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EVALUATION OF THIXOTROPIC PARAMETERS FOR LING HEATHER AUTHENTICATION

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Spain

5th International Symposium on Bee Products, 7th-10th May, 2019

Malta
2019

Introduction

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Ling heather honey (*Calluna vulgaris* (L.) Hull)

- Greatly appreciated
- Difficult to extract → authentication
- Expensive

Melissopalynology

+

Sensory

Rheological

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Introduction

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Newton's law

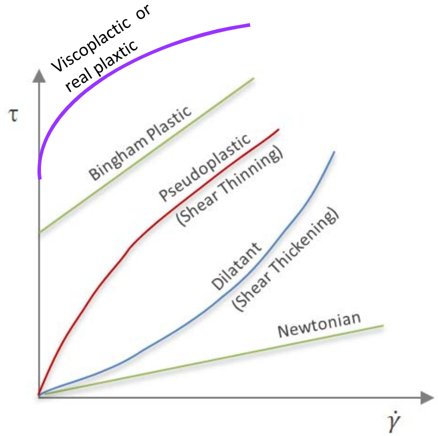
$\tau = \mu \dot{\gamma}$

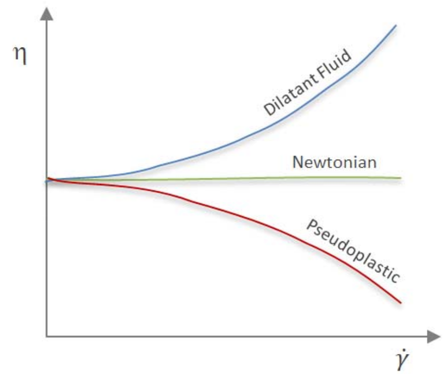
$\mu \neq f(\dot{\gamma}, t)$

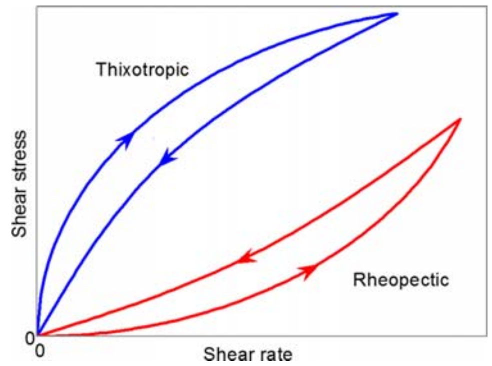
NEWTONIAN

NON-NEWTONIAN

- **TIME-INDEPENDENT**
  - **PURELY VISCOUS PSEUDOPLASTIC BEHAVIOUR**
  - **PURELY VISCOUS DILANTANT BEHAVIOUR**
  - **PLASTIC BEHAVIOUR**
- **TIME-DEPENDENT**
  - **THIXOTROPIC**
  - **RHEOPECTIC**







$\mu = f(\dot{\gamma}, t)$

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OBJECTIVE

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The aim of this work was to assess the reliability of four rheological parameters to help characterize ling heather honeys in comparison with multiflora honeys rich in ling heather pollens and bell heather samples (*Erica* spp.), some of them very close to ling heather monoflorality.

Materials & Methods

22 artisanal honeys. 2015. Spanish regions

HONEY	Chestnut	Multifloral	Honeydew	Heather
Number	2	4	1	15
Abreviation	C	M	HD	

H	HL	L
• Ericaceae > 45% • *C. vulgaris* < 10%	• Ericaceae > 45% • *C. vulgaris* > 10%	• Ericaceae < 45% • *C. vulgaris* > 10% • Sensory characteristics

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Materials & Methods

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40ºC→ 30ºC

Moisture

Haake VT 550

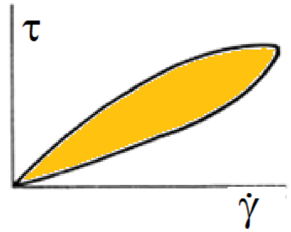
Exp. 1 $\dot{\gamma} \neq$ cte → **Shear rate cycles (25°C)**

up-flow (γ =0.09-100 s^{-1} , 300 s)
stationary (100 s^{-1}, 60 s)
down-flow (100-0.09 s^{-1}, 300 s)

Ostwald-de Waele

$$\mu = k\dot{\gamma}^{n-1}$$

Time-dependent



Exp. 2 $\dot{\gamma}$ = cte → **Constant shear rate (γ = 100 s^{-1}, 600 s, 25°C)**

Weltmann model $\tau = A - B(lnt)$

$$a = \int_1^i f(x)dx = (x_2 - x_1)\frac{\Delta y_1 + \Delta y_2}{2} + \ldots + (x_i - x_{i-1})\frac{\Delta y_{i-1} + \Delta y_i}{2}$$

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RESULTS

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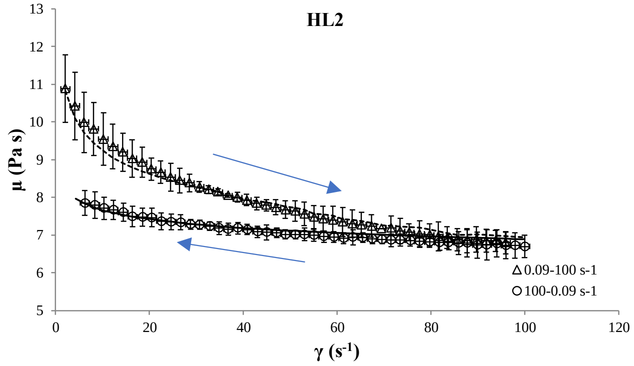
Exp. 1

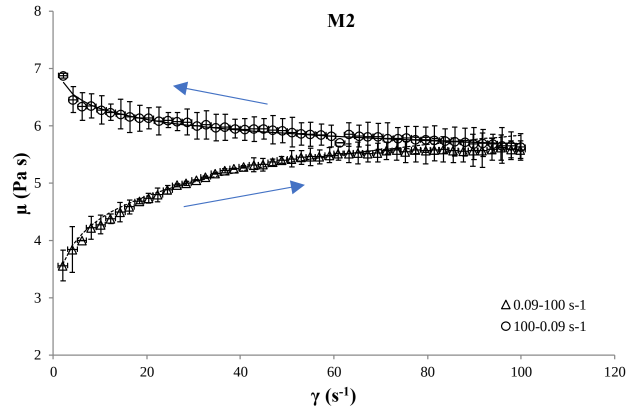
Shear rate cycles

All samples are time-dependent fluids

Good correlation between experimental and calculated apparent viscosity

Successfully fitted to Ostwald-de Waele model (r>0.996)





RESULTS

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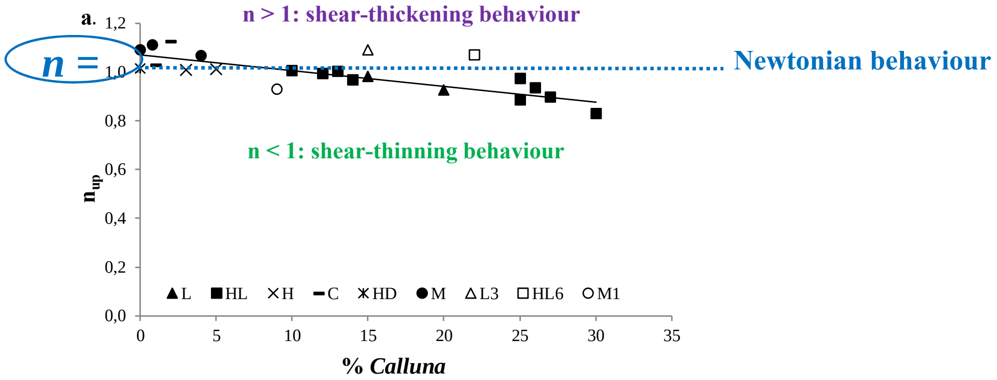
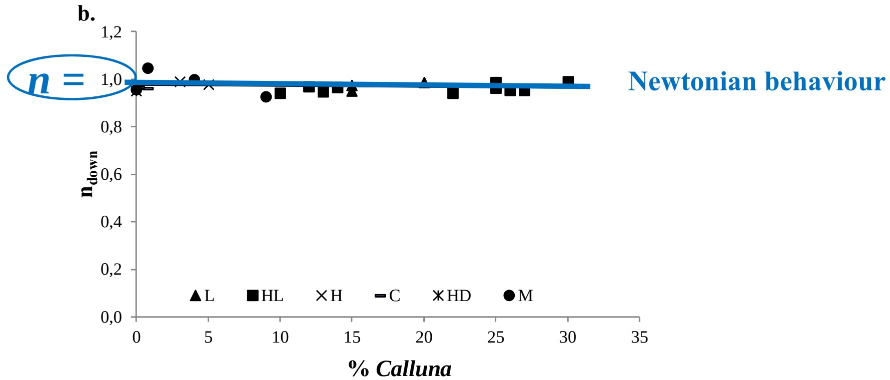
Exp. 1

Shear rate cycles

Ostwald-de Waele model $\mu = k\dot{\gamma}^{n-1}$



Different n_{up} with *Calluna* pollen %



Similar $n_{down} \approx 1$

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RESULTS

Exp. 1 **Shear rate cycles**

Linear relationship between n_{up} and *Calluna* pollen %

- Calluna > 10% → pseudoplastic fluids
- Calluna < 10% → Newtonian or dilatant behaviour

Sample	$n_{up\ 0.05}$
L1	0.93 ± 0.03
L2	0.98 ±0.06
L3	1.09 ± 0.04
HL1	0.88 ±0.08
HL2	0.90 ±0.03
HL3	0.94 ± 0.12
HL4	0.89 ± 0.04
HL5	0.97 ± 0.02
HL6	1.07 ± 0.03
HL7	0.97 ± 0.07
HL8	1.00 ± 0.06
HL9	0.99 ± 0.08
HL10	1.01 ± 0.02
H1	1.01 ± 0.01
H2	1.01 ± 0.04
C1	1.03 ± 0.07
C2	1.13 ±0.06
HD	1.02 ± 0.03
M1	0.93 ± 0.05
M2	1.07 ± 0.10
M3	1.11 ± 0.13
M4	1.09 ± 0.02

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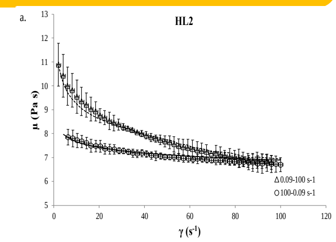
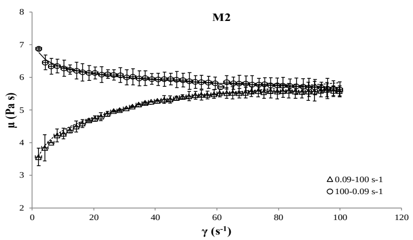
PSEUDOPLASTIC



NEWTONIAN OR DILATANT



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RESULTS

Exp. 1 **Shear rate cycles**

Area of hysteresis loop

$$a = \int_1^i f(x)\,dx = (x_2 - x_1)\frac{\Delta y_1 + \Delta y_2}{2} + \dots + (x_i - x_{i-1})\frac{\Delta y_{i-1} + \Delta y_i}{2}$$

All honeys presented **a hysteresis loop** with quite different values of "**a**"

The values of "a" were higher for LING HEATHER honeys than for non-ling heather.

Sample	a [Pa/s]
L1	2250
L2	5549
L3	2026
HL1	6994
HL2	3447
HL3	3870
HL4	7763
HL5	3272
HL6	4340
HL7	8035
HL8	729
HL9	4548
HL10	148
H1	27
H2	992
C1	289
C2	958
HD	780
M1	560
M2	2058
M3	429
M4	4106

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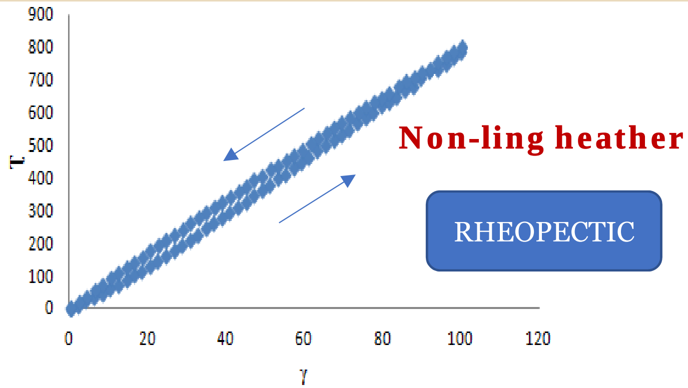
RESULTS

Exp. 1 **Shear rate cycles**

Area of hysteresis loop

$$a = \int_1^i f(x)\,dx = (x_2 - x_1)\frac{\Delta y_1 + \Delta y_2}{2} + \dots + (x_i - x_{i-1})\frac{\Delta y_{i-1} + \Delta y_i}{2}$$

Except for M1, all our **non-ling heather** honeys (H, HD, M, and C) exhibited a **rheopectic behavior.**



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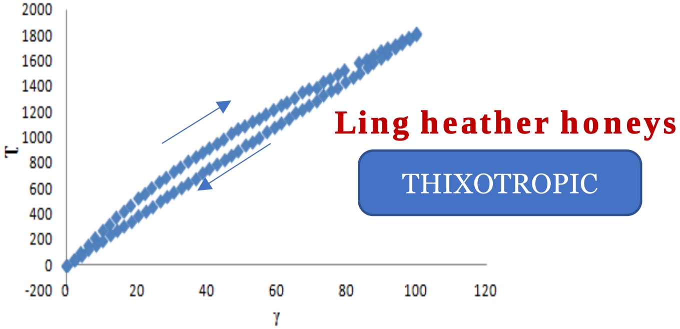
L and HL samples were the honeys that showed a **thixotropic behavior**, but **samples HL6, HL10, and L3** did not follow that trend.



M1 → % Calluna pollen higher

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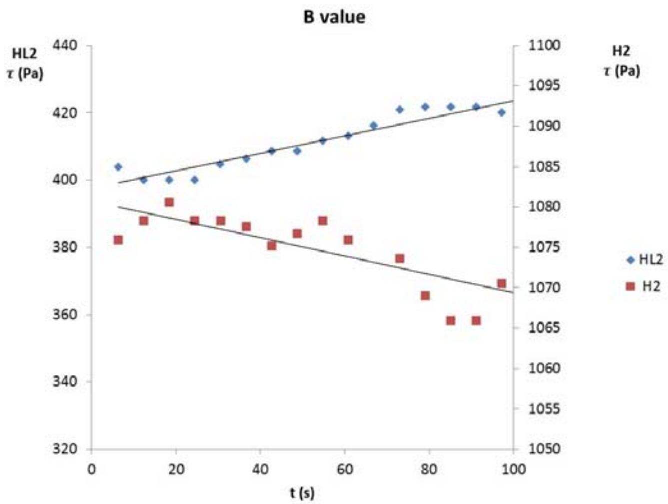
RESULTS

Exp. 2 **Constant shear rate assays**

Weltmann model $\tau = A - B(\ln t)$



Sample	B (Eq.3) [Pa]	r
L1	11.40	0.987
L2	8.48	0.978
L3	50.06	0.974
HL1	33.12	0.987
HL2	3.44	0.932
HL3	7.78	0.964
HL4	32.89	0.975
HL5	10.16	0.978
HL6	28.68	0.970
HL7	65.32	0.995
HL8	19.15	0.965
HL9	9.79	0.984
HL10	33.49	0.984
H1	-1.36	0.940
H2	-0.27	0.935
C1	-6.56	0.975
C2	-18.44	0.974
HD	-1.24	0.955
M1	-2.82	0.937
M2	-6.12	0.963
M3	-9.67	0.984
M4	-24.27	0.965

THIXOTROPIC

RHEOPECTIC

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CONCLUSIONS

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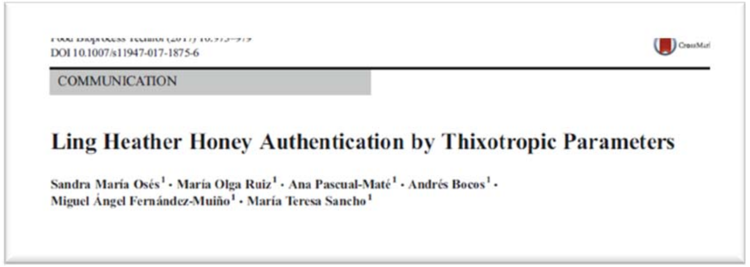
DOI 10.1007/s11947-017-1875-6

COMMUNICATION

Ling Heather Honey Authentication by Thixotropic Parameters

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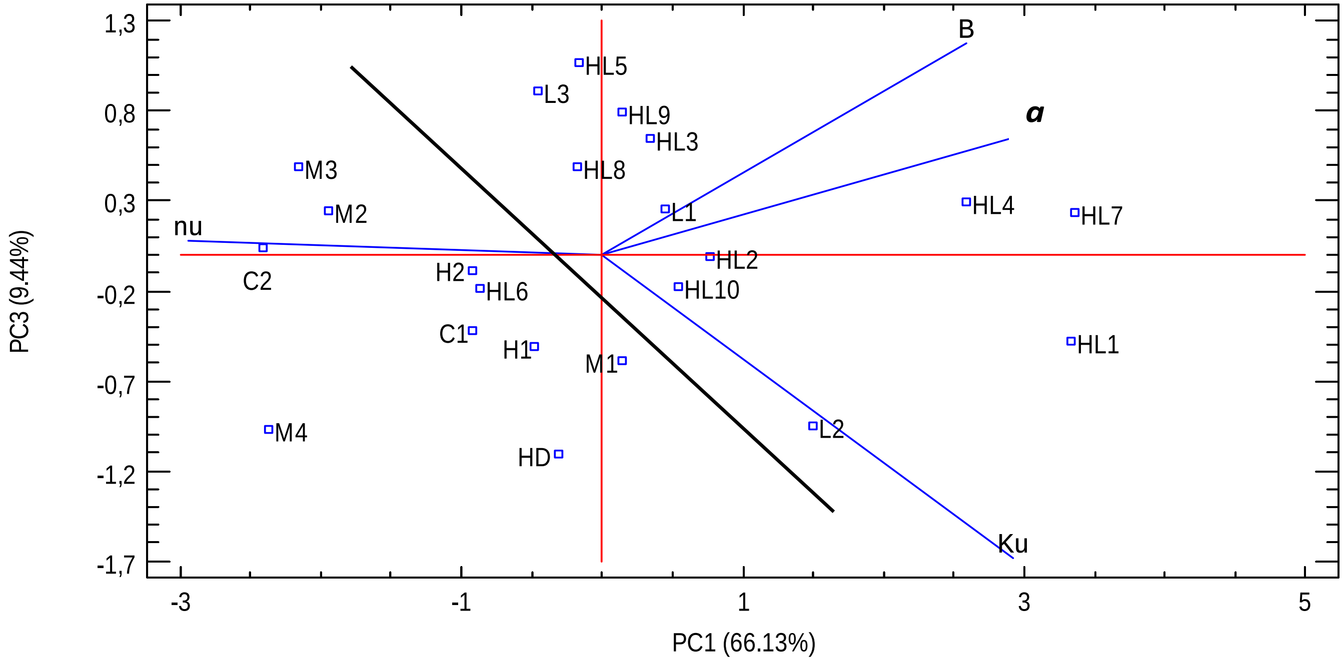

Weltmann index B was the best rheological parameters for ling Heather honey authentication

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THANK YOU

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