





KU LEUVEN

Rheology and Fracture Mechanics of Foods by Ton van Vliet

Chapter 13 - Gels

Mathieu Meerts
NNF Summer Reading Group, August 1, 2017



Who is this "Barrel in the Canal"?

- Ton van Vliet
- Prof. at Wageningen University
- Main research topics
 - Structure of food systems
 - Non-linear behavior
 - Fracture mechanics
- Retired in 2010



Journal of Cereal Science 48 (2008) 1-9

Review

Strain hardening as an indicator of bread-making performance: A review with discussion

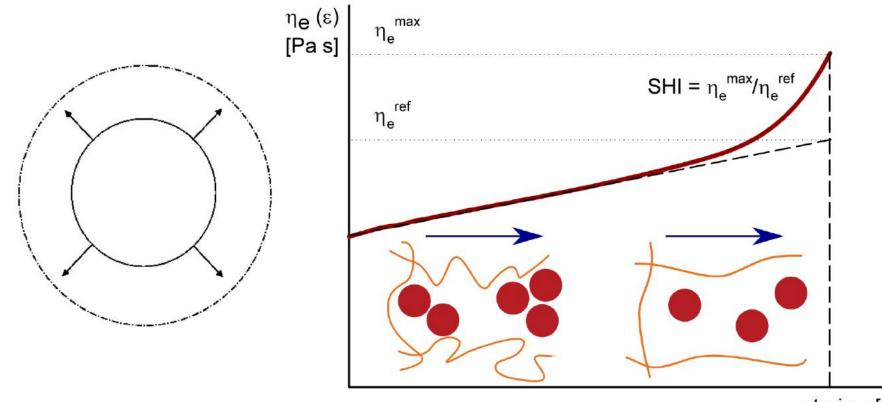
Ton van Vliet^{a,b,*}



Side note: van Vliet & dough rheology

Wheat flour dough

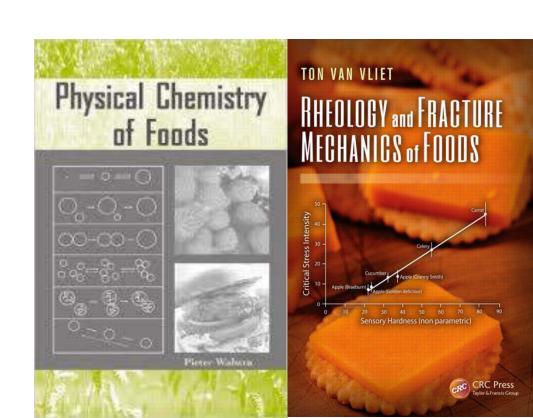
- Gluten proteins (very broad MW distribution)
- Starch particles (ellipsoidal 25-40 μm; spherical 5-10 μm)



What is the book about?

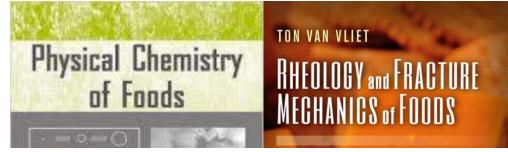
- Deformation, flow, and fracture behaviors of food systems
- Relation with structure at mesoscopic & macroscopic scale
- Material classes:
 - Dispersion
 - Macromolecular solution
 - Solid
 - Gel (Chapter 13)
 - Emulsion and foam

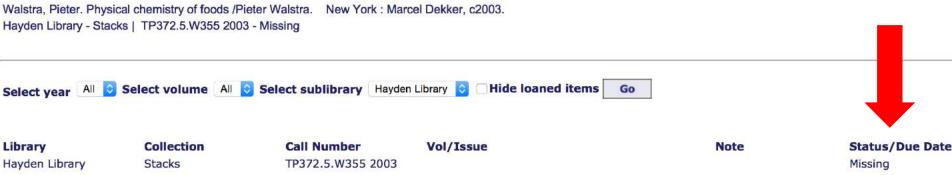
P. Walstra, Physical Chemistry of Foods Marcel Dekker (NY), 2003



What is the book about?

- Deformation, flow, and fracture behaviors of food systems
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- Material classes:
 - Dispersion
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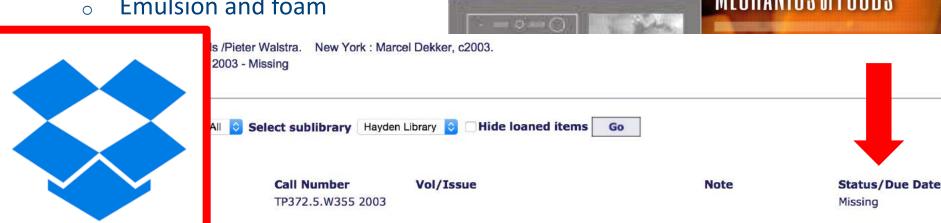


What is the book about?

- Deformation, flow, and fracture behaviors of food systems
- Relation with structure at mesoscopic & macroscopic scale
- Material classes:
 - Dispersion

Having trouble figuring out what to do next? Ask Us!

- Macromolecular solution
- Solid
- **Gel (Chapter 13)**



Physical Chemistry

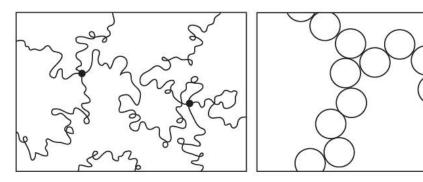
TON VAN VLIET

RHEOLOGY and FRACTURE

Starting point

Gels in food products

- Polymer networks (cf. Jianyi)
 - Chemically vs. physically
 - Flexible vs. stiff chains

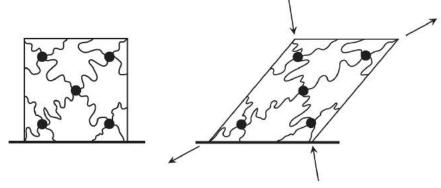


- Particle networks (cf. Yacouba, Bavand, Michela)
 - Hard vs. deformable
- Large deformation properties important for the handling, usage, and eating characteristics of food products

Starting point

Formula for G:

$$\sigma = -N \frac{\mathrm{df_s}}{\mathrm{dx}} \Delta x$$



N = # stress-carrying strands per cross section

$$\Delta x = C \cdot \gamma$$
 (C = a/26 for isotropic gel with straight strands cf. Bavand) $f_S = -\frac{dF}{dx}$ (F = Gibbs energy)

$$G = CN \frac{d^2F}{dx^2} = CN \frac{d(dH - TdS)}{dx^2}$$

long flexible chains

particle networks

stiff chains

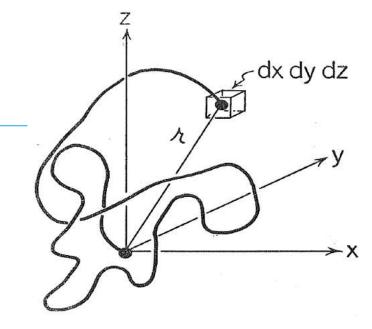


Case study: alginate gels

Side note: pectin gels

Formula for G:

$$G = CN \frac{d^2F}{dx^2} = CN \frac{d(dH - TdS)}{dx^2} \approx -CNT \frac{d(dS)}{dx^2}$$



Boltzmann equation: $\mathbf{S} = \mathbf{k}_{\mathrm{B}} \ln \mathbf{\Omega}$

 Ω = probability that situation will occur

• Probability of any given chain having components x_i , y_i , z_i

$$\omega_{i} = W(x_{i}, y_{i}, z_{i}) \Delta x \Delta y \Delta z$$

• Probability that each chain in the entire system complies with specified set of coordinates

$$\prod_i \omega_i^{\beta_i}$$

Particular selection of chains is of no importance, so

$$\Omega = \alpha! \prod_{i} \left(\omega_{i}^{\beta_{i}} / \beta_{i}! \right)$$

Formula for G:

$$G \approx -CNT \frac{d(dS)}{dx^2}$$

$$S = k_B \ln \Omega$$

 Ω = probability that situation will occur

• Probability that the chain vector distribution, specified by β_i , will occur

$$\Omega = \alpha! \prod_{i} \left(\omega_{i}^{\beta_{i}} / \beta_{i}! \right)$$

• Taking logarithm + Stirling's approximation formula: $\ln n! \approx n \cdot \ln(n) - n$

$$\ln \Omega = \sum \beta_i \ln \left(\frac{\omega_i \alpha}{\beta_i} \right)$$

• Substitute in expression for G + use Gaussian function for ω_i + assume constant volume α = number of chains between 2 cross-links

c = g chains between 2 cross-links per unit volume M_C = av. molecular weight of chain between 2 cross-links

$$\mathbf{G} = \frac{\alpha \mathbf{k_B T}}{\mathbf{V}} = \frac{\mathbf{cR_g T}}{\mathbf{M_c}}$$

Formula for G:

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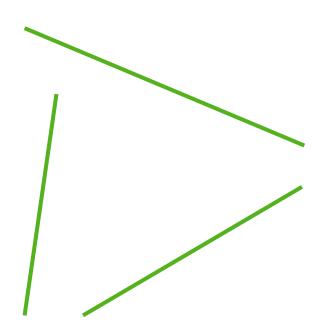
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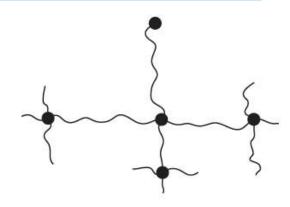
• Substitute in expression for G + use Gaussian function for ω_i + assume constant volume

 α = number of chains between 2 cross-links c = g chains between 2 cross-links per unit volume M_C = av. molecular weight of chain between 2 cross-links

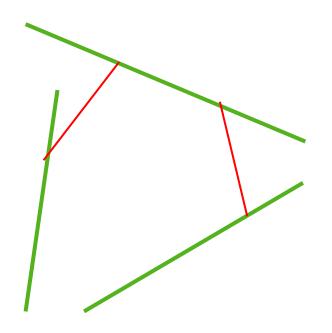
$$G \sim T$$

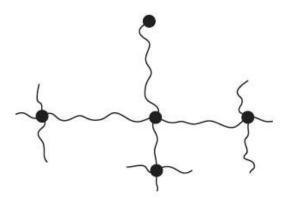
$$G = \frac{\alpha k_B T}{V} = \frac{c R_g T}{M_c}$$



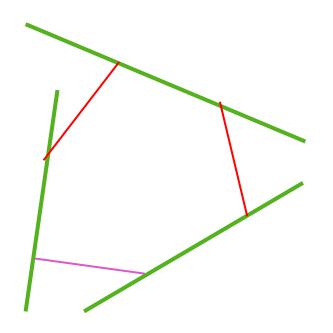


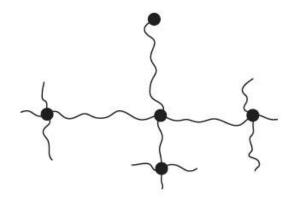
Correction for dangling ends



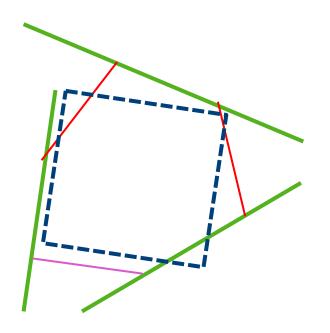


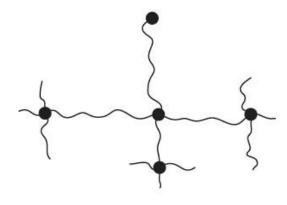
• N − 1 cross-links to create giant molecule



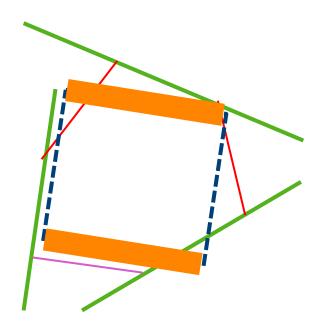


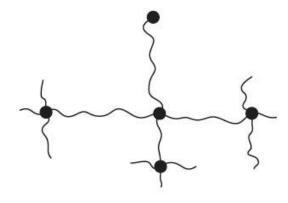
- N-1 cross-links to create giant molecule
- Each additional cross-link





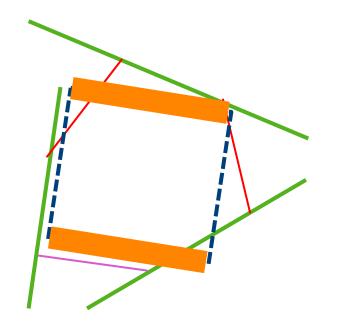
- N − 1 cross-links to create giant molecule
- Each additional cross-link = closed-loop circuit

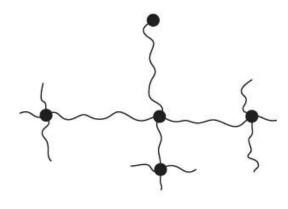




- N − 1 cross-links to create giant molecule
- Each additional cross-link = closed-loop circuit
- Closed-loop circuit = 2 elastic elements

Correction for dangling ends





- N − 1 cross-links to create giant molecule
- Each additional cross-link = closed-loop circuit
- Closed-loop circuit = 2 elastic elements

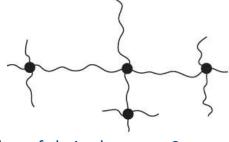
$$\alpha_e/2 = \alpha/2 - N$$

 $\alpha_e/2$ = number of effective cross-links $\alpha/2$ = number of cross-links N = number of primary chains

Correction for dangling ends

$$\alpha_e/2 = \alpha/2 - N$$

 $\alpha_e/2$ = number of effective cross-links $\alpha/2$ = number of cross-links N = number of primary chains



 $\alpha_{\rm e} = \alpha - 2N = \alpha(1 - 2N/\alpha)$

 α_e = number of chains between 2 cross-links

= twice the number of loops

with

$$N = \frac{N_0 M_0}{M_N} = \frac{V}{\overline{v} M_N}$$

$$\alpha = \frac{N_0 M_0}{M_C} = \frac{V}{\overline{V} M_C}$$

$$G = \frac{k_B T \alpha_e}{V} = \frac{k_B T \alpha}{V} \left(1 - 2 \frac{M_C}{M_N} \right)$$

 N_0 = number of units

 M_0 = molecular weight of unit

 \overline{v} = specific volume

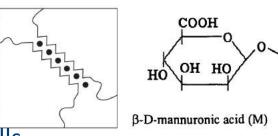
M_N = number average molecular weight of primary molecules

M_C = average molecular weight of chain between two cross-links

Other complications:

sol fraction, formation of entanglements

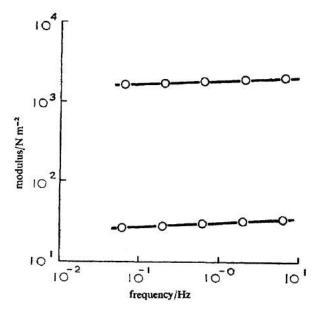
Case study: alginate gels*



Alginate = polysaccharide from algae cell walls

= linear copolymer of M-blocks, G-blocks and MG-blocks

Dilute aqueous solution → gel by cross-linking corresponding blocks with Ca²⁺



$$G \neq f(\omega)$$
; $G \sim T \rightarrow$ rubber elasticity?

$$\mathbf{G} = \frac{\mathbf{c}\mathbf{R}_{\mathbf{g}}\mathbf{T}}{\mathbf{M}_{\mathbf{c}}}$$

 M_C = average molecular weight of chain between two cross-links M_C = 12 kg/kmol

M = 130 kg/kmol $\rightarrow 11 \text{ cross-links per alginate molecule}$

COOH

α-L-gluluronic acid (G)

[η] indicates only 2 statistical chain elements between 2 cross-links

Limitations of the Rubber Elastic Theory

Linear behavior

Why did it work for the case study?*

Long, flexible chains with many statistical elements

- → polysaccharide chains are stiffer (bulky side groups)
- → no Gaussian distribution: Rubber Elastic Theory in linear behavior

Limitations of the Rubber Elastic Theory

Linear behavior

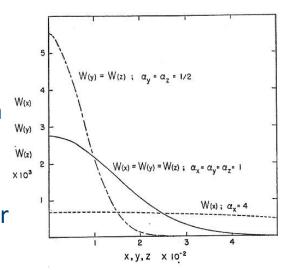
Why did it work for the case study?*

Long, flexible chains with many statistical elements

- → polysaccharide chains are stiffer (bulky side groups)
- → no Gaussian distribution: Rubber Elastic Theory in linear behavior
- Non-linear behavior

Deformation: distortion of Gaussian distribution Finite chain extensibility: increase in enthalpy (Strain-induced crystallization)

- → Rubber Elastic Theory in non-linear behavior
- → Treloar theory**





Contents lists available at ScienceDirect

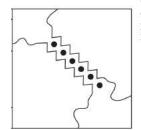
Innovative Food Science and Emerging Technologies

d Science and Emerging Technologies



Side note

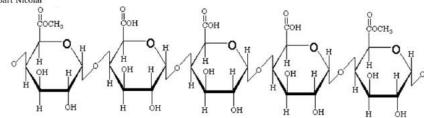
Case study: pectin gels*



Pectin based food-ink formulations for 3-D printing of customizable porous food simulants



Valérie Vancauwenberghe^{a, a}, Louise Katalagarianakis^a, Zi Wang^a, Mathieu Meerts^b, Maarten Hertog^a, Pieter Verboven^a, Paula Moldenaers^b, Marc E. Hendrickx^c, Jeroen Lammertyn^a,



Pectin = polysaccharide from plant cell walls

= galacturonan backbone (or other) + partially methyl esterified

Low methoxyl pectin: strong gels with Ca2+ ions through egg-box junctions







Ca²⁺/
pectin

^{*} Vancauwenberghe et al. IFSaET 42:138-150 (2017)



Case study: casein gels

Small deformation properties for fractal particle gels

• Starting point: $G = CN \frac{d^2F}{dx^2}$

N = # stress-carrying strands per cross section

Small deformation properties for fractal particle gels

- Starting point: $G = CN \frac{d^2F}{dx^2}$ N = # stress-carrying strands per cross section
- Fractal clusters are scale invariant: $N \propto R_g^{-2}$

Small deformation properties for fractal particle gels

- Starting point: $G = CN \frac{d^2F}{dx^2}$
- N = # stress-carrying strands per cross section
- Fractal clusters are scale invariant: $N \propto R_g^{-2}$
- In a similar fashion, C and $\frac{d^2F}{dx^2}$ also relate to R_g , but the exact scaling relation depends on the structure of the strands

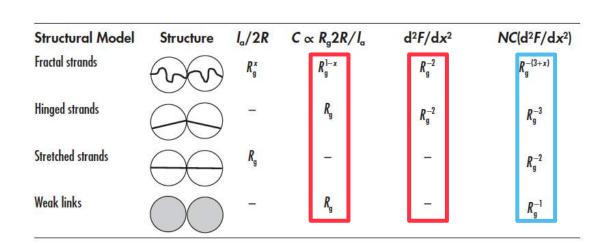
Structural Model	Structure	$I_a/2R$	$C \propto R_{\rm g} 2R/I_{\rm g}$	d^2F/dx^2	$NC(d^2F/dx^2)$
Fractal strands	(N/V)	R_{g}^{x}	$R_{\rm g}^{1-x}$	$R_{\rm g}^{-2}$	$R_{\rm g}^{-(3+x)}$
Hinged strands		-	$R_{\rm g}$	$R_{\rm g}^{-2}$	$R_{\rm g}^{-3}$
Stretched strands	\bigcirc	$R_{\rm g}$	-	-	$R_{\rm g}^{-2}$
Weak links		-	$R_{\rm g}$	-	$R_{\rm g}^{-1}$

Small deformation properties for fractal particle gels

• Starting point: $G = CN \frac{d^2F}{dx^2}$

- N = # stress-carrying strands per cross section
- Fractal clusters are scale invariant: $N \propto R_g^{-2}$
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- So we can write:

$$G = CN \frac{d^2F}{dx^2} \propto R_g^{-k}$$



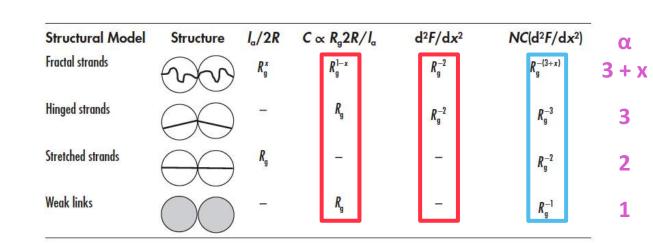
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$$G = CN \frac{d^2F}{dx^2} \propto R_g^{-k}$$

$$G \propto R_{eff}^{-\alpha} \phi^{\alpha/(3-D_f)}$$



Small deformation properties for fractal particle gels

To prove: $N \propto R_g^{-2} \propto R_{eff}^{-2} \phi^{2/(3-D_f)}$ N = # stress-carrying strands per cross section

Starting point:

$$N_p \propto \left(\frac{R_{agg}}{R_p}\right)^{D_f}$$
 N_p = # particles in fractal aggregate R_p = particle radius $N_s \propto \left(\frac{R_{agg}}{R_p}\right)^3$ R_{agg} = aggregate radius N_s = # sites that can be occupied in aggregate by particle

Small deformation properties for fractal particle gels

To prove: $N \propto R_g^{-2} \propto R_{eff}^{-2} \phi^{2/(3-D_f)}$ N = # stress-carrying strands per cross section

• Starting point:

$$N_p \propto \left(\frac{R_{agg}}{R_p}\right)^{D_f}$$

$$N_s \propto \left(\frac{R_{agg}}{R_p}\right)^3$$

$$\phi_{agg} = \frac{N_{agg}}{N_s} \propto \left(\frac{R_{agg}}{R_p}\right)^{D_f - 3}$$

$$\phi_{agg} = \text{volume fraction particles in fractal aggregate}$$

Small deformation properties for fractal particle gels

To prove: $N \propto R_g^{-2} \propto R_{eff}^{-2} \phi^{2/(3-D_f)}$ N = # stress-carrying strands per cross section

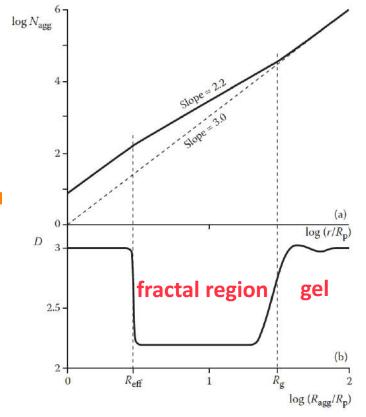
Starting point:

$$\begin{aligned} N_p & \propto \left(\frac{R_{agg}}{R_p}\right)^{D_f} \\ N_s & \propto \left(\frac{R_{agg}}{R_p}\right)^3 \end{aligned} \qquad \phi_{agg} = \frac{N_{agg}}{N_s} \propto \left(\frac{R_{agg}}{R_p}\right)^{D_f - 3} \quad \log N_{agg} = \frac{N_{agg}}{N_s}$$

- $\bullet \quad \phi_{agg} \text{ decreases as } R_{agg} \text{ increases} \\$
- Aggregates touch each other → gel formation
- At scale \gg R_{agg}: gel is homogeneous; D = 3

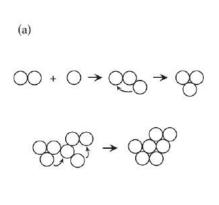
$$\begin{split} & \longrightarrow \phi_{agg} = \phi \\ & \longrightarrow R_g \propto R_p \phi^{1/D_f - 3} \end{split}$$

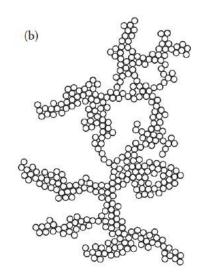
 R_g = average radius of fractal clusters forming the gel



Small deformation properties for fractal particle gels

Complication: interparticle rearrangements during aggregation





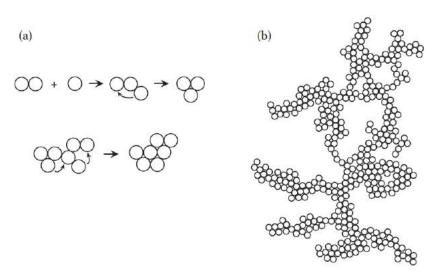
Cluster forming unit ≠ primary particles = small aggregates

Use R_{eff} instead of R_p :

$$R_g \propto R_{eff} \phi^{1/D_f - 3}$$

Small deformation properties for fractal particle gels

Complication: interparticle rearrangements during aggregation



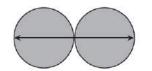
Cluster forming unit ≠ primary particles = small aggregates

Use R_{eff} instead of R_p :

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Note: other types of rearrangement during gelation and aging are also possible (C&D)

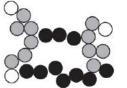
Particle fusion



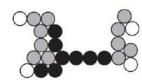
Subparticle rearrangements



Strand rearrangement

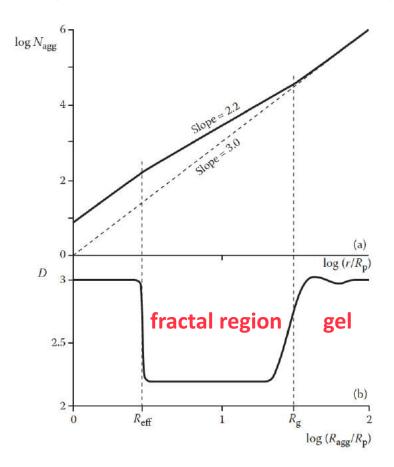






Small deformation properties for fractal particle gels

Complication: interparticle rearrangements during aggregation



Cluster forming unit ≠ primary particles = small aggregates

Use R_{eff} instead of R_p :

$$R_g \propto {R_{eff}} \phi^{1/D_f-3}$$

Fractal behavior only between R_{eff} and R_p For high ϕ : aggregate no longer fractal

Example: D_f = 2.34 and ϕ = 0.3: $R_g \propto R_{eff} \phi^{1/D_f-3} = 6R_{eff}$

Derivation:

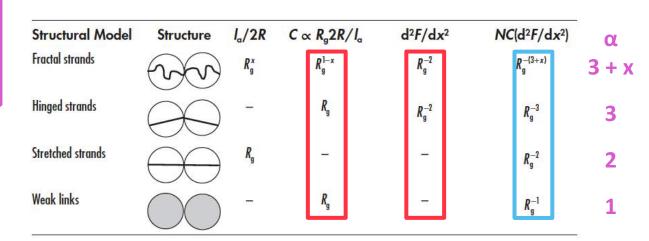
- Mellema et al. J. Rheol. 46:11-29 (2002)
- Lecture Michela

Small deformation properties for fractal particle gels

$$G = CN \frac{d^2F}{dx^2} \propto R_g^{-k}$$

$$R_g \propto R_{eff} \phi^{1/D_f - 3}$$

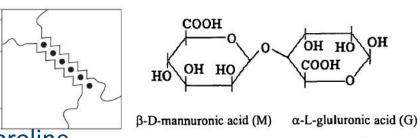
$$G \varpropto R_{eff}^{-\alpha} \phi^{\alpha/(3-D_f)}$$



	Fractal strands	Hinged strands	Stretched strands	Weak links
Rigid units	Particles forming the aggregates	Aggregates, following rearrangements	Particles forming the aggregates	Aggregates, following rearrangements
Type of strands	Fractal strands	Bonds between aggregates	Series of particles linked in a straight line	Weak links between aggregates

Particle networks

Case study: casein gels* (Bavand)



Casein = milk proteins largely composed of proline

= forms micelles in aqueous solutions, kept apart by protruding chains

Isoelectric point (pH 4.6) → chains collapse and gel is formed

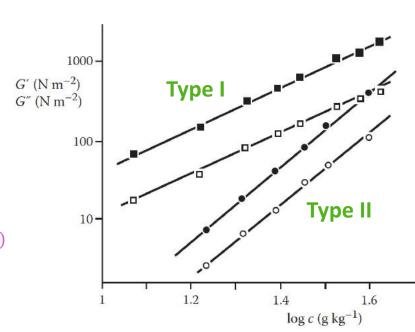
Type I: pH 4.6 @ T = 4°C, heat up to 30°C

→ Stretched strands

Type II: T = 30°C, add gluconolactone

→ Hinged strands

$$G \propto R_{eff}^{-\alpha} \phi^{\alpha/(3-D_f)}$$



Particle networks

Large deformation properties for fractal particle gels

	Modulus G	Fracture stress σ_{fr}	Fracture strain γ_{fr}
Starting point	$G = CN \frac{d^2F}{dx^2}$	$\sigma_{fr} \propto f_{fr}/R_g^2$	-
General equation	$G \propto R_{eff}^{-\alpha} \phi^{\alpha/(3-D_f)}$	$\sigma_{fr} \propto R_{eff}^{-\upsilon} \phi^{\upsilon/(3-D_f)}$	$\gamma_{fr} \propto R_{eff}^{-\beta} \phi^{\beta/(3-D_f)}$
Parameters	D _f , α	$\mathrm{D_f},\!\mathbf{v}$	$\mathrm{D_f}$, $oldsymbol{eta}$

Structural Model	α (Equation 13.14)	υ (Equation 13.19)	β (Equation 13.20)
Fractal strands	3 + x	2	-X
Hinged strands	3	2	0
Stretched strands	2	2	0
Weak links	1	2	1



Case study: soy protein isolate

Formation of protein gels

Parameters inducing the gelation process

- Temperature (case study: soy protein isolate)
- pH (presentation Bavand: casein gels)
- Ionic strength
- Addition of specific salts
- Enzyme action

Case study: soy protein isolate (SPI)*

Soy protein = combination of β -conglycinin and glycinin

= hydrophobic and sulfhydryl (-SH) protein residues

(A) Denaturation of globular proteins T个

→ exposure of functional groups

(B) Aggregation to form spherical particles

→ SS bridges, hydrophobic, H-bonds

(C) Particles form network

 \rightarrow initially more or less fractal

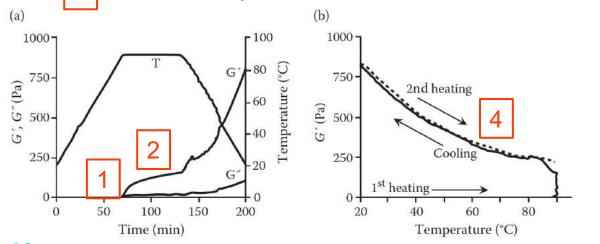
→ loss of fractality due to rearrangements

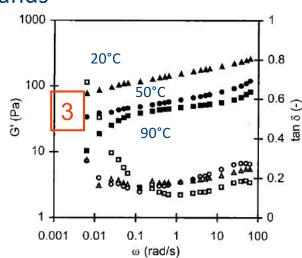
⁽strands just become thicker, pores smaller) * Renkema and van Vliet. J. Agri. Food Chem. 50:1569-1573 (2002) & Renkema. Food Hydrocolloids 18:39-47 (2004) & Walstra. Physical chemistry of foods (2003), Chapter 15 & Gosal and Ross-Murphy, COCIS 5,188-194 (2000)

Case study: soy protein isolate (SPI)*

Effect of temperature

- 1 Denaturation is a prerequisite for gel formation
- 2 Increasing network strength by incorporation of protein and rearrangements
- 3 Short lifetime → no covalent SS bonds
- 4 Reversibility → no covalent bonds/fracture of strands





¹⁹

Case study: soy protein isolate (SPI)**

Effect of pH and ions

Factors affecting rheological properties of heat-set network

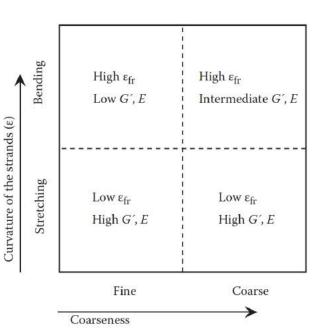
- Amount of protein incorporated
- Network structure
 - o Coarseness of strands (< CLSM, permeability) ↑ at isoelectric point or high ionic strength

Relative number of straight strands (G', E)

Curvature of strands (< fracture strain)

- 1 Fracture strain $\varepsilon_{fr} \sim curvature$
- 2 Storage modulus G' ~ curvature + coarseness
- Fracture stress $\sigma_{fr} \sim \text{coarseness} + \text{amount}$

... provided that interaction forces do not change





D. Plastic fats

Case study: palm oil in sunflower oil

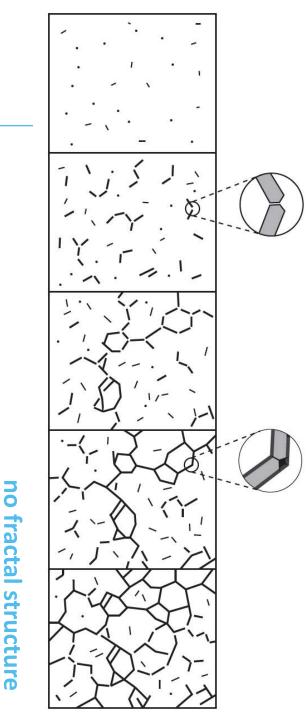
Formation of plastic fats

Network of triglyceride fat crystals in a triglyceride oil

- T↓
- (1) Formation of fat crystal nuclei
- (2) **Growth** of fat crystals
- (3) Aggregation of fat crystals

Special characteristics of these particle gels

- Processes occur simultaneously (slow cryst.*)
- Sintering (particle fusion, cf. part B)
- Recrystallization (composition/polymorph**)
- Crystals are strongly anisometric



^{*} Kloek, Mechaniclal properties of fat (1998), Chapter 3

^{**} Walstra. Physical chemistry of foods (2003), Chapter 15

Case study: hydrogenated palm oil in sunflower oil*

Palm oil = reddish pulp of the fruit of oil palms

= saturated palmitic acid and mono-unsaturated oleic acid

Sunflower oil = compressed seeds of sunflower

= poly-unsaturated linoleic acid and mono-unsaturated oleic acid

Case study: hydrogenated palm oil in sunflower oil

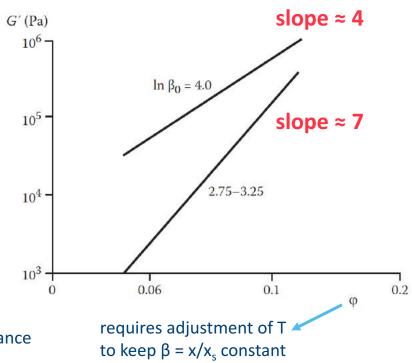
Small deformation properties

- Very small linearity limit (10⁻⁴)
- Scaling law for shear modulus G

$$G \propto \frac{\delta^2 F}{\delta H^2} \phi^{\alpha/(3-D_f)}$$

$$G \propto \frac{A_H}{H_0^3} \varphi^{\alpha/(3-D_f)}$$

A_H = Hamaker constant H₀ = hardcore inter-particle distance



 D_f (LS) = 1.7

- !! Rheology and light scattering do not agree on D_f:
- → fractal nature is lost

D_f	slope = 4	slope = 7
α = 1	2.75	2.86
$\alpha = 4$	2	2.42

Case study: hydrogena

Small deformation propert

- Very small linearity limi
- Scaling law for shear m

Kamphuis and Jongschaap Colloid & Polymer Sci 263:1008-1024 (1985)

Lennard-Jones energy potential for the potential energy content ΔE of a bond between two spherical nonpolar particles each having a diameter D :

$$\Delta E = \frac{A_H D}{24H} \left\{ 1 - \frac{1}{420} \left(\frac{r_o}{H} \right)^6 \right\}.$$

$$f_c = -\frac{\partial \Delta E}{\partial H} = \frac{A_H D}{24H^2} \left\{ 1 - \frac{1}{60} \left(\frac{r_o}{H} \right)^6 \right\}$$

$$\frac{\delta f_c}{\delta H} = \frac{A_H D (15 H^6 - r_0^6)}{180 H^9}$$

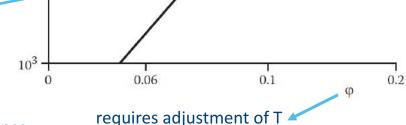
and therefore $\frac{\delta f_c}{\delta H} \propto \frac{A_H}{H^3}$

$$G \propto \frac{\delta^2 F}{\delta H^2} \phi^{\alpha/(3-D_f)}$$

$$G \propto \frac{A_H}{H_0^3} \varphi^{\alpha/(3-D_f)}$$

 A_H = Hamaker constant

H₀ = hardcore inter-particle distance



 D_f (LS) = 1.7

to keep $\beta = x/x_s$ constant

!! Rheology and light scattering do not agree on D_f:

→ fractal nature is lost

D _f	slope = 4	slope = 7
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α = 4	2	2.42

Case study: hydrogenated palm oil in sunflower oil

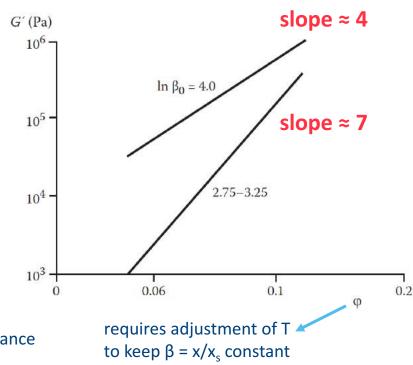
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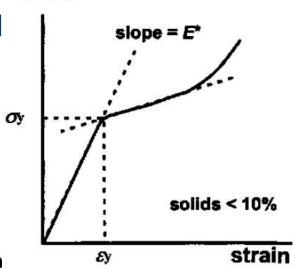
Case study: hydrogen. palm oil in sunflower oil

Large deformation properties

- Uniaxial compression/penetration tests
- Very small linear region: E → E_{app}
- Stress-overshoot
- Yielding (σ_v) (not fracture, unless high φ)
- Plastic flow

$$E_{app}$$
, $\sigma_y \propto \varphi^{\mu}$

Parameter	Rest	High Shear	
		20 kg/h	50 kg/h
σ_{y}	3.9	1.1	1.9
E_{app}	3.8	1.2	2.4



stress

