dielectric fingerprints, based on a compatible poly(vinylidene fluoride) (PVDF)/poly(methyl methacrylate) (PMMA) system fabricated by forced assembly micro-/nanolayer coextrusion. Interestingly, the layer morphology is found to prevail in all investigated multilayer films, even for the nanolayered system where the interdiffusion is substantial. It is also demonstrated that, in the presence of macromolecular and geometrical confinements, interfacial diffusion significantly alters the crystalline morphology and microstructure of the resulting micro-/nanolayered films, which leads to quantitatively different dielectric and rheological properties. More importantly, the combination of dielectric relaxation spectroscopy and energy dispersive X-ray analysis further reveals that multiple diffuse interphases with various length scales exist in the multilayer structures. The presence of these multiple interphases is explained in terms of a proposed physical picture for the interdiffusion of fast-mode mechanism occurring in coextrusion process, and their length scales (i.e., interphase thicknesses) are further mapped quantitatively. Our work provides new insights into the effects of interfacial diffusion and diffuse interphases, towards tailoring interfaces/interphases in micro-/nanolayered polymer structures and for their advanced applications.

Keywords: forced assembly coextrusion, multilayer films, interfacial diffusion, interface, interphase, structure, properties

*Speaker

Rheological Hysteresis in Soft Glassy Materials

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Abstract

The nonlinear rheology of soft glassy materials is captured by its constitutive relation, shear stress versus shear rate, which is most generally obtained by sweeping up or down the shear rate over a finite temporal window. For a huge amount of complex fluids, the up and down sweeps do not superimpose and define a rheological hysteresis loop. By means of extensive rheometry coupled to time-resolved velocimetry, we unravel the local scenario involved in rheological hysteresis for various types of well-studied soft materials. We introduce two observables that quantify the hysteresis in macroscopic rheology and local velocimetry, respectively, as a function of the sweep rate. Strikingly, both observables present a robust maximum with the sweep rate, which defines a single material-dependent time scale that grows continuously from vanishingly small values in simple yield stress fluids to large values for strongly time-dependent (or thixotropic) materials. In line with recent theoretical arguments, these experimental results hint at a universal time scale-based framework for soft glassy materials, where inhomogeneous flows characterized by shear bands and/or plug-like flow play a central role.

Keywords: Hysteresis, yield stress fluids, thixotropy, shear banding, Mnemosyne, memory effects, shear history

^{*}Speaker

Rotational and capillary rheometers complementarity

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Abstract

Rotational rheometers are used to characterize materials flowing and formulated products structure. As a function of instrument and chosen geometries, viscosity measurements are performed on a range of relatively low shear rate. By comparison capillary rheometers are able to determine sample's viscosity on a range of relatively high shear rate. Consequently, both technologies are complementary. Moreover a lot of accessories can be add on rheometers to extend their capabilities. We will discuss of these instruments and accessories to explain how to choose the best technology and the best configuration for each application sectors like coatings, molten polymers, asphalt, cosmetics, suspensions, emulsions and more generally complex fluids.

Keywords: Rotational, Capillary, Rheometer, Netzsch

*Speaker

Rheology and sustainable development

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Abstract

The use of renewable biological resources from land and sea, like crops, forests, fish, animals and microorganisms to produce materials and energy is often considered as a possible substitute to former fossils resources. Clearly this is an important part of today's industrial activity, and of bioeconomy. However, bioeconomy is not synonymous to sustainability, since the latter requires a high level of change of our production modes, whereas the former is rather based on the substitution between environmental and economic capitals. Sustainability raises significant challenges in terms of resource efficiency, wastes treatment, recycling use, eco-design, etc. All these challenges involve flows of matter, with variable compositions and ill-known properties. Such hurdles make very difficult the control of these flows. To overcome these difficulties, rheology can have a significant contribution because of its interdisciplinarity, connecting different scientific domains, and of universality, due to its wide range of applications. In this introductory presentation we will illustrate this contribution through several examples from various fields of applications. For instance, the golden age of polymer rheology was in the last century, but it can be applied now to much more complex systems, integrating new functions, and taking into account environmental aspects as well as the circular economy. Then we will strive to draw some prospects for rheology to improve its impact in solving the problems that industrial activity faces to tackle the sustainability goals.

Keywords: biomass, circularity, environment, flow, industry, matter

*Speaker

Systemic rheology : Couette and Poiseuille analogies

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Abstract

Rheological characteristics of formulated products affect drastically their performances and their end-use properties and are particularly dependent on the manufacturing process. Therefore, in semi-batch or continuous processes, the inline monitoring of the viscosity is a major issue. However, when complex fluids are blended, emulsified or are part of a reaction in reactors (e.g. agitated tanks or static mixers), the description of their rheological behavior, as finished or intermediate products, is not an easy task since industrial flow conditions are very far from being well defined and are systematically rheologically heterogeneous. To overcome, to some extent, these difficulties, we have developed a method to estimate the in – line process viscosity through a simplified analysis of complex flow situations equivalent to a macroscopic momentum balance. This global or "systemic rheology" approach is based on a "Couette analogy" and in a "Poiseuille analogy" consisting in determining the geometrical dimensions of an empty cylindrical cup or tube, equivalent in terms of "torque *vs.* angular velocity" and "pressure drop *vs.* flow rate" relationships than the real device. It is shown that "torque *vs.* angular velocity" and "pressure drop *vs.* flow rate" data can be transformed into "shear stress *vs.* shear rate" curves that are in fairly good agreement with off-line measurements, obtained with conventional rheometers. The "Systemic Rheology" approach (and the development of rheo-reactors) constitutes an original methodological alternative for formulation engineering or engineering of formulated products. Several examples of applications, concerning different industrial sectors, illustrate the potential of this approach.

Keywords: Systemic rheology, Couette and Poiseuille analogies, Formulation, Product engineering

*Speaker

Investigation of the rheological properties of methylcellulose in pharmaceutical products

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Abstract

Methylcellulose is used in the pharmaceuticals industry for a wide range of different applications from solid to liquid. Therefore, it is important to generate as much information as precisely as possible about the substance. Rheological measurements can provide valuable information for production, filling and storage. Continuous quality control is essential in order to maintain consistently high quality. Tests like oscillatory, thixotropic and temperature tests on methylcellulose at different concentrations were carried out. The viscoelastic behavior is illustrated and discussed.

Keywords: methylcellulose, pharmaceuticals, rheological properties

*Speaker

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Interfacial engineering in complex multiphase systems

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Abstract

In the past several decades, a great deal of human endeavour has been made to manufacture complex structured materials (e.g. blends/composites, multi-layered and porous materials, etc.) based on multiphase systems to meet emerging demands in a vast range of industries. The phases that are combined together are often thermodynamically incompatible or dissimilar while the multiphase system can remain kinetically metastable thanks to the irreplaceable role of the interface and the presence of surface active agents (e.g. polymers, particles and else) being anchored at the interface. However, due to the technical challenges in examining interfaces, few insights have been given to the interface and its effect on the properties of multiphase systems is rarely understood.

This work is to evidence how to engineer an interface in multiphase systems to tune the macroscopic properties of the systems. Firstly, interfacial rheology will be introduced to disclose and quantify dynamics at interfacial region based on different systems, i.e. the interface between different polymer melts (PMMA/PVDF) and the interface between gas/liquid or liquid/liquid phases. The understandings of such interfacial dynamics in controlling properties of coextruded multi-layered polymer structures and of Pickering foam/emulsions will be then presented. Secondly, interface engineering by designing functional particles to be anchored at liquid interface provides ample opportunities of advanced materials manufacture. Examples such as novel caesium-selective adsorbent particles with dual functionality to be recovered via froth flotation and pH-responsive polymeric microgels that stabilize smart emulsions are to be presented. Finally, potential research directions with new interface engineering technology will be envisaged.

Keywords: multiphase system, interfacial rheology, polymer, Pickering emulsion

*Speaker

Impact de la rhéologie volumique d'un gel faible de Kappa-Carraghénane sur l'interface Huile-Eau.

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Abstract

Une émulsion est un mélange hétérogène de deux liquides normalement non miscibles, où la dispersion de goutte dans la phase continue est maintenue par un agent stabilisant qui réduit la tension à l'interface entre goutte et phase continue. Ces agents de surface " sont soit des tensioactifs ; molécules à caractère amphiphile leur conférant une affinité à l'interface, soit des particules (émulsion de Pickering). L'avantage d'utiliser des particules solides est l'irréversibilité de leur adsorption à l'interface conduisant à des émulsions extrêmement stables.

Cependant, l'introduction des particules modifie les propriétés de l'interface mais peut également modifier les propriétés volumiques des deux phases. Une compréhension complète du processus de stabilisation et d'émulsification avec des particules doit donc prendre en compte aussi bien les propriétés de l'interface que du volume. Par ailleurs, la mesure des propriétés de l'interface, rhéologiques en l'occurrence, met en action aussi bien la déformation de l'interface que du volume ? La question qui se pose alors, comment décorrélérer les deux réponses, ou comment la rhéologie du volume impacte-elle les mesures de la rhéologie interfaciale ?

Ce travail vise à anticiper l'impact de la phase continue d'un microgel sur la rhéologie interfaciale en prenant comme modèle une goutte de polybutène (Indopol L-6) dans un microgel faible : Le Kappa-Carraghénane (KCar), un polysaccharide largement utilisé comme gélifiant dans l'industrie alimentaire, choisi particulièrement pour sa gélification réversible en fonction de la température. Il présente la particularité de ne pas présenter des propriétés tensioactives et, compte tenue de l'hystérèse de la réversibilité de la transition sol/gel, on pourrait obtenir un liquide ou un gel d'une solution donnée à la même température. En modifiant

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la concentration en KCar, il est possible de faire varier le seuil d'écoulement, ou le module élastique des gels sur une large gamme et surtout d'obtenir des seuils d'écoulement très faible.

La question principale abordée dans ce travail est de comprendre l'effet du seuil d'écoulement sur les mesures des modules viscoélastiques interfaciaux, ainsi que sur la tension de surface par la méthode de la goutte pendante.

Keywords: Fluide à seuil, rhéologie interfaciale, gel, interface huile, eau

Non-linear rheology characteristics of the gel point: large amplitude oscillatory shear behavior of chemically and physically crosslinked hydrogels

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Abstract

Non-linear rheology and particularly large amplitude oscillatory shear (LAOS) has gained considerable attention in the last decade [1]. Analyses based on Fourier Transform rheology (FT-Rheology), Chebyshev polynomial expansion, and Lissajous plots have been suggested as means of characterizing LAOS behavior in different polymeric systems. Yet, little attention has been devoted to the non-linear behavior of crosslinked systems and more specifically, the evolution of non-linear rheological properties at the different stages of the crosslinking process. Poly(vinyl alcohol) hydrogels can be formed by chemical crosslinking with glutaraldehyde or by freeze-thaw cycles. In these cryogels, the linkages of a three-dimensional network of polymer chains are formed through non-covalent interactions. Reaction induced evolution of viscoelasticity during gelation of semi-dilute poly (vinyl alcohol) (PVA) in aqueous dispersions was studied through rheological measurements in the linear and non-linear domains. These measurements are contrasted with the properties of the physical gels formed by the freeze/thaw cycles. The work primarily focuses on the characteristics of the LAOS behavior near the gel point. 1. Hyun et al., "A review of nonlinear oscillatory shear tests: Analysis and application of large amplitude oscillatory shear (LAOS)" *Prog Polym Sci* 2011, **36**, 1697–1753.

Keywords: Nonlinear, rheology, SAOS, LAOS, Crosslinked polymer

*Speaker

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Structure-rheology-properties relationships of biocomposites suspensions based on poly(lactic acid) and bioactive glass fillers

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Abstract

Poly(D,L-lactide) PDLLA/Bioglass® (BG) biocomposite for medical devices was obtained by a new approach process at elevated temperatures. The aim of the present work was to gain a fundamental understanding of the relationships between morphology, elaboration conditions and final properties of these biomaterials. The filler contents as well as their thermal treatments (i.e. from 580°C to 800°C to develop specific crystalline structures), and particle sizes (ranging from 3.5 μm to 0.5-1mm diameter) were investigated. PDLLA degradation was analyzed with Fourier Transform Infrared (FTIR) spectroscopy, Size-exclusion chromatography (SEC) and mechanical characterization. Therefore, the rheological behavior in the linear viscoelastic regime of the controlled PDLLA/BG systems was assessed and modeled taking into account the molar masses changes during the manufacturing process. Hence, the effects of BG contents and nature on the melt rheological properties were investigated and discussed. The obtained results corroborated the rheological and the molar masses measurements. The present findings is a guideline to obtain biocomposites with a well-controlled dispersion and optimized mechanical properties. Finally, the formation of a carbonate-hydroxyapatite (caHA) layer in composite surface was confirmed with in vitro test highlighting their huge potential in medical devices.

Keywords: Biocomposites, rheology, Degradation, modelling, bioactive glass, biodegradable composites, bioglass®, Extrusion at elevated temperature, Dispersion

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Miscibility, rheological and thermo-mechanical properties of compatible biopolymer blends: influence of process parameters and natural surfactants

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Abstract

In the recent years, a growing interest in biodegradable plastics as an alternative to the conventional fossil fuel-based polymeric materials was developed. Particularly, PLA is broadly applicable for use as an alternative to petrochemical- derived products. In fact, this polymer is biodegradable and biocompatible and its properties are very similar to those of some synthetic fossil fuel-based polymers. Nevertheless, the range of application of PLA is limited due to its fragility, poor barrier properties and the limited temperature range at which it can be used. Various strategies were proposed to overcome these limitations, such as modifying the chemical structure of the polymer with plasticizers or blending with other polymers. In this work a polylactic acid PLA (70 wt%) and poly- hydroxy butyrate PHB (30 wt%) blend was prepared to obtain a bioplastic with mechanical properties intermediate between those of the two polymers. Specifically, the aims of the work were improve the miscibility of the blend and increase the thermo-mechanical properties. Two different approaches were used to achieve these goals: the study of the influence of process parameters and the introduction of natural compatibilizers in the blend. For the first, a co-rotating twin screw extruder LEISTRITZ ZSE 18/40D ($\Phi = 18$ mm, $L/D = 40$) was used with three different screw profiles. The investigated formulations were: unfilled PLA/PHB blend and containing 5 wt% of an organo-modified clay (Cloisite 5). In the second part two types of natural surfactants with different chemical structure were used: an ethylene oxide/propylene oxide block copolymer (Synperonic) in the form of flakes and a mixture of two liquid surfactants with a variable lipophilic–hydrophilic index (HLB 12). In this case, PLA/PHB blends were prepared using a DSM Explore twin screw mini-extruder ($T=180^{\circ}C$ and screw speed=100 RPM). The investigated formulations were: PLA/PHB with HLB12 ranging from 0.1 wt% to 5 wt% and PLA/PHB with Synperonic in the same range of content. Morphological, thermo-mechanical and rheological analyses were performed on each formulation in both case studies. Firstly, a correlation between the observed morphology and the screw profile was found; in particular, the milder screw profile was the best solution. This result is supported by rheological analyses: an increase of the storage modulus (G') was obtained after the adding of Cloisite, while the unfilled PLA/PHB blend exhibits a shoulder in the G' curve caused by the relaxation of the dispersed phase which is in form of droplets, showing the typical rheological response of an immiscible blend. In the second part of the study, the

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morphological and the rheological analyses showed that HLB 12 was more effective than Synperonic. In fact, the trend of G' in this last formulation was similar to that of the uncompatibilized blend. Conversely, samples containing HLB 12 showed a different trend of G' curve and a decrease of the curve slope in the terminal region can be observed, as well. This behaviour can be attributed to the obtainment of a complex morphology, significantly different from that of the neat blend PLA/PHB. Nevertheless, while for HLB 12 system, it was necessary the use of a solvent for their introduction into the extruder, the Synperonic presents the advantage of introducing a solid additive during the process. As far as the thermo-mechanical analyses are concerned, both types of compatibilizers induced excellent mechanical properties at high temperatures, resulting in an increased HDT value that allows to widen the application range of the obtained materials.

Keywords: PLA, PHB, polymer blends, rheology, small amplitude oscillatory shear

Detection of damage localization in Polymer Networks by Space-resolved Multi-speckle Diffusing Wave Spectroscopy (MSDWS)

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Abstract

In order to investigate the mechanism of toughness of soft polymer networks, it is necessary to cover a wide range of length scale: from the molecular scale with bond breaking in stretched chains to the macroscopic scale with the crack propagation and rupture of the material. Though it is essential to understand the mechanics of the networks in the intermediate length scale where the stress and damage are spread in a certain damage zone near the crack tip, experimental tools adequate to this mesoscopic scale are limited. We developed time- and space-resolved multi-speckle diffusing wave spectroscopy (MSDWS) coupled with a macroscopic mechanical tester, to provide a new approach to study fracture mechanics in mesoscopic scale. The key requirement for DWS measurements in polymer networks is introduction of probe particles to provide multiple scattering in the system. With MSDWS where 2D images of speckles are acquired by a CCD camera, ensemble averaged dynamics of the probes reflecting local changes in the damaged network can be achieved with both temporal and spatial resolutions. With a polydimethylsiloxane elastomer as model system, we performed simultaneous MSDWS measurements in backscattering and macroscopic delayed fracture tests in uniaxial traction. Dynamic distribution mapping was performed with high temporal (1 s) and spatial (400 μm x 400 μm) resolutions. During induction period of macroscopic fracture, we discovered heterogeneous dynamics distribution presumably due to fracture precursor. The zone with accelerated dynamics grows before macroscopic crack propagation, over a period up to 1 h. Under different deformation, the dynamics distribution on the sample shows different patterns, which corresponds to the stabilization or propagation of the crack. The technique and the combination with other characterization methods will provide us complete vision to the research of fracture mechanism and advanced molecular design for soft material development.

Keywords: Diffusing Wave Spectroscopy, elastomer, fracture, in situ

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Multi-Micro/Nanolayer Films Based on Polyolefins: New Approaches from Eco-Design to Recycling

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Abstract

The present work describes a future-oriented approach for the valorization of polyethylene-based multilayer films. The method involves going from eco-design to mechanical recycling of multilayer films via forced assembly coextrusion.

The originality of this study consists in limiting the number of constituents, reducing/controlling the thickness of the layers and avoiding the use of tie layers. The ultimate goal is to improve the manufacturing of new products from recycled multilayer materials by simplifying their recyclability. Within this framework, new structures were developed with two polymer systems: polyethylene/polypropylene and polyethylene/polystyrene, with nominal micro and nanometric thicknesses.

Hitherto, the effect of the multi-micro/nanolayer architecture as well as initial morphological and mechanical properties was evaluated. Several recycling processes were investigated, including steps such as: (i) grinding; (ii) monolayer cast film extrusion; or (iii) injection molding with or without an intermediate blending step by twin-screw extrusion.

Subsequently, the induced morphological and mechanical properties were investigated depending on the recycling systems and the relationships between the chosen recycling processes or strategies, and structure and property control of the recycled systems was established accordingly. Based on the results obtained, a proof of concept was demonstrated with the eco-design of multi-micro/nanolayer films as a very promising solution for the industrial issues that arise with the valorization of recycled materials.

Keywords: recycling, eco, design, coextrusion, multilayers

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Injectability of thermosensitive chitosan hydrogels in terms of capillary rheometry

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Abstract

In recent years, the field of biomedical engineering has seen a growing interest in innovative treatment and regeneration methods. Thermosensitive polymer systems characterized by a lower critical solution temperature (LCST), which undergo sol-gel phase transition with increasing temperature, seem to be an interesting solution. Most often, this phenomenon is used to design injectable cell scaffolds forming a three-dimensional structure *in vivo*. For this purpose, polymers of natural origin such as chitosan or alginate are increasingly used. Although these systems are often studied, their application potential as low invasive injections is determined solely based on the LCST values. Simultaneously, there were almost no studies determining the possibility of injecting them.

Colloidal chitosan systems, which underwent sol-gel phase transition under physiological conditions, were prepared in accordance with the methodology described in detail in the literature. Clearly, 400mg of crab-derived chitosan was dissolved in 16ml of 0.1M hydrochloric acid or 20ml of 0.1M acetic acid. After 24h storage at room temperature, the sample was cooled for 2h at 4 °C. Next, a suspension of disodium β -glycerophosphate (2g of powder distributed in 2ml of distilled water) was added dropwise to the colloidal chitosan solution. The prepared sample was stored at 4 °C for 24 hours.

Injectability tests were carried out using the Brookfield CT3 texture analyzer operating in compression mode with a 2g trigger. During the measurement, the force needed to inject the medium was recorded. The 2ml syringes filled with 0.5ml experimental medium and 14G-25G needles were used. The measurements were used to assess both the composition of the hydrogels (solvent used), storage conditions as well as the impact of injection parameters such as needle diameter and length, and the injection rate. For this purpose, the values of the characteristic forces of the injection process were determined, i.e. the initial glide force IGF, the dynamic glide force DGF as well as the maximum force. Moreover, the analysis was also based on the value of the total work needed for injection. The rheological properties of colloidal chitosan systems were determined using an Anton Paar MCR301 rotational rheometer with a cone-plate measuring system, using a frequency sweep test and measurement at constant deformation under non-isothermal conditions (5-60 °C).

Based on the measurements, it was clearly stated that in most cases it is possible to inject the tested systems. For a typical injection rate, the use of the smallest needles (23G-25G) led to exceeding the recommended dynamic glide force values, while not exceeding the maximum allowable value. Based on the raw experimental curves of the force against distance, the critical role of the initial stage of shear thinning or flow resistance was determined. Regardless

*Speaker

of the measurement conditions as well as the needles used, the IGF force was not critical, which is observed for most other systems. Moreover, it was found that the injection rate used significantly affects the force values obtained, due to other Poiseuille flow parameters, mainly shear rate values.

Analysis of the influence of the solvent used allows to state that smaller values of the force necessary for injection were obtained in the case of chitosan systems obtained by dissolving the biopolymer in acetic acid. This results from the different viscoelastic properties of the tested systems than in the case of hydrogels with hydrochloric acid.

Based on the measurements carried out, the key impact of injection temperature was also determined. Storing the medium at room temperature for about 2 hours before the injectability test resulted in a decrease in the value of forces needed for injection, which results from a characteristic decrease in the viscosity value with increasing temperature. It is worth noting that the storage time was not long enough to observe a rapid development of the spatial network, which would impede application.

Discussion of the results will be based on the phenomena characteristic for capillary rheometry of non-Newtonian fluid, e.g. by reference the forces needed to injection as the necessary overpressure needed to overcome the flow resistance as well as by determination the influence of the residence time at a constant shear rate value.

Keywords: injectability, thermosensitive hydrogels, chitosan, capillary rheometry

Studies on Rheology of Wood Polymer Composites

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Abstract

Wood polymer composites (WPC) are materials with pseudoplastic and viscoelastic properties. They have yield stress and exhibit slip during flow. Studies on extrusion and rheology, as well as on process modeling of these highly filled materials are limited [1,2]. Extensive rheological studies on the wood polymer composites based on the polypropylene matrix were performed. Viscous and slip flow properties were determined (with Rabinowitsch, Bagley and Mooney corrections) at broad range of shear rate and temperature, using a high pressure capillary rheometer. An effect of the corrections on the rheological characteristics of WPC was discussed. Two characteristic regions of the slip behavior were observed. At the low shear stress, the slipping was weak, which was followed by a sharp increase in the slip velocity at the high shear stress. Viscosity was modeled using the Klein model, and simulations of extrusion process were performed with the use of recently developed computer model of WPC extrusion [2].

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Keywords: wood polymer composites, slip, rheology

*Speaker

Rheology and Process Modeling for Polymeric Materials with Yield Stress and Wall Slipping

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Abstract

There are several materials like filled polymers, composites, elastomers, pure polymers like PVC and HDPE, polymer suspensions, as well as ceramic materials and foodstuffs like meat and dough which display wall slippage under certain conditions. Also, it is well known that many materials have a yield stress, e.g. filled polymers, composites, as well as blood, paints, cosmetics and foodstuffs like margarine and mayonnaise. These materials are called viscoplastics.

Comparing with fundamental studies on the viscoplastic flows and flows with slip effects, much less research has been devoted to these flows in processing. In this paper, rheological studies on wood polymer composites are presented, and modeling of extrusion of these materials is discussed.

Wood polymer composites (WPC) are pseudoplastic, viscoelastic materials that exhibit yield stress and wall slipping. Studies on rheology and extrusion, as well as process modeling of these highly filled materials are very limited. These were recently discussed in review papers [1,2].

An extensive rheological study on the wood polymer (PP) composites were performed. Viscous and slip flow properties were determined (using Rabinowitsch, Bagley and Mooney corrections). Then, flow simulations in the extrusion screw/die system were performed using ANSYS Polyflow CFD software, and the results were discussed with respect to both rheological and modeling aspects.

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Keywords: rheology, wood, plastic composites, modeling, extrusion, slip

*Speaker

New Poloxamer 407 / PEG self-healing suspended hydrogel for 3D printing fluid materials

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Abstract

In the medical field [1], 3D printing by material deposition has limitations on forming soft materials, coming from fluids like silicones [2]. Their deposition in a gelled support medium enable to overcome these limits with a support of the material during its liquid-solid transition. The success of this technique involve a control of the support medium, enabling it to quickly self-heal during 3D printing while holding the printed object [1].

In this study, we developed a new approach which controls the static yield stress of the Poloxamer 407 hydrogel used as a support medium for 3D printing, thanks to the addition of poly(ethylene glycol) (PEG). In order to understand and determine the strengths of this self-healing and supporting medium, differential scanning calorimetry was performed to study the microstructure as well as rheology and 3D printing. In aqueous solutions, Poloxamer hydrogels forms by its micelles entanglement [3]. Here, the PEG disturbs this entanglement, by steric hindering, which adapts precisely the static yield stress of the hydrogel [4]. By the "salting-out" [5] effect, the hydrophobic parts of the Poloxamer 407 dehydrate causing the decrease of the gelation temperature. The obtained results enhances the ability of the gel to perform 3D printing of fluid materials at room temperature.

3D printing of complex anatomical shapes will be exposed to demonstrate the strengths of this technique. The understanding of structure-properties relationship of Poloxamer/PEG hydrogels enable to transfer this technology to the bioprinting field and meet the challenges of the personalized and regenerative medicine.

Keywords: 3D printing, Support medium, Static yield stress, Micelles, Poloxamer 407.

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How to predict cell viability in Bioprinting

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Abstract

The use of biomaterial including living cells in additive manufacturing, is one of the most advance technology in tissue engineering and regenerative medicine [1]. 3D bioprinting presents the capacity to produce efficiently and in a cost-effective way, tissues with cell density and shape recapitulating human tissue behaviours [1]. Nevertheless, the use of LDM (liquid deposition modelling) technic presents limitations such as cells mortality, due to a high shear stress value inside the dispensing system [2,3].

In this study, we evaluated the biomaterial capacities to protect cells in our additive manufacturing process. The method is based on the relation between bioink viscoelastic properties, sizing of deposition system and cells viability. To access the shear stress map inside the bioprinting system, a specific algorithm was developed based on Poiseuille tube flow of a pseudoplastic power law fluid [4]. Different cells source were used and their capacity to withstand stress investigated. Living and labelled necrotic cells were counted before and post-printing process to evaluate cell viability and total cell recovery in various conditions.

In view of results, the shear stress gradient can be controlled through bioink rheological behaviour and sizing of dispensing system. Cells viability seems to be directly related to shear stress value but also duration. In any case, it was shown that using adequate bioink rheological properties, cells viability can be optimal whatever the dispensing system and the applied flow value; protecting cells during the 3D bioprinting process.

In future, building an experimental data library will allow biomaterial engineers to use the algorithm in reverse engineering mode, to tailor 3D bioprinting system and/or biomaterials properties according to cells source. This approach will represent a disruptive innovation for patient specific surgery using autologous living implants.

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Keywords: Bioprinting, biomaterial properties, shear stress, cell viability

The role of solvent on the rheology and the supramolecular self-assembly of N-alkyldiamide organogels

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Abstract

Over the past decade, organogels have aroused the interest in the field of the rheology control of non-aqueous media. Indeed, there is an industrial need to develop new shear thinning viscoelastic formulations capable of suspending various actives for applications such as cosmetics, agrochemistry or solvent borne coatings. Up to now, most of the rheology modifiers of organic media in the industry are based on hydrophobically modified inorganic compounds (such as clays or silica colloids). However, these technologies display industrial implementation issues (Health and Safety environment) and long-term stability problems. To tackle these challenges, we propose an approach based on the supramolecular self-assembly of Low Molecular Weight Gelators (LMWG), such as N-alkyldiamides derivatives that lead to organogels in a large scope of solvents.

The aim of this PhD work is to study the solvent effect on the self-aggregation of these molecules, the growth of the anisotropic structures giving organogels and the rheological properties of the obtained materials. More specifically, the role of solvent has been rationalized using different solvent scales such as Hansen parameters (δ_d , δ_p , δ_h), Kamlet-Taft parameters (α , β , π^*) and Catalan parameters (SA, SB, SP, SdP) in order to identify the governing factors of the solvent responsible for the rheological properties and structural features of these organogels.

The N-alkyldiamide organogels are physical gels which self-assembly is due to non-covalent interactions such as Van Der Waals forces or H-bonding, hence these materials are thermoreversible. Indeed, the gels are prepared by cooling down a hot solution of the gelator in the solvent. During the sol-gel process, the material tends to contract, leading to misestimated rheological values. Thus, we propose here to study by rheology the gel behavior of N-alkyldiamides in different solvents using a Normal Force Controlled (NFC) protocol[1] instead of a constant gap protocol. The gap is adjusted during the sequence to compensate for the sample contraction. (*Figure 1b*) The kinetics of gelification (viscoelastic modulus G' and G'' , vs. time and temperature) and the linear viscoelastic region (LVER) of these materials (frequency and shear strain sweeps) have been determined.

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It was found that the value of the G' plateau during the kinetics of gelification is highly linked to the solvent capability of H-bonding (Hansen δ_h parameter and SA, SB Catalan parameters). Indeed, correlation plots with different solvent parameters have been established in order to show the dependency of the G' value towards the solvent parameters. For the N-oleyl-diamide organogels, the gel strength seems to increase with the H-bond solvent capability which can be a sign of a specific solvent-gelator cooperative interaction. (*Figure 1b*) Structural analysis of these organogels such as SAXS-WAXS and AFM have pointed out the existence of a fibrillar network which density and typical lengths are correlated to the solvent nature and the organogelator concentration. Finally, the thermodynamics of gelification has also been studied by differential scanning calorimetry (DSC) to investigate the sol-to-gel transition with regards to the solvent parameters.

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Keywords: organogels, self assembly, supramolecular, solvent, rheology

From microstructure to rheology of soft dense suspensions

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Abstract

Soft dense suspensions, as microgels for example, cover a large spectrum of materials made of soft particles immersed in a liquid phase. Although the nature of those solid particles can be very different, dense suspensions share some common rheological properties under simple shear, such as shear-thinning (below jamming) or Herschel-Bulkley relation between stress and shear-rate (above jamming) with a yield-stress. Establishing an evolution equation for the stress tensor as a response to a given deformation is of great importance in order to describe the flow of such systems. Most attempts to get such a constitutive model are purely phenomenological ones and based on symmetries and frame indifference. In this work we were interested in getting such a constitutive model starting from the particle dynamics in order to connect the microscopic particle properties to the rheology. For this, we used tools from statistical physics to establish an equation on pair correlation function- a function describing the structure of the system- getting then a constitutive equation on the stress tensor by using the link between this pair correlation function and the stress tensor. Indeed, by some considerations on the structure of the suspension we can get two evolution equation for the stress and the pressure both below and above the jamming. Those equations allow us to describe shear-thinning below jamming and yield-stress fluid above jamming for simple shear deformation. We thus use this model to get some predictions on various rheological protocol and compare it to simulations’ result.

Keywords: Yield, stress, shear, thinning, statistical physics, dense suspensions

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Percolation theory applied to Pickering Emulsions: Rheological behavior from dilute to concentrated domain

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Abstract

Pickering emulsions, also known as particle-stabilized emulsions, are a type of fluids presenting interesting rheological behaviors due to the coexistence of particles and droplets dispersed in a continuous phase. While at low dispersed phase fractions and low particles concentration these emulsions behave as Newtonian dispersions of particles and droplets, increasing these parameters can lead to complex viscoelastic systems such as yield stress fluids. In recent years, particle-stabilized emulsions have been a major subject of research mainly because they offer long-term stability and greater resistance against shear. Indeed, the high anchoring energy at the water-oil interface makes particle adsorption an almost irreversible process. Besides, following their nature, using particles to stabilize interfaces represents an attractive alternative to design emulsion-based process and products such as encapsulation activities and eco-friendly systems.

The analysis and modelling of the rheological behavior is shown to be an interesting method for the design of particle-stabilized emulsions. Indeed, from the bulk rheology of Pickering emulsions it is possible to study macroscopic properties of the system while formulation parameters are considered through inter-particle and inter-drop interaction phenomena. For classical emulsions such as direct (O/W), reverse (W/O) and High Internal Phase Emulsions (HIPE), the dispersed phase volume fraction (ϕ) is a major parameter that has been used to model and predict the rheological behavior. Simultaneously, it has been showed that rheological behavior of Pickering systems is also mainly dependent on particle concentration. As both parameters are directly related to percolation threshold, our main objective was to study the rheological behavior of Pickering emulsions from dilute to concentrated domain, giving special attention to the shift point between these regions based on dispersed phase volume fraction as well as explaining the resulting behavior from a percolation approach.

As W/O Pickering emulsions are an accurate example of complex, water-in-dodecane emulsions stabilized with hydrophobic silica nanoparticles were used as model system. First, formulation parameters as silica concentration and ionic strength were analyzed. Results showed shear-thinning behavior, elastic modulus prevalence over viscous modulus and allowed to confirm the concentration of particles as the main parameter affecting Pickering

*Speaker

emulsions mechanical behavior. Subsequently, shear viscosity and elastic modulus of emulsions were characterized at $\phi \in [0.1;0.55]$ and $\phi \in [0.40;0.75]$ respectively. Finally, a model based on percolation theory was adjusted and compared to other existing models of Pickering and classical emulsions, demonstrating that our approach provides a physical explanation and is well suited to this type of complex system.

Keywords: Pickering, Rheology, Dispersed Phase Volume Fraction, Percolation theory, Emulsions

3D real-time and in situ characterisation of the forming of ice-templated nanocellulose-based foams using ultrafast X-ray synchrotron microtomography

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Abstract

Nanocelluloses (cellulose nanocrystals, CNCs, or cellulose nanofibrils, CNFs) are the elementary reinforcing constituents of plant cell walls [1]. Because of their pronounced slenderness and outstanding intrinsic mechanical properties, nanocelluloses constitute promising building blocks for the design of future biobased high-performance materials such as nanocomposites, dense and transparent films, continuous filaments, and aerogels and foams. The research interest in nanocellulose-based aerogels and foams is recent but growing rapidly. These materials constitute novel lightweight bio-based materials with high mechanical and insulation properties and thus have a great potential in a wide diversity of engineering fields. Nanocellulose foams can be used in the building and car-manufacturing industry as heat/and or sound insulation board, as porous template for the storage and generation of energy, as membrane for fluid purification, or even as scaffold for biomedical applications [2]. Nanocellulose foams can be obtained by ice templating, *i.e.*, by freezing aqueous colloidal nanocellulose gels and sublimation of the formed ice crystals (freeze-drying) [1, 3]. The ice templating process has received considerable attention in recent years owing to its simplicity and to the wide variety of porous materials that this technique can provide. However, in the case of nanocellulose gels, growth mechanisms of ice crystals formed during the freezing phase, *i.e.*, when they squeeze and consolidate the remaining nanocellulose gels up to very high nanofibre concentrations, are complex and still poorly understood. This severely hinders the design and optimization of the cellular microstructure of ice-templated nanocellulose foams. In this study, we performed in situ solidification experiments of CNF and CNC gels using ultrafast X-ray synchrotron microtomography 3D imaging. Thanks to the 3D images acquired during these experiments, we could follow in real time, for the first time for this kind of materials, the solidification front, the growth of ice crystals and the associated mechanisms and kinetics of foam cell wall formation.

Keywords: Nanocellulose gels, yield stress fluid, foams, ice templating, Xray synchrotron microtomography, in situ characterisation

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Influence of ultrasonication process parameters on the rheological behavior of cellulose nanocrystal dispersions

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Abstract

Cellulose nanocrystals (CNCs) show promise for use as nanofiller in polymers. Indeed, CNC provides high mechanical strength as the hydroxyl groups on their surface form a percolating network. But the presence of these hydroxyl groups induces low CNC compatibility with non-polar matrices: surface modification is needed to better exploit these biosourced nanoparticles.

For handling purposes, CNC is usually dried and has to be dispersed in the right medium before any modification. However, the drying process (industrial spray-drying in our case) leads to agglomeration; the interparticle forces are very strong and difficult to break, because of the CNC's high aspect ratio. Ultrasonication is efficient to overcome these forces, employing acoustic waves that create compression and rarefaction zones. These lead to cavitation, generating energy levels high enough to break interparticle bonds. Herein, we recommend a standardized approach, which has been unfortunately lacking in the available literature.

Rheological measurements help validate these guidelines, by allowing us to evaluate the dispersion state. A double-Couette flow geometry is used in both shear rate and frequency sweep modes. Results show that viscosity values present lower intra- and inter-variability when CNC are dispersed efficiently. The comparison of different parameter sets is done by studying their standard deviation rather than the viscosity values. For best results, the sonication probe is placed off-center and near the medium-air interface in a small volume to promote fluid circulation and increase the probability that agglomerates are exposed to cavitation.

The influence on rheology and on the dispersion state of different concentrations (ranging from liquid-like to gel-like suspensions) is considered. The effect of the intrinsic medium properties such as polarity, viscosity or density, is also studied.

While the focus of this work is on CNC suspensions, the agglomeration issue is encountered nearly all types of nanoparticles due to their high surface-area-to-volume ratio (and resulting strong interparticle forces). This work could thus be transposed to several other systems.

Keywords: cellulose nanocrystal, ultrasonication, rheology, dispersion

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Relation between inter-particle force profiles, particle friction, and rheology in dense suspensions: an experimental proof using AFM.

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Abstract

TBD

Keywords: dense suspensions, shear thickening, AFM, inter, particle force profiles, friction measure

*Speaker

CONSTRAINING THE ORIGIN OF FORE-AFT ASYMMETRICAL SHAPE OF BUBBLES RISING IN VISCOPLASTIC FLUIDS

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Abstract

A significant number of fluids used in our daily life like toothpaste, hair gel and peanut butter, as well as working fluids in geophysical and industrial settings, such as magma and cement slurry, are categorized as viscoplastic fluids. The key feature of viscoplastic fluids is their characteristic yield stress. More precisely if such fluids are subjected to a stress more than their yield stress they deform and flow, otherwise they exhibit a solid-like behavior. The transition from a solid-like regime to a fluid-like regime is often approximated using rheological models such as Bingham, and Herschel-Bulkley constitutive laws. In these models, the build-up and breakdown of structure are assumed to be reversible and instantaneous, i.e., the material regains its value of the yield stress upon the flow reversal. This ideal yield-stress behavior implies that elastic and thixotropic effects are negligible. Upon adopting this assumption, the rise of a bubble and creeping motion of a sphere settling in a yield stress fluid have been studied numerically, e.g. [1,2]. Their results confirmed that the developed flow in the yielded envelope zone enclosing moving objects or bubbles is symmetric, when the particles/bubbles are symmetric.

However, the experimental observations published in [3-6] started researchers thinking that real yield stress fluids might not be as simple as predicted from the aforementioned constitutive laws. The results published in [3,4] dramatically illustrated the fore-aft asymmetry of flow around a spherical particle settling in a yield stress fluid. And the cusp shape at the bubble's lower side, observed in [6] markedly contradict the fore-aft symmetry expected theoretically in Stokes flows. These results were more surprising since the yield stress fluid used in those experiments, Carbopol, was often assumed to be a simple yield-stress fluid, with negligible thixotropic behaviour.

The inverted teardrop shape could be attributed to a number of reasons. One of which is the elasticity of the Carbopol before yielding and a little sign of thixotropy/hysteresis in its flow curves around the yielding point. There have been many studies aiming to address

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this shortcoming by including time-dependent parameters into the constitutive law equations. Assuming the bubble is injected from a nozzle, one other possibility is that the shape is formed at the origin and the yield stress of material prevents evolution of bubble's shape and causes the shape to remain 'frozen' [2].

The goal of this study is to explore the potential contribution of the entry point on the bubble shape in a yield stress fluid and constrain the origin of fore-aft asymmetry. A series of novel experiments are conducted exploring a potential cause of fore-aft asymmetry of a rising bubble in a simple yield stress fluid. It has been believed that the yield stress could prevent bubbles injected from a nozzle to freely adjust their shape and smooth the developed cusp on their lower side. This possibility is investigated by introducing a bubble, with controlled shape, and speed, into a yield stress fluid, Carbopol, without direct injection from a nozzle. Our results rule out this possibility and show that a slight change in the rheology can have significant effect on the shape of rising bubbles, though still fore-aft asymmetric.

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Keywords: Fore and aft asymmetrical bubbles' shapes, Yield stress fluids, Time-dependent rheology, Stokes flow, Bubbles

The influence of starch microparticles concentration on nonlinear rheological properties of Pickering emulsions

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Abstract

Emulsions are heterogeneous systems consisting of at least two immiscible liquids, one of which is a continuous phase and the other a dispersed phase, in the form of fine droplets. Emulsions are thermodynamically unstable systems and therefore tend to destabilise due to phenomena such as creaming, sedimentation, flocculation or coalescence. The most important for the durability of the emulsion is the correct selection of the substance that serves as a stabilizer for the given type of emulsion. There are different types of emulsifiers used in food emulsions, whose main mechanism of operation is to limit the aggregation of dispersed phase particles by reducing the interphase tension, i. e. surfactants, phospholipids. Currently, there is a growing interest in Pickering emulsions, where the stabilization mechanism consists in adsorption of very small particles on the interphase surface instead of the surfactant, thus creating a physical barrier around the dispersed phase. This is solid adsorption and Pickering emulsions are therefore considered more durable than surfactant-based emulsions. Therefore, an attempt was made to obtain emulsions which would provide the basis for a variety of products due to the expected structure. Emulsions were obtained by selecting the optimal ratio of water to oil phase and the amount of stabilizer.

The first stage of this study was to obtain the starch fractions of increased crystallinity by enzymatic hydrolysis and then use them to stabilize food Pickering emulsions. In order to obtain the desired properties of starch fractions, the process of enzymatic hydrolysis of starch was optimized, including selection of raw material, selection of enzyme and selection of appropriate parameters of the preparation process. The next stage assumed the characteristics of the obtained starch fractions.

In particular, the determination of the degree of crystallinity and the analysis of particle size distribution using dynamic light scattering. On the basis of the results obtained, the starch fractions were selected, which were used as a stabilizer to produce an oil in water (O/W) type emulsion. The obtained emulsions were subjected to microscopic image analysis, stability testing, including checking the stability of the obtained emulsions over time, and above all rheological characteristics. Rheological characteristics are crucial when designing a new product to be manufactured on an industrial scale. The obtained emulsion is the basis for the new product, however, on the basis of rheological characteristics of the material, it is possible to assess the technological suitability for a specific end product.

The experiment plan involved influence starch microparticles concentration on rheological

*Speaker

properties of O/W emulsions. Large Amplitudes Oscillatory Shear (LAOS) were used to study the nonlinear viscoelastic properties of selected emulsions. It was demonstrated that all tested systems exhibited viscoelastic properties, but the area of linear dependency was limited. The systems exhibited the intersection of G' and G'' (first Fourier harmonic) in the range of deformation from 0.01 to 1.0. Analysis was shown characteristic maxima on G'' which is typical for multiphase systems.

Also dissipation coefficient was calculated as a function of strain amplitude. Also geometrical decomposition of Lissajous curves was used to calculate Chebyshev coefficients for elastic and viscous parts. Viscous Lissajous curves were characterized double loops. This rheological study allowed

to point the main factor determining rheological properties of O/W emulsion and shown the future direction of industrial application of this kind multiphase systems.

Keywords: Pickering emulsion, LAOS, rheological behavior

Transfer ratio for viscoelastic solutions

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Abstract

The printing industry deals with viscoelastic inks, which contain particles, surfactants and polymers. While printing, liquid from one surface is transferred to another surface through the formation of capillary liquid bridge. This transfer of the liquid from one surface (donor) to another surface (accepter) becomes complex because of the various factors such as gravity, capillarity, viscosity, elasticity, surface properties and stretching properties, which affect the amount of liquid transferred to the accepter. Here, the transfer ratio is the amount of liquid transferred to the moving plate (accepter) to the total amount of liquid on both the plates, after stretching. Our study mainly focuses on the study of the transfer ratio for viscoelastic fluids with pinned contact lines (i.e. no-slip condition) on the border of circular plates. The viscoelastic solutions of various concentrations are prepared using high molecular weight poly-ethylene oxide (PEO) and poly-ethylene glycol (PEG) polymers in water. By moving the top plate, the liquid bridge formed in-between the flat plates of capillary breakup extensional rheometer (CaBER) is stretched, and then, the volumes of solution pools formed on the each plate are measured by using high speed image microscopy. From the experiments conducted, it is observed that the transfer ratio decreases with increase in concentration of PEO. To understand the influence of stretching parameters on transfer ratio, the plate diameter, initial stretching height, final stretching height and the stretching speed are varied. Increase in the plate diameter, initial stretching height and the final stretching height lead to a decrease in the transfer ratio, whereas an increase in transfer ratio is observed for high stretching speeds. The influence of the initial bridge shape, the movement of bottom plate and initial, as well as, final stretching height on the transfer ratio will be discussed.

Keywords: Transfer ratio, viscoelastic solutions, capillary liquid bridge

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Spreading and impregnation of drops of emulsions with banana leaves for phytosanitary applications

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Abstract

Sigatoka is a foliar fungal, disease, largely widespread in tropical areas that can ravage banana plantations. In collaboraton with la Compagnie Fruitière, we aim to optimize the spraying of phytosanitary products on banana leaves to fight more effectively against Sigatoka, by minimizing the quantity of phytosanitary products to be delivered in order to reduce the environmental impact of these sanitary treatments To do that we are developing original formulations of these emulsion-based phytosanitary products (called slurries), we will characterize their rheological properties, including , the spraying , impact and spreading of drops of these slurries onto banana leaves as well as the impregnation of the leaves by these products using different techniques (high speed image analysis, and micro-infrared spectroscopy).

Keywords: Sigatoka, emulsions, banana leaves, rheological properties

*Speaker

ATR-FTIR, a potential tool to evaluate apple puree rheology and structure

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Abstract

Apple purees are typical non-Newtonian foods, being concentrated of soft and deformable insoluble particles in an aqueous solution of sugars, organic acids and pectins. Rheological parameters such as apparent viscosity and visco-elastic moduli, as well as structural properties (e.g. particle size and shape, and cell wall contents) are thus relevant indicators to understand the variability of food quality. The measurements are however time consuming, require a significant amount of sample, and do not allow simultaneous measurements. Attenuated total reflectance Fourier transform spectroscopy (ATR-FTIR) is a potential solution to rapidly and accurately qualify the biochemical composition of agricultural commodities and processed food [1]. The possibility of this technique to determine rheological and textural variations of fruit purees has been explored in this work insofar as it could be highly beneficial to the integrated evaluation of fluid food quality.

In this study, ATR-FTIR (4000-700 cm⁻¹) was applied on fresh and freeze-dried apple purees processed under various conditions: fruit variety, fruit cold storage and mechanical puree refining, in order to evaluate the ability of ATR-FTIR to predict rheological properties (viscosity and viscoelasticity), particle distributions (sizes and volume) and cell wall content (alcohol insoluble material), in comparison with classical reference measurements. Using linear partial least squares (PLS) regressions, ATR-FTIR applied directly on fresh purees satisfactorily predicted the apparent puree viscosity at 50 s⁻¹ (0.69-2.50 Pa.s, R² > 0.86), which is admittedly the shear rate corresponding to the in-mouth perception of food particle dispersion. ATR-FTIR also showed a surprising ability to monitor fresh purees' particle sizes averaged over volume (R² > 0.90) and over surface area (R² > 0.86). ATR-FTIR on freeze-dried purees provided specific assessments of visco-elastic moduli in amplitude sweep tests (6-3612 Pa of storage modulus and 2-860 Pa of loss modulus, R² > 0.83), yield stress (0.58-57.58 Pa, R² > 0.84) and cell wall content (R² > 0.88).

Consequently, ATR-FTIR technique has been proved to be a valid method, using few steps

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to provide simultaneous assessments of biochemical, rheology and textural properties of fluid foods. Rapid and direct spectral measurements could offer valuable and sufficient information on food rheology for industrial and laboratorial demands. Based on our results, future works could be extended to a wide span of complex shear-thinning food or non-food materials.

Reference:

S. Bureau, D. Cozzolino, C.J. Clark, Contributions of Fourier-transform mid infrared (FT-MIR) spectroscopy to the study of fruit and vegetables: A review, *Postharvest Biol. Technol.* 148 (2019) 1-14.

Keywords: apple purees, Mid infrared spectroscopy, rheology and structure, shear, thinning food

Percolation theory applied to Pickering Emulsions: Rheological behavior from dilute to concentrated domain

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Abstract

Pickering emulsions, also known as particle-stabilized emulsions, are a type of fluids presenting interesting rheological behaviors due to the coexistence of particles and droplets dispersed in a continuous phase. While at low dispersed phase fractions and low particles concentration these emulsions behave as Newtonian dispersions of particles and droplets, increasing these parameters can lead to complex viscoelastic systems such as yield stress fluids. In recent years, particle-stabilized emulsions have been a major subject of research mainly because they offer long-term stability and greater resistance against shear. Indeed, the high anchoring energy at the water-oil interface makes particle adsorption an almost irreversible process. Besides, following their nature, using particles to stabilize interfaces represents an attractive alternative to design emulsion-based process and products such as encapsulation activities and eco-friendly systems.

The analysis and modelling of the rheological behavior is shown to be an interesting method for the design of particle-stabilized emulsions. Indeed, from the bulk rheology of Pickering emulsions it is possible to study macroscopic properties of the system while formulation parameters are considered through inter-particle and inter-drop interaction phenomena. For classical emulsions such as direct (O/W), reverse (W/O) and High Internal Phase Emulsions (HIPE), the dispersed phase volume fraction (ϕ) is a major parameter that has been used to model and predict the rheological behavior. Simultaneously, it has been showed that rheological behavior of Pickering systems is also mainly dependent on particle concentration. As both parameters are directly related to percolation threshold, our main objective was to study the rheological behavior of Pickering emulsions from dilute to concentrated domain, giving special attention to the shift point between these regions based on dispersed phase volume fraction as well as explaining the resulting behavior from a percolation approach.

As W/O Pickering emulsions are an accurate example of complex, water-in-dodecane emulsions stabilized with hydrophobic silica nanoparticles were used as model system. First, formulation parameters as silica concentration and ionic strength were analyzed. Results showed shear-thinning behavior, elastic modulus prevalence over viscous modulus and allowed to confirm the concentration of particles as the main parameter affecting Pickering

*Speaker

emulsions mechanical behavior. Subsequently, shear viscosity and elastic modulus of emulsions were characterized at $\phi \in [0.1;0.55]$ and $\phi \in [0.40;0.75]$ respectively. Finally, a model based on percolation theory was adjusted and compared to other existing models of Pickering and classical emulsions, demonstrating that our approach provides a physical explanation and is well suited to this type of complex system.

Keywords: Pickering, Rheology, Dispersed Phase Volume Fraction, Percolation theory, Emulsions

Coalescence in complex physicochemical systems

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Abstract

The mechanisms of coalescence is of huge importance in the process of soft matter and divided materials such as emulsions and foams. It controls their stability and their final properties in many applications including cosmetics, food engineering, energy, biotechnology etc. It has a significant influence in wastewater treatment. Actually oil removal relies on gravitational methods that are favored by coalescence. In Enhanced Oil Recovery, water treatment is all the more challenged by residual polymers and surfactants that constitute complex physico-chemical systems. The objective of the thesis is to characterize the mechanisms involved in the coalescence of such complex systems. Three main configurations will be investigated: coalescence between two oil droplets, coalescence between oil droplet and interface and heterocoalescence between oil droplet and gas bubble (in the case of flotation). Each case includes successive drainage and rupture steps during which both hydrodynamic and molecular forces are implied at different scales between viscoelastic interfaces. Their relative influence will be identified and studied using appropriate physico-chemical compositions : brines of different salinities, various organic phases, polymers and surfactants solutions. They will allow to deal with various interfaces and thin films. Interfaces will be characterized by IFT measurements and interfacial rheology. Flow properties of the thin films will be addressed using rheometers and microfluidic devices. A dedicated set-up will enable to film the coalescence of an oil droplet with various interfaces at mesoscopic scales. Film thinning will be characterized by induction times using a high speed camera. Their thickness evolving will be measured by interferometry. Additional macroscopic experiments will be performed. Lab flotation column will be used for the study of oil droplets and air bubbles attachment. Centrifugation and bottle tests will assess the tendency of oil droplets to merge. This set of experiments will demonstrate how small scale phenomena can govern macroscopic ones. The obtained results will promote understanding of the coalescence and identification of key parameters. They will constitute relevant data to achieve coalescence physical modeling.

Keywords: coalescence, interfaces, rheology, polymers, surfactant

*Speaker

A novel measuring setup for characterizing the interfacial rheology of molten polymer systems: effect of molecular weight and temperature on the interfacial properties

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Abstract

Interfacial rheology studies on low viscosity liquids such as oil–water interfaces and foams have been the subject of a large body of scientific works. However, academic reports related to the interfacial rheology applied to high viscosity molten polymer systems were never carried out until today’s date. It is related to the limited available rheological equipment used to perform interfacial rheology at the melt state in addition to the restricted measuring temperature. In the present work, the interfacial shear properties of molten semi-crystalline polymer systems have been successively probed. To do that, a custom design of a new interfacial rheological cell based on lightweight biconical geometry was developed. This measuring setup allows interfacial rheology measurements up to 200 °C with a temperature measurement accuracy of 1 °C at the sample interface. Two incompatible molten polymers were used here, the Polycaprolactone (PCL) and the Polyethylene glycol (PEG). The apparent interfacial shear properties were measured in both oscillatory and steady flow modes. The contribution of the bulk subphases was corrected during the processing of the data. The effects of the temperature and the molecular weight were highlighted. It was found that from a critical molecular weight and with increasing the temperature, the formation of an interphase was evidenced.

Keywords: interfacial shear rheology, bicone, interface, interphase.

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Puncture Mechanics of Ultra-soft Hydrogels at Elasto-capillary Length Scale

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Abstract

Although the mechanical properties of hydrogels have been intensively studied worldwide in the last two decades, the mechanism of fracture initiation in brittle and/or ductile hydrogels has not been fully understood. We think that a key element which needs to be studied is the effect of the elasto-capillarity: the surface tension can play a significant role in mechanics at the length scale shorter than the elasto-capillary length, defined as the ratio of the surface tension to the modulus, in particular at the very short length scale in the crack initiation process. In order to investigate fracture mechanics in the capillary regime, we focus on ultra-soft hydrogels having a long elasto-capillary length. Since it is difficult to carry out reliable fracture experiments with ultra-soft hydrogels with conventional methods, we performed puncture experiments [1] in order to detect and visualize large local deformation and failure in such soft solids.

We used a laboratory-made puncture setup using a precision laboratory balance as force sensor, and a translation stage for the indenter (flat-end needles). Poly(vinyl alcohol)-glutaraldehyde chemical hydrogels with elastic moduli ranging from 80 to 2000 Pa were prepared and used as a model soft hydrogel system. The elastic moduli of the hydrogels were measured by both indentation at small displacement and shear rheometry. We found that the moduli measured by indentation match well those measured by rheometry. At a constant indentation velocity, fracture occurs when the indenter breaks through the surface of the gels in deep indentation and can be detected as a sharp drop of the puncture force. The critical force P_c and the critical distance dc at fracture point were characterized as a function of the gel modulus G' and the indenter radius R .

For gels with a modulus higher than 400 Pa, we found that the critical distance dc at fracture was practically independent of the modulus, as reported previously for gels in a kPa range. Interestingly, dc increases with decreasing gel modulus for $G' < 400$ Pa, suggesting that the softer gels are more resistant to puncture. This could be due to the strain-hardening behavior of the soft PVA gel observed in the large strain region, presumably due to heterogeneities in cross-linking chains. Most importantly, we found that the normalized nominal stress $P_c/(3.14R^2 \cdot G')$ shows two distinct regimes when it is plotted against the indenter radius normalized by the elasto-capillary length, $R \cdot \gamma/G'$. Above the characteristic length around $R \cdot \gamma/G' \sim 2$, $P_c/(3.14R^2 \cdot G')$ is almost constant while it sharply increases below the characteristic length. This observation suggests the transition from elastic fracture to

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capillary fracture.

Fakhouri, S., S. B. Hutchens and A. J. Crosby (2015). "Puncture mechanics of soft solids." *Soft Matter* **11**(23): 4723-4730.

Keywords: Puncture, Fracture, Surface tension, Elastocappillary length, Ultra, soft hydrogel

Analysis and Modelling of Extrusion Foaming Behaviour of Low-Density Polyethylene using Isobutane and CO₂

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Abstract

In this work, modelling of physical foaming extrusion of LDPE is carried out in order to achieve a better understanding of the mechanisms involved in foam manufacturing. Foaming by extrusion is a four-step process. First, the pellets are introduced and molten in an extruder. Gas is then injected under pressure and dissolved in the polymer matrix. The mixture is then significantly cooled to give more strength to the material while maintaining a certain level of pressure. Finally, foam expansion occurs at the die exit. At this location, the dissolved gas undergoes a strong decompression leading to the nucleation and growth of bubbles. The objective of this study is to better understand the origin of the limitation of foaming based on the combination of an experimental analysis of the foaming process and the prediction of a model.

The modelling tools are focused on the expansion occurring at the die exit, in order to quantify all the important parameters for the control of the foaming structure. The model, labelled as "cell model", is based on previous works [1,2] and considers the growth of a single bubble in a mixture of polymer matrix and dissolved gas (blowing agent) [3]. In order to take into consideration the viscoelastic character of the polymer, the rheological behaviour is represented by a multi-Maxwell model.

The foaming behaviour of two LDPE commercial grades provided by Total Research & Technology Feluy (Belgium) is compared for two different foaming agents (isobutane and carbon dioxide). The extrudate expansion at the die exit is analysed experimentally for different conditions (mainly temperature and gas concentration). An analysis of the extrusion parameters is performed to determine the quantity of dissolved gas that is effectively used for the foaming process. In order to compare with the experimental results, the cell model considers the gas concentration and the relaxation spectrum of each LDPE grade. As a consequence, particular attention is devoted to the determination of the solubility and the diffusivity of the blowing agent in the molten polymer based on literature data. The main effort concerns the analysis of the influence of the rheological properties of the two LDPE grades and the properties of the blowing agent on the size and stability of the cells.

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The modelling predictions are compared with the foam expansion and the foam density, revealing that the use of the cell model provides an accurate estimation of the final properties of the foam in the case of isobutane. The difference in final foam density is used to make hypotheses on the physical phenomena which can limit the foam expansion. Indeed, gas loss or polymer crystallization can limit the foam expansion and this is related to the temperature at the die exit [4]. Nevertheless, there is an open question on the role of strain hardening behaviour on foam expansion [3,5]. These different hypotheses will be discussed.

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Keywords: Physical foaming, Modelling, Polyolefins, Cell Model

Numerical simulation of the behavior of thermoplastic polymers during the extrusion and forming process

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Abstract

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Key Words: Thermoplastic polymer; extrusion; rheological simulation; single layer

The extrusion of polymers is a mature process for the production of films, tubes and pipes mainly used in the transport of automotive fluids [1] [2]. It is with this in mind that multilayer parts are more and more in demand. The subject of extrusion is very broad and several works have tackled many aspects, from the design of equipment and tools for extrusion (die, extrusion head, feed, etc.) to fluid mechanics and multilayer flow analysis and interfacial defects.

The study consists in establishing rheological behavior models making it possible to simulate the flow in the molten state of the material for the polyamide polymers used during the extrusion phase for the manufacturing of polymer tubes.

The study of rheological behavior as a function of temperature, the design of extrusion heads and distributors will be compared between experimental results and simulations. In particular, the influence of the different extrusion parameters on the flow of material at the die level for the extrusion of tubes, where the control and stability of the process will be examined.

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Keywords: Thermoplastic polymer, extrusion, rheological simulation, single layer

Rheology of hyper-concentrated nanocellulose gels during lubricated compression

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Abstract

Nanocelluloses such as cellulose nanofibrils (CNF) or cellulose nanocrystals (CNC) are mainly extracted from vegetal biomass and prepared in the form of colloidal aqueous suspensions. CNF's are slender and tortuous nanofibres (length $L > 1 \mu\text{m}$, width $d \approx 5\text{-}100 \text{ nm}$) [Moon *et al.*, 2011; Klemm *et al.*, 2018]. CNC's are rod-shaped nanoparticles ($L \approx 100\text{-}200 \text{ nm}$, $d \approx 5\text{-}20 \text{ nm}$). Nanocelluloses have attracted a considerable attention due to their outstanding mechanical properties. They can be used to produce various types of materials such as cellular materials (aerogels, foams) [Gupta *et al.*, 2018], nanocellulose films [Larsson *et al.*, 2014], nanocomposites [Moon *et al.*, 2011], hydrogels or continuous filaments [Håkansson *et al.*, 2014], using various processing routes during which nanocellulose suspensions behave as complex fluids. For a better control of the flow conditions of these gels during their processing, it is crucial to characterise and model their rheology under various loading conditions such as shear and compression, in concentrated and also hyper-concentrated regimes (from 1%wt to 20%wt). However, experimental data in the hyper-concentrated regime are scarce. Thus, we developed an original methodology to prepare concentrated (1%wt) to hyper-concentrated (15wt%) nanocellulose suspensions with CNC's, TEMPO-oxidized CNF's and enzymatic CNF's, i.e., with three different particle types, which were characterised with optical and TEM micrographs, and colloidal stabilities. The prepared gels were then subjected to uniaxial compression experiments using an universal tensile testing machine (Shimadzu) equipped with a 100N load cell. Tests were performed in perfect slip conditions at various strain rates. Some of the compression experiments were carried out with optical visualization techniques to investigate their flow mechanisms. The tests were performed at room temperature (25 C) with initial strain rates varying from 0.001 s^{-1} to 0.8 s^{-1} and axial strain levels up to 0.9.

The experimental results show that Nanocellulose suspensions exhibit various types of compression behaviour, depending on their colloidal stability and their meso- and microstructure. TEMPO-oxidised CNF and CNC gels are stable suspensions, they exhibit yield stress fluid behaviour at lower strain rates followed by shear thinning behaviour at higher strain rates. Enzymatic CNF gels are unstable and flocculated and also exhibit a yield stress. Above it, these suspensions flow with cracks of their fibrous networks and from which substantial water permeation is observed, especially at lower strain rates. The experiments also revealed the effect of concentrations of nanocellulose suspensions on the rheological properties of these gels. Their yield stress tends to follow power law with the concentration c^n , where c is

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concentration and n is 1.5 for TEMPO-oxidized CNF suspensions, 3.8 for CNC suspensions and 2.7 for enzymatic CNF suspensions.

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Keywords: Nanocellulose gels, Hyper concentrated regimes, Compression rheology, Mesoscale flow visualisation, Flow heterogeneities

Rheological criteria to predict polymer foamability using ScCO₂

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Abstract

Polymer foams have a major role to reduce the consumption of plastic and to enhance mechanical properties, thermal insulation, etc.... Basically, foaming can help us having the same result or better with less material.

Foaming can occur through two ways, chemical, a chemical reaction produce gas like during the formation of PU foam, and physical, which is to use a dissolved physical agent, which go through instability and create bubble while undergo a thermodynamic instability produced by an increase of temperature or a reduction of pressure. The use of CO₂ as a physical blowing is not new, nevertheless the prediction of the foamability of a polymer is not completely achieved. So there is a need for simple criteria depending of the physical property of the polymer which can be easily measure to predict the foaming behavior of polymers.

Our work is to use rheological measurements, linear shear and non-linear elongationnal, to predict the foaming behavior of different Polypropylene formulations having really different rheological behaviors. Based on different PP formulations, we defined two rheological criteria for linear and non-linear rheological behaviors. We showed from some foams examples (cell morphology and foam density) that these criteria are really relevant can be used to optimize the polymers formulations.

Keywords: Physical Foaming, elongationnal rheology

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Structure, shear and elongation rheology of Multi-Micro-Nanolayers polymers based on polyethylene with varying macromolecular architectures

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Abstract

The aim of this study is to understand the confinement effects on the rheology and molecular dynamics in multilayer polymeric structures fabricated by the forced-assembly multilayer coextrusion process. Alternating multilayers of polyethylene (PE) and rigid polymers (higher Tg as well as PS and PC) have been studied. The rigid polymers act as confining system on PE (i.e., confined polymer). The PEs with varying macromolecular architectures, short (SCB) and long chain branching's (LCB) were used. Various nano-structured multilayer structures with stable flow and layered architecture were then obtained using the layer multiplying technique. Various characterization techniques such as wide and small angle X-ray scattering (WAXS/SAXS), electron microscopies (SEM or TEM), and rheology were used to probe the structure, molecular dynamics of polyethylene chains and morphology in the obtained multi-micro/ nano-layered systems. Interestingly, we found that macromolecular and geometrical confinements arising respectively from the high Tg layers. Micro and nanolayered instabilities were observed depending of the viscoelastic mismatched properties and interfacial tensions. The layer multiplication affect strongly the crystallization microstructure /morphology and molecular orientations of polyethylenes. Moreover, the molecular and geometrical confinement influences remarkably the rheology and molecular dynamics of polyethylene depending of the amount of LCB. From micro- to nano-layers, rheological and dynamic behaviours are strongly dependent on the number of layers (or the individual layer thicknesses) and the compositions. Relevant mechanisms involving molecular rheology theories are proposed to elucidate the reasons underlying the changes especially in the extensional rheology behaviour. Results of this work will contribute to better understand the confinement effects in multi-layered assembly. The obtained materials with tailored nanostructures are dedicated for various applications ranging from ultra-barrier films from cast extrusion to flexible and high transparency sheets for thermoforming process.

Keywords: Rheology, morphology, interface, nanostructured polymer films, Coextrusion, instabilities

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Flow of heterogeneous viscoelastic fluids in porous media

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Abstract

The problematic of fluid mixing has been widely studied from both fundamental and industrial perspectives. However, the case of heterogeneous non-Newtonian fluids, i.e. viscoelastic inclusions within a viscoelastic matrix, is essentially not considered in the literature. These fluids are of primary relevance in sectors such as petrol, food and health industries. For some applications, the use of conventional homogenization procedures (vortex, mixers) is not always possible, due for instance to the small amount of sample available (e.g., biofluids: blood, mucus), or when homogenization takes place in specific environments (e.g., enhanced oil recovery).

In the present study we propose and characterise a passive homogenization method based on the injection of the sample through a porous media. We therefore analyse how the contrast in viscoelasticity between the inclusion and matrix phases affect the heterogeneous fluid, as well as the influence of the curvature of the channel.

We analyse two main configurations: (i) viscoelastic inclusions within viscoelastic matrix (V/V case), and (ii) viscoelastic inclusion within Newtonian matrix (V/N case). We use solutions of Flopaam 6035S (SNF Floerger), a synthetic polymer acrylamide-based with high molecular weight, and of glycerol, as viscoelastic and Newtonian phases respectively. The rheological contrast between the inclusion and matrix phases is varied through their relative concentrations. Furthermore, the inclusion phase is seeded with graphite particles to characterise the flow with bright-field imaging.

The homogenization devices consist of milli fluidic channels (1 mm x 1 cm) containing porous sections with a porosity of 70% formed by arrays of randomly shaped pillars. To address the curvature effect, we consider a wavy channel, where porous sections alternate with half-circular channels, and a long straight one. In each case, the matrix and inclusion phases are

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injected in an upstream T-junction, continuously or through periodic pressure pulses respectively. This method allows the production of isolated inclusions, of size typically comparable to the width of the channel.

When flowing through the porous medium, the inclusion stretches and follows preferential paths. At the output of the porous medium the V/N and V/V cases are markedly distinct.

In the first case, the homogenization process is dominated by the creation of filaments which elongate and become thinner along the channel. The length of these filaments can reach several times the length of the porous section; this effect is further amplified in the curved sections of the wavy channel. We believe this amplification relates to the gradient of velocity within the transversal section observed in these areas.

In contrast, the V/V homogenization seems dominated by the fragmentation of the inclusions, although short filaments were also observed. In addition, the fragmented parts were often observed to come back together forming clusters after the porous medium, which suggests that setting several porous sections in series would improve homogenization by ensuring that fragments keep a uniform small size.

The present study reveals the very rich behaviour of heterogeneous viscoelastic solutions within a porous media. It also raises the question of the dominant mechanism at play in the multiphase homogenization process consisting of fragmentation, stretching and folding, or diffusion. Investigations are in progress to quantitatively assess these mechanisms.

Keywords: Homogenization, multiphase flows, porous media

Rheological and morphological investigation of Poly (Lactic Acid)/Polyamide 11 biosourced multiphase systems from blends to multi-Micro-Nanolayers polymers.

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Abstract

The objective of this study is to gain a deep understanding of the Micro-nano-structuration of biosourced polymers multiphase systems based on polylactide (PLA) and polyamide 11 (PA11). Generally, PLA cannot challenge regular commodity polymers due to its weak thermo-mechanical properties and its poor elongation properties.

Firstly, we present a promising route to overcome these drawbacks in order to enhance the processability of PLA blending with other ductile biopolymers such as PA11, as well as mixing PLA/PA11 blends with a chain extender, Joncryl ADR-4368, containing reactive epoxy functions.

To attain our objectives, different blending approaches were used. The effects of those strategies on the morphology and rheology properties were investigated.

Results showed that a "self compatibilization" between PLA and PA11 chains can occur but it was found to be insufficient. The role of Joncryl as a compatibilizer for the PLA/PA11 system has been demonstrated by the significant decrease of particle size and interfacial tension as well as the improvement of ductile properties. Moreover, a new relaxation peak appeared in the relaxation spectrum, indicating the generation of a copolymer at the polymer-polymer interface.

Secondly, the obtained blends were structured by the innovative coextrusion process allowing the in-situ evolution of their morphology by confinement. Hence, the work is in progress to investigate their morphological, rheological and mechanical properties.

Keywords: Rheology, morphology, biosourced polymers, compatibilization, reactive extrusion, co-extrusion.

*Speaker

Data-Driven Modelling of Polyethylene recycling under High Temperature Extrusion

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Abstract

Polyethylenes (high density polyethylene (HDPE) and ultra-high molecular weight polyethylene (UHMWPE)) were extruded in a corotating twin-screw extruder under high temperatures – ($320\text{ °C} < T < 420\text{ °C}$) for various process conditions (flow rate and screw rotation speed). From the in line measurement of the die pressure, a numerical method based on the Carreau-Yasuda model was developed to predict the rheological behaviour (variation of the viscosity versus shear rate). The results were successfully compared to the viscosity measured from off-line measurement assuming the Cox-Merz law. Finally, Machine learning techniques, able to operate in the low-data limit, were tested to build predicting models of the process outputs and material characteristics.

Keywords: Data science, Extrusion, Recycling

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The Influence of Stearic Acid on Rheological Characteristics of Highly Loaded Polypropylene-based Compounds

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Abstract

The Stearic acid (SA) (C₁₈H₃₆O₂) belongs to the family of saturated fatty acids. It is made from a long hydrocarbon chain (18-carbon atoms) representing the a-polar portion, and a carboxyl group placed at the end of carbon-chain, constituting the polar part. This additive is often introduced in several industrial and academic formulations, for ease of traditional polymer and composite processes, or of advanced modern technologies, such as powder injection moulding (PIM) or 3D printing, or for improving structural and functional properties of final products.

In polymer-based systems, its primary usefulness consists in a lubricant effect, by reducing distortions and flow irregularities (processing aid role). In the compounding phase of composites, the involvement of Stearic acid is due to its ability of interacting with mineral, carbon, ceramic or metal surfaces, by creating mono-or multi-layer structures (surface treatment). This leads to modify particle charge, to promote steric hindrance, and increase the hydrophobic character of treated surfaces. In this way, filler/matrix compatibility is improved, the melt viscosity is reduced, and the filler dispersion is increased. The aforesaid benefits become essential in developing highly filled (HF) polymers. In these formulations (solid content > 40 vol.%), the presence of dispersant agent changes the percolation thresholds, and promotes an extension of the processability window of mixtures by determining a decrease of feedstock viscosity.

In this framework, this study was focused on the effect of stearic acid on the rheological behavior of a commercial polypropylene resin, filled with content of Tin particles up to 50 vol/vol.%. Samples were prepared by melt blending in a batch mixer at a temperature of 190°C, and screw speed of 30 rpm for 10 min, and 50 rpm for 3 min. An amount of dispersant agent equal to 6 and 9 vol. % was incorporated in systems at 30 and 50 vol.% of microparticles, respectively.

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Dynamic rheological tests were conducted on developed specimens at temperatures above or below the melting point of Tin microparticles ($\sim 230^{\circ}\text{C}$). In detail, preliminary strain sweep tests were carried out at fixed frequency of 10 rad/sec in range of deformation between 10⁻² and 10² to identify the linear viscoelastic region. Then, frequency sweep tests were conducted at 190°C in range of 0.1 and 100 rad/s. A further rheological characterization, involving cyclic tests at temperatures of 190°C, continued on systems at 30% of particles loadings for highlighting possible differences, ascribed to the SA presence. Other specific investigations were also performed on the same systems at temperatures of 245°C, representing melted state systems both for polymer and particles. The optical microscopic technique has been used for exploring structural changes of composite morphology and the solid-melting transition of metal filler under controlled conditions of temperature and shear rate.

Experimental results confirmed a reduction of linear viscoelastic domain, by increasing filler loadings, with an effect more emphasized in presence of SA. The trend of storage (G') and loss modulus (G''), as a function of angular frequency, allowed to attest liquid-like behavior for composites realized with 30% of Tin particles, by showing a completely overlapping of curves corresponding to systems, with or without SA. For these compositions, it was observed that gelling point was achieved in less time when the SA was added to the feedstock. Solid-like behavior was displayed for systems containing 50 vol/vol % of Tin, with a slope of both curves approaching to zero in the low frequency region. Comparing systems with or without SA, at an equal filler content (50%), both moduli resulted to be superior for formulations containing the dispersing agent. In contrast to previous literature researches, this outcome confirmed a higher propensity in building up 3D filler network in systems containing the fatty species. Calculations to attest the content of SA able to create a monolayer around solid Tin particles allowed to conclude that the used amount of dispersing agent in developed formulations was much over the minimum required. In this situation, the excess of SA remained entrapped with Tin particles by causing the filler aggregation.

Keywords: Stearic acid, Polypropylene, Highly Filled Composites, Rheological Investigation

Rheological characterization of injectable hydrogel based on bioceramic microspheres/ Pluronic (F127+F68)/Water system for bone repair

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Abstract

Bone injuries are serious health problems, and with the increase in life expectancy, these problems will be more aggravated and frequent. In the last decades, many studies have been done in the area of tissue engineering with the aim to develop ideal systems that can promote the regeneration of bone tissue defects. Bioceramic materials (such as bioglass) have been proven to be excellent scaffolds for bone tissue growth. These can be incorporated, along with active cargo (pharmaceutical drugs, growth factors, cells), within a carrier hydrogel that will be implanted, or injected, in the damaged area of the bone, to increase the regeneration of bone tissues.

The injectable hydrogel systems make the administration minimally invasive. These systems behave like a low viscosity liquid at the temperature of the operating room, however they suffer gelation at higher temperatures, close to the body one. To study the sol-gel transition and the injectability of those systems, and relate them with the system composition, helping in its optimization, rheology is a powerful tool

In this work, the rheological characterization of a composite hydrogel based on 20 wt% Pluronic (18 wt% F127+2 wt% F68) filled with bioceramic microspheres (5 and 10 wt%) is presented. The systems have a sol to gel transition between 28 and 30 °C, as determined by SAOS, the presence of the microspheres lowering the transition temperature. The injectability was studied by measuring the steady state viscosity at 18 and 20 °C (about the operating room temperature), the results showing viscosities in the order of 10-2 Pa.s, which turns it perfectly appropriate as injectable systems.

The composite hydrogel based on Pluronic/water/bioceramic microspheres is then a promising injectable system for the regeneration of damaged bone tissue.

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Keywords: Rheology, biomaterials, bone regeneration, thermosensitive hydrogels, composites, ceramic microspheres, injectable systems, drug loading.

Advantages of systemic rheology to measure the rheological behaviour of an evolving system: How to deal with the kinetic instability of a Pickering emulsion ?

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Abstract

Particle stabilized emulsions, otherwise known as Pickering emulsions, are claimed to be very stable against coalescence. As a consequence, this behaviour has not only drawn the scientists' attention but it has also promoted the development of new products. So Pickering emulsions appear as an interesting alternative in comparison to the classic surfactant stabilized emulsions.

In order to understand the stabilisation mechanisms of Pickering emulsions, a rheological approach is chosen in this work. Information as the distribution of solid particles between the continuous and dispersed phases, the interactions of the dispersed phase drops and the evolution of the drop size distribution can be obtained *via* rheological measurements. These data will lead to the optimisation of the formulation and production process of Pickering emulsions. However, the rheological characterisations of these emulsions can be complicated because of the physico-chemical complexity of this system, *i.e.* the presence of one aqueous dispersion of solid particles and one oily phase. Actually, the difference in density of these phases promotes their demixing during the rheological tests, specially *via* the creaming of the dispersed phase, whose mean drop diameter (d_{50}) is over 10 μm . This phenomena lead to artefacts during measurements. As a result, the rheological characterisations of these systems are a current challenge.

This work aims to exploit systemic rheology, which is a tool developed in our laboratory, for the study of this complex systems. The main advantage of systemic rheology lies in the fact that the rheological mesures can be made under non-conventional conditions. In order to do this, a helical ribbon is introduced into a cylindrical tank, the whole system constituting a "rheo-reactor". Therefore, the geometry of the mixing tool favours the suspension of the dispersed phase drops to avoid the creaming during the measurement procedure. In this work, nanosilica stabilized emulsions of 20 % and 50 % of volume fraction of paraffin oil (Φ_d) were made. Firstly, the emulsions were studied by means of a classic "parallel plate" geometry and afterwards with the rheo-reactor. Figure 1 shows that, while the results obtained from the classic configuration are not repeatable for the same system (*e.g.* at $\Phi_d = 20\%$), those

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obtained from the rheo-reactor exhibit the same behaviour for the same system. Moreover, a shear thinning behaviour of the emulsions is observed in the rheo-reactor measurements, which is expected for this kind of system. Considering these first encouraging results, the rheo-reactor is going to be used to follow up *in situ* the elaboration process of Pickering emulsions.

Keywords: Pickering emulsion, paraffin oil, nanoparticles, silica, systemic rheology, rheo, reactor, rheological behaviour, kinetic stability

Recycling opaque and colored PET bottles into fibers by spunbond process

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Abstract

The recycling of PET packaging is increasingly competitive due to new environmental standards, imposing recyclers to accept poor quality PET matrix in their process with more and more additives, fillers, and reduced molar masses. In particular, the introduction of opaque PET containing particles can impact the PET recycling into fibers. Actually, the manufacturing of PET fiber by continuous high speed melt spinning requires certain rheological properties, in shear and in extension, and stability at high temperature. The rheological behavior of opaque PET and the influence of have been studied to assess the spinnability of these new products before incorporating it in the production line.

Different opaque bottles grades have been characterized and compared to transparent PET bottles. Virgin PET blends with different amount of have been formulated and characterized to study the influence of on rheological behavior. The Intrinsic viscosity, the shear complex viscosity and the evolution of zero shear viscosity with processing time have been measured with an Ubbelohde viscometer and ARES G2 dynamic rheometer respectively. A capillary rheometer with a spinning system has been used to study the spinnability of the different samples via the measurement of the melt strength and the maximal draw ratio before breaking.

It have been observed that incorporated in virgin PET have minor influences at low concentration (< 5%wt) on molar mass, thermal degradation and spinnability. However, Opaque PET samples have lower molar masses, higher thermal degradations and lower spinnability than transparent PET. This is due to the increase of Newtonian viscosity with filler content, involving the bottle manufacturer to reduce the PET mass molar to conserve the same range of MFI. The high photo-activity of is also responsible of reducing the molar mass of PET during the lifetime of the product.

Keywords: PET, recycling, melt spinning, capillary rheometer

*Speaker

RHEOLOGICAL BEHAVIOR OF NANOFLUIDS: INFLUENCE OF SILICA NANOPARTICLES IN XANTHAN GUM SOLUTIONS

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Abstract

Xanthan gum (XG) is a polymeric hydrocolloid widely used as a thickener and gelling agent for different purposes in petroleum, food, cosmetics and other industries. Many of these applications require a polymeric fluid of high viscosity dissolved in brine, usually of NaCl. However, the changes in conformational structure of the XG macromolecules in a buffered ionic solution such as NaCl directly impact the rheological behavior of aqueous solutions. In particular, the dissolution of XG in ionically charged environments decreases the polymer solubilization process by reducing intramolecular repulsion and subsequently reducing the viscosity of the solution.

In the present study, the incorporation of charged surface particles such as hydrophilic silica nanoparticles (NpSiO₂) is proposed to counteract this phenomenon. This incorporation is expected to improve the rheological behavior of these polymer solutions by increasing their viscosity in monovalent ion environments (NaCl).

Initially, the rheological behavior of XG solutions and suspensions of silica nanoparticles at different salinities was characterized considering variations in concentration, particle size, dispersion and suspended agglomerates. Consecutively, different protocols for the elaboration of the XG-NpSiO₂-NaCl solutions were evaluated, in order to select the best process to obtain a satisfying dispersion of the nanoparticles in the solutions as well as a substantial viscosity increase. The following operating parameters were varied: the order of introduction of the components, the hydration time of the polymer, the duration of ultrasound application and the mixing time. Finally, the influence of the concentration of different the components (XG, NpSiO₂ and NaCl), was investigated.

Various characterization techniques were used such as Dynamic Light Scattering (DLS), electrophoretic measurements of Zeta potential and rheological characterization with a Rheometrics Scientific RFS II rheometer equipped with a helical ribbon geometry at variable shear rate and constant temperature (25°C).

*Speaker

A conventional one-factor-at-a-time was applied obtaining comparative rheograms that allowed the characterization of nanofluids at each stage of their elaboration. The results evidenced the immediate degradation of the polymer due to ultrasonic effects, as well as the influence of nanoparticles and their dispersed aggregates in ionically charged environments, generating a significant effect on changes in conformational structure of XG in solution. This study provides a better understanding of the interactions between XG and silica nanoparticles in solution, through the rheological behavior of fluids at low concentrations.

Keywords: Xanthan gum, Silica nanoparticles, Rheological behavior, Nanofluids, Dispersion processes

RHEOLOGY OF NON-BROWNIAN DENSE SUSPENSIONS: AN OPTIMISED IMPLEMENTATION OF THE FICTITIOUS DOMAIN METHOD IN OPENFOAM

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Abstract

Non-Brownian dense suspensions of particles in low-Reynolds-number flows are ubiquitous in industrial as well as in biological or natural flows (e.g. fresh concrete, rocket fuel, blood, mud or lava). These suspensions exhibit a complex rheological behaviour that is far from a complete understanding. Complexity mainly stems from the wide variety of fluid-particle or particle-particle interactions (Brownian, hydrodynamic, frictional or collisional) as well as from the physical properties of particles (roughness, shape, size distribution, etc.). Even idealised cases involve strong non-Newtonian effects such as shear-thinning, shear-thickening, particle migration and anisotropic normal stresses and microstructures. Numerical simulations can help shed light on the intricate physics of suspensions at the particle scale.

Due to the importance of flow-particle interactions, particle-scale numerical methods are of primary interest. For low-Reynolds-number suspensions, different techniques have been previously developed; some of them are highly specialised and may not tackle any kind of flow or fluid. Therefore, direct numerical simulations (DNS) have emerged as an attractive alternative, allowing to solve particulate flows with arbitrary particle shape, Reynolds number or rheological constitutive equation. The very first class of DNS methods dedicated to particulate flows followed a boundary-fitted approach wherein only the domain occupied by the fluid is meshed. For sheared concentrated suspensions, in which particle separation can be vanishingly small, remeshing becomes extremely involved and makes this approach impractical for more than a few particles. In contrast, non-boundary-fitted methods are much more suited for the simulation of suspensions with a high number of particles: the whole domain is mapped onto an Eulerian fixed grid and particles are embedded in this regular non-moving mesh. These techniques include different methods, such as the immersed boundary method, the lattice Boltzmann method or the fictitious domain method, being the latter considered in the present work.

In the fictitious domain method, particles are modelled via a body-force introduced in the momentum equation to enforce a rigid body motion. For the sake of computational efficiency,

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the body-force quantities are defined in the present approach at the Eulerian grid points and an advection step is inserted into the method, so that the body-force remains attached to the particles as they move along.

A peculiar feature of concentrated suspensions is that the average separation distance between particles becomes extremely small. The so-called lubrication forces arise between particles in near-contact because of the draining of interstitial fluid in the gap, and it is singular in the limit of touching particles. Consequently, the rheology of suspensions is modified by lubrication forces and important effects occur. Keeping the grid spacing high enough for large scale simulations while accounting for the low-scale lubrication effects requires the modelling of the latter. A last essential ingredient for accurate simulations of non-Brownian suspensions is the modelling of contact interactions between particles, as contacts inevitably occur despite lubrication because of particle roughness.

The equations are split into two sub-problems. The fluid sub-problem is a standard Navier-Stokes problem whereas the particle sub-problem mainly consists in enforcing the rigid body motion inside the particles and in coupling the interaction and the sub-grid forces to the flow. These coupled sub-problems are solved using the algorithm described by Gallier et al. (2014).

In the present study, the fluid sub-problem is solved using the open-source finite volume library OpenFOAM on an isotropic Cartesian mesh with a constant grid spacing. The idea is to take benefit from the parallelisation facility available in OpenFOAM to make the simulation of large systems with a high amount of particles possible. The algorithm described by Gallier et al. (2014) is adapted to work under the OpenFOAM environment, paying particular attention to the optimisation of the code. Different classical particle configurations are studied in order to validate the method.

Keywords: rheology, suspensions, numerics, openfoam, cfd

Rheological behavior of concentrated phosphate slurry

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Abstract

Phosphate ore slurry is a suspension of insoluble particles of phosphate rock, the primary raw material for fertilizer and phosphoric acid, in a continuous phase of water. This suspension has a non-Newtonian flow behaviour and may exhibit a yield stress as the shear rate tends to zero. The suspended particles in the present study are assumed to be non-colloidal. The knowledge of rheological properties of concentrated phosphate slurry is essential for simulations, calculations in engineering processes and designing transport systems. Various grades and phosphate ore concentrations are used for this rheological study. We established some experimental protocols adapted to the determination of the main characteristics of these complex fluids, to establish relevant rheological models with a view to simulation numerical flow in a cylindrical pipeline. Rheograms of these slurries were obtained using a Anton Paar RheolabQC rotational rheometer and modelled using commonly used yield pseudoplastic models. The effect of concentration on the slurry is described through the changes in apparent viscosity at a given shear rate. For the concentrated phosphate slurry, yield stress and apparent viscosity follow an increasing power law with the concentration. It is also discussed how the effect of particle size distribution can be interpreted in the context of highly concentrated slurries. The results show that solids concentration in a solid-liquid mixture can be increased while maintaining a desired apparent viscosity.

Keywords: Rheology, Non, Newtonian, Suspensions, viscosity, Polydispersity

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Rheology, dynamics and morphological investigations of PLA, PHBV and their blends: A suitable tools to probe miscibility and thermal stability

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Abstract

The purpose of the present work is to gain a true and fundamental understanding of thermal stability and miscibility of PLA/PHBV biopolymer blends before their coextrusion in multi-micro/nanolayered nanostructured films for ultra-high barrier applications. Owing their lower differences of solubility parameter values, good miscibility between both biopolymers should be thermodynamically expected. However, the actual miscibility between PLA and PHB is also dependent on the processing temperature, the proportion of each polymer in the final blend as well as their molecular weight. To attain our objective, Polylactide (PLA) and poly(hydroxybutyrate-co-hydroxyvalerate) (PHBV) blends were prepared at different compositions by melt mixing. Their thermal properties as well their molar masses and polydispersity were subsequently measured. Small-amplitude oscillatory shear rheology (SAOS) and melt dielectric relaxation spectroscopy (DRS) properties were assessed. Hitherto, the composition dependence of thermorheological complexity of PLA/PHB-V blends in the melt was revealed. The molecular entanglement state involving intra- and interchain entanglements was found to support the scenario of thermorheological complexity and dynamic heterogeneity with composition dependence. Besides and a part PLA or PHB-rich blends, the obtained two experimental glass transition temperatures of blends were resolved in view of segment motions in the miscible phase. only one T_g can be observed. This miscibility suffer also from the lower thermal stability of PLA and especially PHBV. Hence, the melt-induced degradation process of PLA/PHB blends seems to be a responsible of some in-situ self-compatibilization mechanisms which influence the crystallization properties. Lastly, effects of multifunctional epoxide on melt rheological properties and dynamics are investigated. This study will guide a better understanding of interfacial phenomena and in-situ nanostructuring of PLA/PHBV blends in multilayer coextrusion.

Keywords: Rheology, miscibility, dynamic, morphology, nanostructured biopolymer films

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Relaxation Mechanisms at long times of Crosslinked EPDM: Influence of the Chemistry and Nanofillers

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Abstract

The objective of this work was to study the elastic recovery of EPDM samples crosslinked either by a phenolic resin (resol) or by a radical peroxide (dicumyl peroxide). From compression set experiments, it was observed that radically crosslinked EPDMs have better elastic recovery properties. On the other hand, for the same crosslinking density, radically crosslinked EPDM shows better compression set than EPDM crosslinked with phenolic resins. The Chasset-Thirion equation was then used to successfully fit the experimental relaxation curves. As a notable result, the preferential statistics of a peroxide-crosslinked network over a phenolic resin (resol) showed that better elastic recovery properties were obtained. Finally, the influence of fillers (carbon black and silica) was also studied. Carbon black with DCP crosslinking was shown to improve elasticity recovery whereas silica fillers lead to worse properties. It was then assumed that the interaction between particle surface and crosslinking agents induced crosslinking gradients in the inter-particle volume.

Keywords: compression set, EPDM, crosslinking, relaxation mechanisms

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Extensional rheological behaviour of polyolefin based nanocomposites exhibiting yield stress

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Abstract

Rheological properties play an important role during plastic processing. In the context of thermoforming a product for the automotive industry, the rheological properties of an industrial grade elastomer are optimised. The elastomer in this case is EPDM (Ethylene-Propylene Diene Monomer), a thermoplastic-polyolefin (TPO) offering significant advantages such as lightweight, low-cost and ease of processing. To obtain specific shear-rheological properties, we reinforce EPDM with a nano-scale fractal filler. It is well documented in literature that the extensional behaviour of filled molten polymer systems exhibiting a yield stress is primarily strain softening. Such a behaviour poses challenges for products which need to be thermoformed where an extensional strain-hardening behaviour is preferred. The extensional behaviour of EPDM is identified to be linear i.e. neither strain-hardening nor strain-softening. Addition of nano-scale fractal filler at varying concentrations leads to a strain-softening behaviour. In order to improve the processability for thermoforming, the EPDM-nanocomposites are cross-linked via an electron beam. This results in a complex extensional behaviour where strain softening is observed at low strains followed by a hardening behaviour due to an elastic response at large strain. We present our work on the effect of cross-linking on the rheological behaviour of EPDM-nanocomposites. Additionally, we discuss the validity of changing hydrodynamic reinforcement during deformation in contrast to the classical Leonov model for molten filled polymer systems involving free and trapped chains.

Keywords: Extensional rheology, Polymer nanocomposites, Rheology of filled polymer melts, Strain hardening, Strain softening, Electron beam modification, Fractal fillers, Yield stress fluids

*Speaker

Preliminary Rheological Study of Highly Loaded Composites by Magneto-Caloric Powder for Additive Manufacturing Applications

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Abstract

Global warming, over-consumption of energy and noise pollution are encountered problems with current and conventional refrigeration systems [1-2]. An innovative and promising alternative is so-called magnetocaloric refrigeration [3]. This technology is based upon of magnetocaloric effects produced by magnetocaloric regenerators (MR). Several MR prototypes have been realized and actually very few have been marketed. Indeed, RMs exploited so far have limitations [4]. The challenge, in this topic, is to obtain functional regenerators which must operate under various combined stresses of mechanical, magnetic and thermal fields. One solution is to manufacture MRs by extrusion or injection-moulding processes with high loaded feedstocks with La(FeSi)₁₃ magnetocaloric have been previously investigated [5-6]. In this current work, the polymer forming of the micro-composites (feedstocks) based on polymers (PLA + EVA + SA) dedicated to additive manufacturing with highly loaded polymer (46 to 50% by volume) of magnetocaloric powder La(FeSi)₁₃ have been investigated and analysed. Continuously compounding by twin screw extruder and full-filament 3D deposit have been studied. During the elaboration process, focus will be placed on the powders fluidity properties studied by powder rheometer, mixture polymer/powder homogeneity characterized at the twin-screw mixer and feedstocks rheological characterization (under shear rates ranging from 102-104 /s) by capillary rheometer have been analysed. DSC (differential scanning calorimetry) and VSM (vibrating-sample magnetometer) analyses will performed to verify the influence of each elaboration process on magnetic properties of magnetocaloric plates. References: X. Hou et al. / Journal of Alloys and Compounds. 646 (2015) 503-511

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Keywords: Micro, composites, Loaded polymer, Rheology, Mixing, Magnetocaloric properties, full, filament 3D deposit, additive manufacturing

Investigating asphalt modified with polyurethane: morphology and rheology relationships

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Abstract

Bitumen materials are widely used for waterproofing and road pavement applications. Since more than 40 years, these ones are modified with polymers in order to increase their thermo-mechanical properties over a wide range of temperatures, i.e. to reduce rutting and cracking. Polymers commonly used to modify bitumen are thermoplastic elastomers such as poly(styrene-*b*-butadiene-*b*-styrene) block copolymers (SBS) due to their good compatibility with bitumen and specific interactions with the different chemical species (SARA fractions – Saturated, Aliphatic, Resins, and Asphaltenes-). Nevertheless, SBS polymers display poor resistance to UV and therefore give poor aging resistance to the polymer-modified bitumen materials.

To overcome these drawbacks, thermoplastic polyurethanes (TPU) are considered in the present study due to their improved durability and better resistance to UV compared to SBS. Furthermore, the architecture and microphase-separated morphology of the thermoplastic polyurethanes which controlled the miscibility and interactions with the bitumen can be easily tuned from the polymerization step by a proper copolymerization of the nature of the soft and hard segments. The aim of this work is to study the relationships between the physico-chemical interactions between the TPU and the bitumen fractions, multi-scale microstructures, and rheological properties in thermoplastic polyurethane/bitumen blends, from rheological and surface tension measurements, fluorescence microscopy, and transmission (TEM), and scanning electron microscopies (SEM). Thus, several parameters were considered for the TPU/bitumen blends, such as the amount of TPU and the bitumen composition, i.e. bitumen differing from their SARA fractions (50/70 and 160/220 grades).

The increase in TPU content leads to an increase of the storage shear modulus of the

*Speaker

TPU/bitumen blends in the investigated temperature range. In order to describe G' dependence with TPU content of the blends, the Takayanagi's model which considers a percolation threshold, i.e. a phase inversion change from a TPU rich dispersed phase in a bitumen rich continuum for low TPU contents to a bitumen-rich dispersed phase in a TPU-rich matrix. As the model fits the TPU-content dependence of G' of 160/220 bitumen-based blends, one can conclude that the compositions of the two co-existing phases remain the same whatever the TPU content and that phase inversion occurs for 10% of TPU. A different behavior could be evidenced for 50/70 bitumen-based blends, i.e. the phase inversion occurs for a lower TPU content, and the compositions of the phases change due to specific interactions of the TPU with some fractions of the bitumen according to the discrepancy between experimental data and Takayanagi's model. Thus, this work demonstrates that rheology is a powerful technique for investigating the interactions between SARA fractions of bitumen with polymer chains in polymer-modified bitumen blends.

Keywords: Asphalt, polymer blends, rheology

Synthesis and rheological investigation of aqueous dispersions of graphene oxide

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Abstract

Graphene oxide (GO), a precursor for graphene, possesses abundant negatively charged oxygen functional groups such as hydroxyl, epoxy, carbonyl, carboxyl groups on their basal planes and edges, leading to the good dispersion and stable colloidal suspension in water or organic solvents through stirring or ultrasonication. Meanwhile, these oxygen-containing groups make it possible to have strong interactions between polar small molecular or polymers and sheet-like GO to prepare GO-based composites with many applications. In order to better understand the mechanical properties of aqueous GO dispersion, rheological studies can serve as a powerful tool for mechanical characterization by determining the relationship between microstructure and macrostructure.

In this study, the aqueous GO dispersions were firstly synthesized by modified Hummer's method. Then, X-ray diffraction (XRD) measurements and X-ray photoelectron spectroscopy (XPS) measurements were performed in order to investigate the chemical properties of GO. Simultaneously, scanning electron microscope (SEM) observations were applied to see the physical structure of GO and Zeta potential characterization of GO dispersions was carried out by dynamic light scattering (DLS) technique. The rheological measurements of GO dispersions at different concentrations (from 0.2 to 16 mg/mL) were performed, including oscillatory shear strain, transient and steady shear flow.

Based on the obtained results, from the XRD patterns, the peak (002) and interlayer spacing of graphite respectively shift from 26.65° to 9.39° and from 3.3 Å to 9.4 Å after oxidation and exfoliation. In XPS measurements, survey spectrum clearly shows the elements of carbon and oxygen and the carbon oxygen ratio is 2.1:1. The C 1s XPS signal in GO indicates the presence of carbon sp² (C=C, 284.3 eV), hydroxyls (C-OH, 285.3 eV), carbonyl (C=O, 287.3 eV) and carboxylates (O-C=O, 288.9 eV). Due to these functional groups, Van der Waals forces and electrostatic repulsive forces are altered, contributing to the stable aqueous dispersion without occurring precipitation in several months. The Zeta potential deduced from DLS measurements of graphite is -1.91 mV (85.2% area) and 14.1 mV (14.8% area), and that of GO is -60.9 mV (100% area) and maintain at -60.3 mV (100% area) after 2 months. In addition, the morphologies of graphite and as-prepared GO are obtained by SEM observations. Compared with graphite (about 100 μm width and 50 μm length), the size of GO (about 20 μm width and 10 μm length) decreases significantly, indicating that GO has been exfoliated successfully as single or several layers, some of them fold to induce wrinkles. Additionally, GO dispersions exhibited, shear-thinning behavior with an apparent

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yield stress, fitted well with Herschel-Bulkley model, and the transient viscosity reveals the time-dependent response, strongly depending on GO concentration and shear rate. More importantly, the increasing GO concentration contribute to the formation of a strong and highly ordered structure because GO sheets tend to organize themselves at higher concentration.

Finally, the rheological behavior of GO dispersions, investigated in detail as a model system, can provide a basis of the elaboration and study of GO-based composites in the future.

Keywords: graphene oxide, sheet like fillers, aqueous graphene oxide dispersion, viscosity, viscoelastic moduli

Effect of geometry on measured rheological properties of zeolite-based pastes

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Abstract

Zeolites are porous materials found in nature and synthetically obtained with multiple dimensional structures. Physicochemical properties such as cation exchange and adsorption capacities and their industrial applications as ion exchangers, molecular sieves and catalysts depend on channels and cavities structural interconnection. For industrial use, zeolites have to be conformed from pastes formulated specifically to obtain a desired performance. Various investigations have been developed in order to establish scientific and technical bases for the formulation of conformed products. For this work the pastes were composed of zeolite A, attapulgite, carboxymethyl cellulose (CMC), and water. Rheological properties of the obtained pastes have been measured using a rotational rheometer with rough parallel plates (PP25 and 40 mm-diameter plate) and four-bladed vane tool (diameter: 25 mm, height: 55 mm). Three different gaps between plates were used (2, 1.5 and 1 mm) to simulate the range of aggregate spacing in zeolite-based pastes. The measured oscillatory rheological properties were significantly affected by the test geometry used and the friction of and gap between its shearing surfaces. Additionally, the yield stress was determined for the measurements obtained with both geometries; which was corroborated with the help of an endoscopic camera that allowed to have a direct observation of the flow of the paste during the test. From the results obtained, it can be seen that the strain and viscosity depend on the gap or the sample thickness, solids, binder and humidity concentration, and friction capability of the shearing wall along with the flow geometry tested.

Keywords: Zeolites, pastes, yield stress

*Speaker

Rheological behavior of hydrothermal conversion products from micro-algae: bio-bitumen mimicking road binders

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Abstract

Road binders are refined petroleum products. They are mainly used in the road construction field. With nearly 100 million tons used per year worldwide, rarefaction of petroleum as well as changes in refinery strategies are expected to bitumen shortages in the coming years (indeed temporary shortages have already been observed in France).

This coming crisis' context has led the Algoroute project funded by the French National Agency for Research (ANR), whose first goal is to find alternative road binders from renewable resources. Trying to mimic the geological process of petroleum formation in a faster way, the Algoroute project focus on hydrothermal liquefaction (HTL) process of micro-algae (*Spirulina*). This process is inspired by the transformation of sedimented micro-algae biomass in oceans' depths into petroleum by a slow increase of temperature and pressure.

The main product of this transformation is a black hydrophobic phase. This hydrophobic phase could be compared to road binders as it has the same unique combination of properties: adhesion, impermeability to water, and specific thermo rheological behavior. Rheological characterization is important, to establish the quality of a hydrophobic phase as road binder. Thereby, Black diagrams are plotted, and they consist of plotting the phase angle versus the complex modulus. Road binders of many kinds have a typical signature in Black diagram as shown in Figure 1. It is also possible to characterize road binders with Cole-Cole diagrams and isochrones curves.

Preliminary characterizations by DSC and XRD have shown the oil phase presents a slight (a few percent) and slow crystallization. This crystalline phase melt at a nearly temperature of 40°C which truly affect the rheological response. DSC and XRD tests were focused on the kinetic of recrystallization, and have pointed out the crystal reorganization takes place from the moment the melted crystals are cooled down to room temperature, and up to 2 weeks

*Speaker

more or less.

This reorganization is a real problem when it comes to discuss the rheological behavior of the hydrophobic phase. To get rid of this problem, and to avoid the obligation of controlling the storage time, the French norm NF 14770 was followed. This norm is used to determine complex modulus and phase angle of bitumen and road binders with little deformations rheological test, and it advocates heating up at 80-85°C the sample for 2 hours before testing. Following this norm, the thermal history of the sample is sure to be erased. Then, this study focuses on the hydrophobic phases obtained after a hydrothermal liquefaction at 260°C, and investigates the impact of reaction time, heating and cooling rates.

The aim of this communication is to better understand the rheological response of the hydrophobic phases obtained by HTL, according to their composition. Indeed, this hydrophobic phase can be described in first approximation as a suspension of solid residues in oily phase (Figure 2).

The main results so far, are the solid residues have a huge impact on the rheological response. The hydrophobic phase has a similar rheological response as elastomeric-added bitumen while the oil phase mimics conventional bitumen (Figure 3). The oil phase is a solid product at low temperature, with a high complex modulus, and as the temperature rises, the complex modulus decreases, and the phase angle increases to the value of 90°, which indicates the fluid behavior. But, the hydrophobic phase (Oil and solid residues together) has another behavior at high temperature. It's still a solid at low temperature, and when the temperature rises, the complex modulus decreases and the phase angle increases. But, at high temperature, the phase angle starts to decrease to return to low values near 40° which can point out hydrophobic phase has a solid behavior at high temperature.

This bell-shaped curve can be explained by the solid residues in suspension in the oil phase which collide together at high temperature and provide the visco-elastic behavior at high temperature. But the real impact of solid residues is still uncertain. Their composition, in terms of organic and inorganic parts, their volumic fraction in the oil matrix, their size and shape are the main scientific locks to better understand and analyse the rheological data. Tricky locks to resolve because of the product's dark color, making visual observations impossible.

Keywords: Hydrothermal Liquefaction, Bitumen, Micro, algae

Transient and steady shear flow of marginally entangled ring polymer melts through nonequilibrium molecular dynamics simulations

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Abstract

Atomistic simulations of ring polymer melts under equilibrium conditions have been proven very useful in extracting scaling laws for the conformational and transport properties (chain center-of-mass diffusion coefficient and zero-shear-rate viscosity) of this unique class of polymers in the crossover regime around the entanglement molecular weight Me [1]. In combination with a detailed geometric analysis [2,3], such simulations have also helped tremendously characterize topological constraints in ring polymers developing due to ring-ring but also ring-linear threading (in the presence of any remaining linear impurities) and their connection with the slow relaxation modes observed experimentally [4].

In the present work, we extend this study to nonequilibrium conditions by investigating in a systematic way the transient and the steady-state flow behavior of ring polymers using as a model system poly(ethylene oxide) (PEO). We have conducted detailed nonequilibrium molecular dynamics (NEMD) simulations of pure ring and linear PEO melts [5], as well as of ring-linear blends, over a wide range of shear rates covering both the linear and nonlinear regime using the p-SLLOD equations of motion [6]. Through these simulations, we have been able to compute the relevant viscometric functions in shear (the dynamic viscosity, the steady-state shear viscosity, the relative maximum viscosities with respect to the steady-state value, the strains at the peak viscosities and the first and second normal stress coefficients) and their dependence on applied shear rate, melt molecular weight and relative concentration of the melt in ring and linear chains. In addition, selected configurations from the NEMD trajectories have been subjected to a detailed geometric analysis [2,3] to provide information on the effect of flow on the degree of ring-ring and ring-linear threading, and thus on the nature of topological constraints in melts of non-concatenated ring polymers, given the tendency of linear chains to penetrate rings and form a network.

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Keywords: ring polymers, shear flow, molecular dynamics, nonequilibrium simulation, polymer rheology

Elasticity and plasticity of highly concentrated suspensions in shearing and squeezing

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Abstract

Suspensions are one of the first and permanent objects of interest for rheology. They have been the object of numerous theoretical and empirical studies generalizing the simplest Einstein case by taking into account the effects of concentration, the shape of solid particles, interactions between particles and/or continuous liquid mediums of a different nature. Among other things, highly concentrated suspensions are the basic materials for powder injection molding which currently plays a significant role in the production of metal and ceramic parts with complex and unusual shape.

An object for this study is model systems which are suspensions of aluminum (Al) powder with varying concentrations of a solid phase dispersed in low-molecular weight ($M=400$) poly(ethylene glycol) as a binder. The average size of Al particles is 24 μm and their density is 2700 kg/m^3 . This model of feedstock melt is aimed to investigate the rheological properties of materials for powder injection molding.

The evolution of rheological properties from a Newtonian liquid to an elasto-plastic solid medium along with increasing concentration of dispersed particles was observed. At low concentrations, the suspensions are Newtonian liquids. Then we meet with non-Newtonian flow, then with yielding and viscoplastic behavior. The elasticity of the suspensions can be detected at concentrations of the order of 55%. Finally, beyond some concentration threshold highly concentrated suspensions (HCS), the gelation or colloidal glass transition takes place. The concentration range of HCS somewhat below this limit is of special interest for powder injection molding technology.

Investigation of the elastoplastic behavior of concentrated suspensions under the uniaxial compression (squeezing) showed that increasing concentration leads to strong increases in elasticity comparing with plasticity. The elastic modulus in compression increases with an increase in the normal stress. The shear modulus measured for samples under compression also increases with an increase in pressure. Meanwhile, the dependence of the shear modulus on the shear stress is very slight in the absence of pressure; but at high pressures, the shear modulus sharply decreases (by several orders) along with an increase in shear stress. A mechanical model consisting of an elastic spring connected with friction element of a varying area of contact between movable weight and a solid surface has been constructed for illustrating the behavior of HCS.

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The experimental results can be presented in the form of a stress-concentration diagram reflecting different types of the rheological states of suspensions.
The studies were supported by the Russian Science Foundation, project no. 17-79-30108.

Keywords: suspensions, viscoelasticity, elastic modulus, powder injection molding, plasticity

SPREADING OF FLUIDS WITH BLADE COATING, MODEL AND EXPERIMENT

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Abstract

The spreading of fluids is a crucial process for industries, such as cosmetic field, food industry and construction industry. Most of the spread liquids are non-Newtonian. However, the parameters that govern the deposit in some usual configurations are not well understood even in the case of simple Newtonian fluids. The consequence is, that for some applications, the material is hard to spread or forms an inhomogeneous film, issues that need to be tackled. This subject is of great interest for the cosmetic field which uses complex fluids (i.e. polymers, emulsions, suspensions) for moisturizers or gels. It is a challenge to mimic by a mechanical system the gesture followed by the consumers. A promising technique is blade coating with a soft and deformable blade that compares better to real applications (Supplementary data 1). A pioneering study was first conducted by Seiwert *et al.* [1], who studied the spreading of Newtonian fluids experimentally and theoretically, with an infinite reservoir of liquid. In their model, a key parameter is the dynamic length, which corresponds to the length of the blade deformed by the fluid. More recent studies, carried out in our group on complex fluids [2], reveal that another parameter should be considered, which is the *wetting length* lc , defined as the length of contact between the soft blade and the fluid measured along the spreading movement x , as shown in Supplementary data 1. Our work shows that the wetting length is essential to explain the dynamics of the spreading of Newtonian fluids, and its impact on the thickness of the fluid layer.

The experiment consists in spreading Newtonian fluids with a flexible blade with a one-way movement. The blade is held at a specific height so that its end is just tangent to the surface [1]. The changes in the wetting length during spreading are followed using an optical camera and the thickness of the liquid layer is measured using an optical profilometer. Such experimental conditions aimed at correlating the thickness of the film e to the wetting length of the blade lc . They show a clear dependency between the fluid thickness and the wetting length, independently on the way the liquid is spread: the relation between e and lc is nearly

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linear (Supplementary data 2). Similar results were obtained with different viscosities or using glycerol.

A model was developed by matching the forces exerted on the blade at the boundary between two regions: the wetted zone of the blade and the dry zone. The wetting length lc thus appears naturally in the film thickness expression and the model is in good agreement with the experimental datas (Supplementary data 2).

The present work shows that spreading with a blade cannot be directly compared with the "meniscus" spreading performed in Landau-Levich situations [1]. In particular, the analogy cannot be made when there is a finite reservoir of fluid, for which the distinction between static and dynamic regions is no longer relevant. A new parameter, the wetting length lc , fully describes the system. Our effort is now focused on the numerical resolution of the model, to determine the prefactor of the scaling law. It is also planned to expand the model to yield stress fluids such as carbopol based systems.

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Keywords: blade coating, soft blade, wetting length, film thickness, Newtonian fluids

Start-up shear experiments investigating the nonlinear shear rheology of model glutes at different glutenin to gliadin ratios

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Abstract

Gluten is one of the most important protein source present in the human daily food. It is a protein network composed of two main species: Polymeric glutenin with very high molecular weight, responsible for the strength and the elasticity of gluten doughs and monomeric gliadin, that is at the origin of the dough viscosity and extensibility. The protein network is governed by intermolecular disulfide bonds between glutenin subunits and hydrogen bonding.

Unveiling the peculiar contribution of each of these species to nonlinear mechanical properties of gluten dough is of a great importance to deepen our understanding of the dough behavior under extreme mechanical solicitations imposed during kneading.

Here, we present preliminary results of a comparative nonlinear rheological investigation of model glutes in water having different glutenin to gliadin ratio "R". We used start-up shear and relaxation protocol at various rates to access the largest possible time and length scales window of gluten gels' viscoelastic spectrum.

By analyzing the transient regime, we figure out that the gluten gels exhibit nonlinear properties lying between polymers and critical gels characteristics. The weak like-damping, for

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example, is attributed to the high functionality of the fractal structure in critical gels. The stress overshoot occurs at strains higher than 2.6 SU which suggests strong stretching of the chains, most probably due to the strength of disulfides bonds.

The relaxation after shear cessation supports the transient findings and suggests localized relaxation modes as it was observed for other polymeric systems with different architectures.

Changing the glutenin to gliadin ratio allows to promote one of the above behaviors. Indeed, higher ratio pushes the properties in the critical gel domain and, in contrast, lower ratio obeys more to polymeric behaviors. In fact, we already have structural evidence that, in dilute good solvent, gluten gels with high 'R' form bigger supramolecular aggregates compared to lower ratios, which resemble very much to the critical gel structure.

Consequently, coupling rheology with structural (X-ray and light scattering) and spectroscopic (RAMAN, IR) techniques will become a crucial task to directly verify the above findings, thus, completing molecular and structural knowledges with gluten doughs mechanical properties.

Keywords: Non linear rheology, Model gluten, Start up shear

Following the evolution of carbon nanotubes network under strain in polymer nanocomposites using simultaneous electrical and rheological measurements.

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Abstract

In the case of polymer composite filled with carbon nanotubes (CNTs), electrical conductivity can be theoretically obtained at content lower than 1 wt. % [1]. This percolated network can be used in industrial applications as protection from electrostatic discharge. Hence, CNTs filled materials can advantageously replace polymer-carbon black composites which contain usually around 20 wt. % of fillers. However, during forming process, the structure created by CNTs connections is strongly modified by the applied stress often resulting in an insulating final product.

To understand the network evolution during process (for example extrusion), a rotational rheometer was used while the conductivity of the sample was monitored. This set-up allowed the observation of both destruction and establishment of CNTs connections during the shear. Under specific conditions, a dynamic equilibrium reports on comparable strength of these mechanisms. The structuration kinetics of the system under shear and in the quiescent melt has been studied to determine key parameters for the conducting network modification. Moreover, in case of semi-crystalline polymer, the percolated network may be disrupted or at least modified, during the cooling. Thus, the effect of crystallization has been followed showing a strong correlation between the conductivity obtained in solid state and this previously measured in the melt state. Consequently, the final conductivity reflects the global thermo-mechanical history of the composite material.

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Keywords: carbon nanotubes, percolation threshold, electrical conductivity, viscoelasticity

*Speaker

Rheological characterization of bronchial mucus: the role of heterogeneity

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Abstract

Secretion and accumulation of abnormal mucus is the hallmark of many respiratory diseases such as Cystic Fibrosis (CF), Asthma or Chronic Obstructive Respiratory Disease (COPD). In CF, mucus accumulation creates an ideal environment for bacterial colonization leading to chronic inflammatory status with the ultimate consequence of pulmonary failure. To deal with these pathologies efficiently, there is a need to better understand and characterize the evolution of mucus rheology throughout the disease and its different phases. As a matter of fact, mucus rheological properties can change over time depending on several factors such as the inflammatory or infectious status. Thus, studies suggested that the storage modulus G' could be used as an indicator of exacerbation phases or infection by pathogens. However, mucus is difficult to study at a macrorheological level, especially because of its heterogeneity. Sputum, i.e. a mucus solution produced and collected when patients expectorate, is a highly complex fluid containing exogenous elements such as extracellular DNA, cell debris and bacteria that can impact rheology.

Our goal is thus to characterize the influence of heterogeneity on mucus rheology, by using micro-rheology. We use three types of samples, of gradually increasing complexity. As a first step, we use an acrylamide-based synthetic polymer, Flopaam 6035S (SNF Floerger), which mimics the macroscopic rheological behavior of sputum from stable CF patients and is assumed fully homogeneous. Then we use mucus produced in-vitro by human bronchial epithelial (HBE) cells harvested from F508del-CF or non-CF lungs and provided by Epithelix (Geneva, Switzerland). This mucus can be considered free from exogenous elements. The final step is patient’s sputa obtained in collaboration with Grenoble University hospital.

Micro-rheology experiments are performed using optical tweezers, by trapping Polystyrene microspheres ($5\mu\text{m}$ in diameter) incorporated to the sample, and measuring the instantaneous force exerted under oscillatory displacement. As expected, cell debris and bacteria are present in mucus samples (both sputa and HBE-cultured). In these heterogeneous samples, we succeeded in trapping and moving beads over relatively long distances within the matrix phase, while noting a strong restoring force, which confirms the visco-elasto-plastic nature of ‘native’ mucus. Finally, the cell debris and bacteria can also be used as tools to visualize the area affected when the trapped bead is displaced, providing local information on the shear field around it.

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Keywords: mucus, micro, rheology, optical tweezers

Recycling of waste multilayer blown films towards the improving of their end-use values: Structure, rheology, tribology and morphological investigations

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Abstract

Nowadays, effective recycling of post- production and post-consumption plastic films has become a big challenge for both industrial and academic communities. In a circular economy context with the dual problems of depletion of natural resources and the environmental impact of a growing volume of wastes, it is of great importance to develop an efficient recycling process of multilayered films dedicated to agricultural, industrial and packaging applications. The main objective of the present work concerns the design and development of high mechanical performance and/or barrier multilayers structures based on recycled PP/PE multilayered waste films. The present recycled materials are shown to be complex and they are based on LDPE, LLDPE, homo or PP copolymers, with also the presence of fillers and PSAs (Pressure-sensitive adhesives). Our first focus is to gain a better understanding of rheological and morphological changes during the thermo-mechanical recycling process of these multi-phase systems as blends and therefore when they are incorporated in multilayer thin film during coextrusion. Different ethylene-propylene copolymers as a physical compatibilizer with different ratios and molar mass distributions. Hence, various recycled formulations were then obtained. Shear and elongation properties were studied to highlight their viscoelastic flow and composition effects during their blown film coextrusion as central layers when they are brought together with virgin or recycled systems in multilayer films. Secondly, a particular phenomenon was observed with the films obtained post- recycling process. It consists on the migration of small pressure-sensitive adhesive molecules (PSA) such as PIB (Polyisobutylene) from bulk to the surface of the films. During the first use of the films, this additive is employed to provide sticky properties to the films surface, which are necessary for certain applications. However after the recycling process, this additive migration represents a drawback for the manufacturing process. To understand this phenomenon, model films were prepared by blowing extrusion with different compositions and processing parameters (BUR and TUR). It was shown that the rheological properties at the molten state were influenced despite the small PIB amounts. The elongation properties are indeed very sensitive in uniaxial flow especially for low molar masses of PIB. Mineral fillers with different nature, size and shape were therefore added, demonstrating their role to decrease and/or inhibit the diffusion process of the PIB. Furthermore, quantitative investigation were performed and developed, using Tack test and Tribology to quantify and model the diffusion process depending of aging time, temperature and the additive properties.

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Keywords: coextrusion, recycling, rheology, multilayer films, polymer blends, compatibilization, morphology, diffusion.

The influence of sugar addition on rheological properties of black currant pectin gels.

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Abstract

Pectins are polysaccharides of plant origin, built from galacturonic acid monomers linked by

$\alpha \rightarrow 1,4$ glycosidic bonds with monosaccharides like arabinose, rhamnose, fructose, galactose or xylose as side chains. Part of carboxyl groups of galacturonic acid are esterified with methanol (DM). DM is the main division criterion for pectins, because it determines the gelation mechanism. According to this criterion one can distinguish high methylated pectin (HM) with DM > 50% and low methylated (LM) one with DM < 50%. HM pectin gels in the presence of simple sugars (like glucose, fructose or saccharose), when LM pectins needs divalent ions like calcium to form gel. Almost all naturally occurring pectins are those of high methyl content, LM pectins are obtained by controlled deesterification. Pectin's properties, in particular the particle size and monosaccharides profile can vary significantly depending on the botanical origin. Differences in pectin chain structure may cause intensifying or suppressing of some properties, like viscosity or gelling ability. Thanks to their useful properties pectins are one of the most common used food ingredient, they act as thickener or gelling agent. Due to high demand for pectins, source of obtaining should be inexpensive, thus the largest source of pectin are apple and citrus pomace. Due to their popularity pectins from that sources are the best known and the most often described in literature. In literature there are also reports of pectin from other sources, such as: blueberry, strawberry, sugar beet, okra pods, cornelian cherry or currants. The variability of pectin's properties due to their origin makes it possible to choose the type of pectin suitable to obtain desired result in industry, but first of all their properties should be well described.

Poland is one of main black currant producers in Europe and due to the good availability of high quality material, it was decided to examine the properties of pectin derived from this fruit. Currants are rarely used source for pectin extraction, only some information about black currant pectin properties was found.

Pectin was isolated from black currant Tisel variety. Average molecular mass and sugar composition was determined using gel permeation chromatography. DM was determined with standard titrimetric method. The tests of the viscoelastic properties were performed using the RS6000 rheometer (Haake, Germany). A cone-plate geometry was used with parameters: diameter: 35 mm, angle 2, gap size 0.105 mm. The sample loading area, as well as pectin

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sample were pre-heated to 50°C before applying. Firstly sample was quenched to 15°C for 30min, than oscillatory test was conducted at 15°C, storage (G') and loss (G'') modulus were measured. The effect of added sugar on gelling properties of black currant pectin was investigated.

Obtained results indicates, that pectin derived from black currant has significantly lower average molecular mass than most commonly used pectin. Moreover it is highly methylated and has strong tendency to form gel. Addition of sugar strongly affects the gelation process, increasing obtained value of storage modulus (G') what indicates gel strengthening.

Keywords: Pectin, rheology, black currant

The influence of starch microparticles concentration on nonlinear rheological properties of Pickering emulsions

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Abstract

Emulsions are heterogeneous systems consisting of at least two immiscible liquids, one of which is a continuous phase and the other a dispersed phase, in the form of fine droplets. Emulsions are thermodynamically unstable systems and therefore tend to destabilise due to phenomena such as creaming, sedimentation, flocculation or coalescence. The most important for the durability of the emulsion is the correct selection of the substance that serves as a stabilizer for the given type of emulsion. There are different types of emulsifiers used in food emulsions, whose main mechanism of operation is to limit the aggregation of dispersed phase particles by reducing the interphase tension, i. e. surfactants, phospholipids. Currently, there is a growing interest in Pickering emulsions, where the stabilization mechanism consists in adsorption of very small particles on the interphase surface instead of the surfactant, thus creating a physical barrier around the dispersed phase. This is solid adsorption and Pickering emulsions are therefore considered more durable than surfactant-based emulsions. Therefore, an attempt was made to obtain emulsions which would provide the basis for a variety of products due to the expected structure. Emulsions were obtained by selecting the optimal ratio of water to oil phase and the amount of stabilizer.

The first stage of this study was to obtain the starch fractions of increased crystallinity by enzymatic hydrolysis and then use them to stabilize food Pickering emulsions. In order to obtain the desired properties of starch fractions, the process of enzymatic hydrolysis of starch was optimized, including selection of raw material, selection of enzyme and selection of appropriate parameters of the preparation process. The next stage assumed the characteristics of the obtained starch fractions.

In particular, the determination of the degree of crystallinity and the analysis of particle size distribution using dynamic light scattering. On the basis of the results obtained, the starch fractions were selected, which were used as a stabilizer to produce an oil in water (O/W) type emulsion. The obtained emulsions were subjected to microscopic image analysis, stability testing, including checking the stability of the obtained emulsions over time, and above all rheological characteristics. Rheological characteristics are crucial when designing a new product to be manufactured on an industrial scale. The obtained emulsion is the basis for the new product, however, on the basis of rheological characteristics of the material, it is possible to assess the technological suitability for a specific end product.

The experiment plan involved influence starch microparticles concentration on rheological

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properties of O/W emulsions. Large Amplitudes Oscillatory Shear (LAOS) were used to study the nonlinear viscoelastic properties of selected emulsions. It was demonstrated that all tested systems exhibited viscoelastic properties, but the area of linear dependency was limited. The systems exhibited the intersection of G' and G'' (first Fourier harmonic) in the range of deformation from 0.01 to 1.0. Analysis was shown characteristic maxima on G'' which is typical for multiphase systems.

Also dissipation coefficient was calculated as a function of strain amplitude. Also geometrical decomposition of Lissajous curves was used to calculate Chebyshev coefficients for elastic and viscous parts. Viscous Lissajous curves were characterized double loops. This rheological study allowed

to point the main factor determining rheological properties of O/W emulsion and shown the future direction of industrial application of this kind multiphase systems.

Keywords: Pickering emulsion, LAOS, rheological behavior

Relation between inter-particle force profiles, particle friction, and rheology in dense suspensions: an experimental proof using AFM.

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TBD

Keywords: dense suspensions, shear thickening, AFM, inter, particle force profiles, friction measure

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